PRODUCTION OF ULTRAFINE ZINC POWDER FROM WASTES CONTAINING ZINC BY ELECTROWINNING IN ALKALINE SOLUTION

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Abstract - Production of ultrafine zinc powder from industrial wastes by electrowinning in alkaline solution was studied. Stainless steel and magnesium electrodes were used as anode and cathode, respectively. Morphology, size distribution and composition of the Zn particles were characterized by Scanning Electron Microscopy, Laser Particle Size Analyzer, and Inductive Coupled Plasma Emission Spectrometer. The required composition of the electrolyte for ultrafine particles was found to be 25–35 g/L Zn, 200–220 g/L NaOH and 20–40 mg/L Pb. The optimal conditions were a current density of 1000–1200 A/m^2 and an electrolyte temperature of 30–40 °C. The results indicated that the lead additive exerted a beneficial effect on the refining of the particles, by increasing the cathodic polarization. Through this study, ultrafine zinc powder with a size distribution of around 10 μm could be produced, and considerably high current efficiencies (97–99 %) were obtained.
Keywords: Electrolysis; Ultrafine; Alkaline medium; Lead.

INTRODUCTION

Zinc is usually present in a variety of industrial wastes such as EAF dust, BOF sludge, and jarosite residue (Oustadakis *et al*., 2010; Trung *et al*., 2011). Due to their metal leaching potential, these wastes are widely classified as hazardous wastes under the US Environmental Protection Agency (USEPA) Classification (St-Pierre and Piron, 1990). Categorization as hazardous wastes greatly increases the cost of the disposal of these wastes, owing to the necessary process for making the wastes non-

hazardous, as well as higher transportation and storage costs (Youcai and Stanforth, 2000).

These hazardous wastes containing zinc can be treated by acidic leaching processes. In general, sulphuric acid is used, if zinc is recycled as electrolytic metal in the latter part of the process (Mukongo *et al*., 2009; Herrero *et al*., 2010). However, besides leachable zinc, elements such as Pb, Ca, Fe, Mg, Al, Cu, etc. may also dissolve in the acid (Swarnkar *et al*, 1996; Youcai and Stanforth, 2001). As a result, a large quantity of extra acid will be consumed and a leach solution with complex components will be obtained.

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Alkaline leaching in caustic soda solution is another process frequently used in the recovery of zinc from oxidized zinc ores and wastes (De Carvalho *et al*, 2010; Gurmen and Emre, 2003). In this method, Fe, Al, Mg, Ca, etc. are not dissolved in the alkaline solution. Therefore, the consumption of leaching agent is reduced to a minimum value. Zn in the alkaline leach solution can be extracted as metallic powder by electrowinning (Youcai and Stanforth, 2000) with a lower electricity consumption of 2.5–2.7 kW hour/kg, which is less than that for Zn recovery in acidic medium (3.3 kW hour/kg). In the alkaline process, hydrogen evolution on the cathode surface is even more impeded than in the acid process. Accordingly, a current efficiency of 100% can be reached when high current densities are applied (Gurmen and Emre, 2003). This characteristic favors the electrowinning of ultra-fine zinc powder.

A major problem associated with extracting zinc from an alkaline electrolyte prepared from an industrial waste product is the detrimental effect exerted by metallic lead. Separation of lead from zinc in both acidic and alkaline medium has been investigated extensively. In acidic solution, Pb can be precipated quantitatively by the addition of zinc powder but not in alkaline solution. It was proposed in our previous work that lead can be cemented quantitatively without concomitant loss of zinc by sulfide precipitation using sodium sulfide as precipitant (Youcai and Stanforth, 2001). Mackinnon and Brannen (1979) investigated the effects of lead on zinc deposition from acid sulphate electrolytes. Also, they showed that lead had a small positive influence on the CE and exerted a grain-refining effect on the cathodic deposits with changing orientation.

Another problem during the electrowinning of zinc concerns the reduction of grain size, because ultrafine powder exhibits high reactivity for treating harmful pollutants NOx and displacing the metal impurities in the solution(Tjong and Chen, 2004; Vaghayenegar *et al*., 2010) Hence, a great majority of industries employ the milling process, which has increased energy cost. Another possibility consists of addition of various organic and inorganic substances to the electrolyte in order to improve the particle structure (Recendiz *et al*., 2007). Many colloidal additives and organic additives are commonly used (Ivanov *et al*., 2004), but finding new convenient additives remains a significant research.

Besides the presence of additives, many other factors contribute to the type of deposit obtained at a cathode. Winand (1991) investigated nucleation during the coil coating process from the theoretical

standpoint. Thus, he concluded that high current density electrocrystallization was feasible. St-Pierre and Piron (1990) showed the range of current densities necessary to obtain the different deposits. However, further work is needed to investigate the influences of the parameters on deposit quality during the electrowinning of alkaline electrolyte.

The present study was carried out to determine the effects of electrolyte composition, temperature, current density and lead additive on the size distribution and morphology of zinc particles electrowon from an alkaline leaching solution prepared from a waste product. Lead oxide is currently used in zinc electrowinning from alkaline electrolytes, playing the role of reducing the zinc particle size and achieving high CE.

EXPERIMENTAL

Materials

All chemicals were of analytical grade, unless indicated otherwise. The electrolyte used for electrowinning contained 10 – 50 g/L Zn and was prepared by dissolution of the industrial waste product in caustic soda (180 – 250 g/L) and purification of the solution impurities. Lead oxide was added to obtain working solutions with 10-60 mg/L Pb.

Small-Scale Electrowinning

The electrowinning experiments were carried out in the apparatus shown in Fig. 1, and they were performed at atmospheric pressure. A Plexiglass cell having a volume of 1 L and one magnesium cathode flanked by two stainless steel anodes was used. The inter-electrode distance was 3 cm. The samples for analysis were taken at the fixed time intervals of 20, 40 and 60 min. The obtained zinc powder could easily be removed from the electrode and was washed thoroughly with water and then with ethanol. Finally, it was dried at 50 °C and weighed. The current efficiency was then determined by dividing the theoretical current required to produce the deposit by the real current supplied. A series of zinc powders with different size distributions were obtained by changing the experimental parameters.

The temperatures of the electrolyte, the current density, Zn^{2+} concentration, and NaOH concentrations were allowed to vary from 30 to 70 °C, 500 to 1500 A/m², 10 to 40 g/L, and 150 to 300 g/L. Lead oxide was tested as grain refiner in the range of concentrations from 10 to 60 mg/L.

RESULTS AND DISCUSSION

Initial Studies

Theoretical Analysis of Zinc Electrowinning

In the electrowinning of zinc from alkaline electrolyte, the overall cell reaction is:

$$Zn(OH)_4^{2-} = Zn + H_2O + 2OH^- + 1/2O_2 \uparrow$$

According to the following calculation,

$$E = E_{O_2/H_2O} - E_{Zn(OH)_4^{2-}/Zn}$$

the value of E in theory for alkaline media electrowinning is lower than that obtained in the typical acid media electrowinning by approximately 0.5 V. The zinc electrowinning process is a one-electron electrode reaction. Since the calculated electron transport number n is about 1, two electrons can be transported by two consecutive reactions. The reaction mechanism may be:

$$Zn(OH)_4^{2-} \rightarrow Zn(OH)_3^- + OH^-$$

$$Zn(OH)_3^- + e \rightarrow Zn(OH)_2^- + OH^-$$

$$Zn(OH)_2^- \rightarrow Zn(OH)_{ad} + OH^-$$

$$Zn(OH)_{ad} + e \rightarrow Zn + OH^-$$
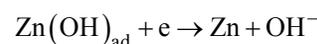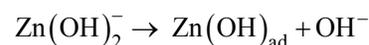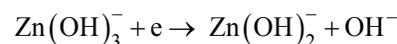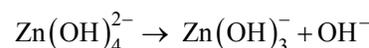
Generally, the zinc electrowinning process is operated under the conditions mentioned in Table 1.

Table 1: Industrial ranges for current density, voltage, electrolyte temperature, zinc and sodium hydrate concentrations and current efficiency in zinc electrowinning process

Parameter	Range
Current density	800-1200 A m^{-2}
Voltage	2.6-3 V
Temperature	30-50 °C
Zinc concentration	30-50 g/L
Sodium hydrate concentration	180-200 g/L
Current efficiency	>99%

Metal Impurities Analysis

EAF dust, BOF sludge and jarosite residue contain various metal impurities such as Co, Ni, Cd, Pb, Al, Cr, Ca, Mg, Fe and Cu. The concentrations of

Figure 1: Schematics of the apparatus for the alkaline zinc electrowinning experiments.
1. Thermostatic bath, 2. Electrolysis cell, 3. Anodes, 4. Cathode, 5. DC power supply, 6. Pump.

Deposit Examination

Zn deposits were examined by laser particle size analyzer to determine the size distribution and by scanning electron microscopy to identify the surface morphology and microstructure.

The samples of the same deposits were also analyzed from liquid state by ICP-OES, so the solid samples must be broken down in an aqua regia-HF solution. All the samples were diluted in order to be in the calibration range.

Electrochemical Studies

Electrochemical studies, such as cathodic polarization and cyclic voltammetry, were performed in a three-electrode Plexiglas cell. High purity aluminum (>99.95%) was used as the working electrode for the cyclic voltammetry and cathodic polarization experiments. A graphite rod was used as counter and a saturated calomel electrode (SCE) as the reference electrode. All potentials were recorded with respect to the SCE. The potential was scanned from – 1.3 to – 1.7 V/SCE for cathodic polarization with a scan rate of 10 mV /s. Cyclic voltammetric experiments were carried out by scanning at a scan rate of 2 mV/s in the potential range of – 0.7 to –2.0 V/SCE.

The electrolyte used in these experiments had the same composition as the electrolyte used during the small-scale electrolysis.

elements (Fe, Ca, Cu, Cd, Mg) in leach solution are all lower than 0.5 g/L. While Cd exerts a deleterious effect on the electrowinning in the acid leach method, the effect of Cd in the alkaline leach solution is found to be negligible. Lead can be separated quantitatively from the alkaline leach solution using sodium sulfide.

Zinc Electrowinning Under Different Conditions

In electrowinning of ultrafine zinc powder, nucleation and growth are in competition to determine grain size. Therefore, a low growth rate and a high nucleation rate for the synthesis of micro-scale materials are unavoidable (Moti *et al*., 2008). In general, factors that result in an increase of overpotential are expected to promote grain refinement.

For the mechanism described above, increasing the nuclear over-potential will facilitate electrowinning of ultrafine zinc powder. The process of zinc powder electrowinning is controlled by mass transport; therefore, we focus on the diffusion over-potential η_d:

$$\eta_d = E_r - E_{ir} = \frac{RT}{ZF} \ln \frac{C_o}{C_s}$$

$$\frac{C_o}{C_s} = 1 - \frac{i_d}{i_{dl}}$$

$$\eta_d = \frac{RT}{ZF} \ln \frac{i_{dl}}{i_{dl} - i_d} = \frac{RT}{ZF} \ln \frac{1}{1 - \frac{i_d}{i_{dl}}}$$

$$i_{dl} = \frac{ZFDC_O}{\delta}$$

$$D = \frac{KT}{6\pi r \eta}$$

where
E_r is the equilibrium potential on the cathode,

E_{ir} is the potential measured in the experiment,
C_o is the concentration of Zn ion at the surface of the electrode,
C_s is the bulk concentration of Zn ion,
i_{dl} is the diffusion limiting current density,
i_d is the apparent cathodic current density,
δ is the thickness of the diffusion layer,
K is the stoichiometric coefficient of Boltzman,
r is the radius of solvation of the ion,
and η is the viscosity of the leach solution.

Based on the above equations, high current density favors the decrease of the grain size, provided other electrowinning conditions are kept constant. It can also be inferred that the ultrafine zinc powder size can be controlled by adjusting the electrolyte temperature, composition, and viscosity at the given current density.

Effect of Zinc Concentration

Scanning electron microscopy (SEM) was used to reveal whether a relationship existed between the Zn ion concentration and zinc electrowinning morphology.

Fig. 2 shows SEM micrographs of zinc electrodeposits produced at 10 to 40 g/L Zn^{2+}, with a current density of 1000 A/m².

It can be seen that decreasing the Zn^{2+} concentration led to a smaller particle size. This can be explained in terms of nucleation overpotential. Because the cathodic deposition of zinc is controlled by mass transport, any decrease in zinc ion concentration during the electrowinning can be assumed to account for an increase in overpotential.

However, decreasing the Zn^{2+} concentration facilitated hydrogen evolution and exerted a deleterious effect on the current efficiency. A CE of 75% was achieved at 10 g/L Zn^{2+}, whereas the CE remained approximately 100% with the Zn^{2+} concentrations of 30 and 40 g/L. The presence of Pb^{2+} (30 – 50 mg/L) was found to increase the CE to about 95-98% in the range of 25 – 30 g/L Zn^{2+}.

(a)   (b)   (c)
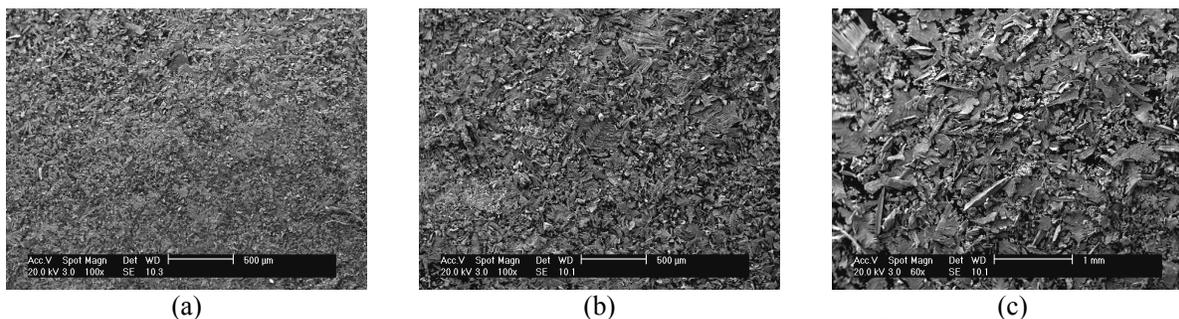
Figure 2: SEM micrographs of the Zn particles synthesized under different Zn^{2+} concentrations of: (a) 10, (b) 25, (c) 40 g/L

Effect of NaOH Concentration

Surface photographs of the deposited zinc showing the effect of NaOH concentration are given in Fig. 3. The electrowon particles are smaller at high NaOH concentration, denoting the beneficial effect of NaOH on the grain refining. The decrease in particle size can be correlated to a corresponding increase in the polarization curves, as shown in Fig. 4.

The polarization curves of zinc deposition recorded under potentiostatic conditions at different NaOH concentrations are shown in Fig. 4. Comparing the curves in Fig. 4, it is obvious that the polarization curve is shifted towards more negative potentials with increasing NaOH, showing an increased polarization.

The change in the overpotential as a result of increasing NaOH concentration can be discussed from two points of view: increase of solution viscosity and increase of $[OH]^-$. (1) High viscosity minimizes the rate of mass transfer, because too much $[OH]^-$ inhibits the discharge rate of zinc, whose electrowinning is diffusion controlled, probably by increasing the viscosity and thus diminishing the diffusion coefficient. (2) The Zn^{2+} ions and the $[OH]^-$ groups could be linked through chelation, thus self-assembling into a chain structure. Moreover, the nucleation and growth of zinc particles depend on the decomposition of $n[Zn(OH)_n]^{n-2}$.

CE values below 180 g/L NaOH were found to be poor; i.e., < 90%. As the NaOH concentration was increased beyond 180 g/L, the CE increased linearly from 90% at 180 g/L NaOH to 100% at 200 g/L NaOH. It was observed that, when the initial NaOH concentration was over 220 g/L, current efficiency of the Zn deposit decreased dramatically as the NaOH concentration increased. This decrease was found to be related to the dissolution of the zinc produced.

Effects of Current Density and Temperature

The optimum range of current density was determined at which the refining of grain size occurred with low energy loss.

Particle size distribution analysis was performed to gain further knowledge of the dependence of ultrafine powder electrowinning on current density. The number of smaller particles present in the mixture and the specific surface area increased with increasing current density, which was consistent with Guillaume *et al*. (2007). This observation may be explained by the easy formation of stable nuclei due to a large number of ions discharged in the cathode. The hydrogen evolution overpotential on the cathode also is high when the current density is high, favoring the discharge of the zinc ions.

| (a) | (b) | (c) |
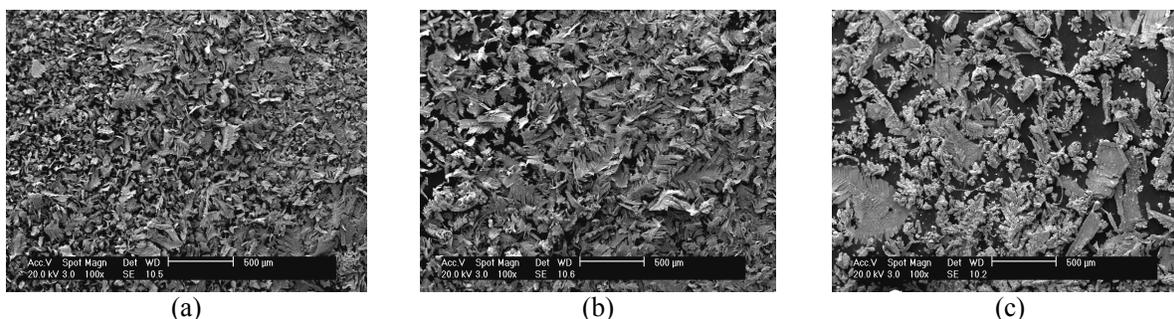
Figure 3: SEM micrographs of zinc particles synthesized at different NaOH concentrations of: (a) 300, (b) 250, (c) 150 g/L
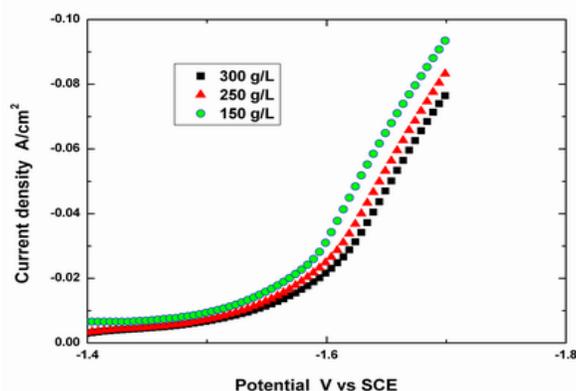
Figure 4: Polarization curves at different concentrations of NaOH

Fig. 5 shows that, the CE of the deposition process was approximately 100% in the range of current density from 600 to1000 A/m^2, but decreased when the current density reached 1000 A/m^2. However, St-Pierre and Pirron (1991) reported that the experimental zinc current efficiency remained high – almost 100% at high current density (1000 – 2000 A/m^2). This difference could be the result of a short period of removing the zinc powder from the cathode in this research.
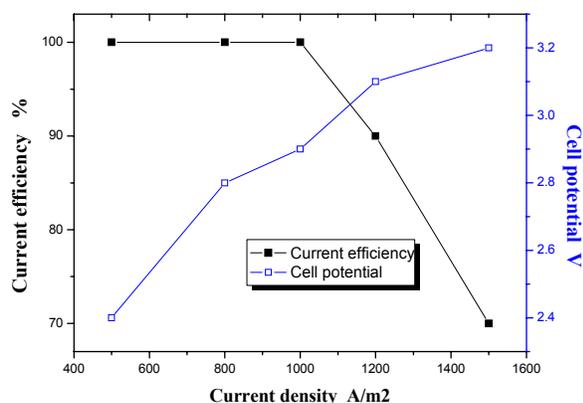
Figure 5: Effect of current density on cell potential and current efficiency

The linear trend of the cell potential versus current density from 500 A/m^2 to 1500 A/m^2 is shown in Fig. 5. Clearly, the deleterious effect of increasing current density on the cell potential is proved.

A low temperature is also anticipated to promote grain refinement. A decrease in the temperature is expected to result in a higher overpotential, which should increase the nucleation rate. The cathodic potentiodynamic polarization curves for zinc electrowinning from alkaline solution at different temperatures are presented in Fig. 6. The evident increase in the cathode overpotential is dependent on the decreased mass transfer rate at the low temperature.
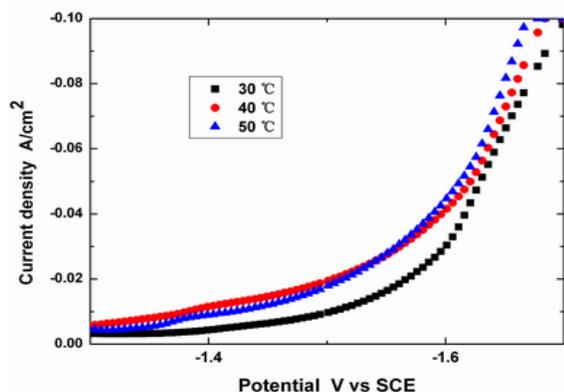
Figure 6: Polarization curves at different temperatures

There is no evident linear relationship between temperature and current efficiency. It was found that the CE drops off much more rapidly at temperatures higher than 50 °C. The reason for this decrease is due to the lower hydrogen overvoltage. On decreasing the temperature to lower than 35 °C, a sharp decrease in the CE by 10% was seen, and this phenomenon could be attributed to the growth in the cell potential.

Effect of Lead Additive

In previous studies, lead was known to impede hydrogen evolution and prevent the CE from decreasing (Ichino *et al*, 1996). In this work, lead was added to the electrolyte to function as a grain refiner. Particle size distribution analysis of electrowinning with 60 mg/L Pb^{2+} showed that the most represented fraction is +4 to 7 μm; however, the fraction + 0.6 to 4 μm is also not negligible. In addition, 90% of the zinc particles have a grain size below 9.04 μm.

The mean particle sizes achieved in the tests are shown in Fig. 7. The data compiled in Fig. 7 indicate that lead addition decreased the absolute diameter of the nuclei formed on the Mg plate. This result was ascribed to the nucleation overpotential with the addition of lead.
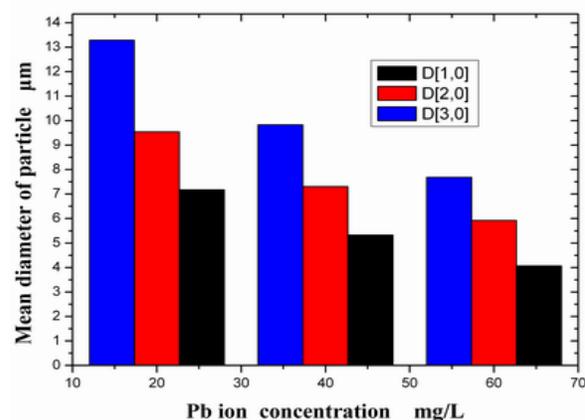
Figure 7: Effect of lead addition on the zinc particle size. D [1, 0] mean diameter by length, D [2, 0] mean diameter by surface, D [3, 0] mean diameter by volume.

This effect can be attributed to the decrease in the rate of a few reactions involved in the zinc electrowinning in the presence of Pb^{2+}. The major inhibitory effect of Pb^{2+} accounts for the higher electrode polarization necessary for micro-scale zinc particle electrowinning to take place.

As shown in Table 2, Pb^{2+} has a negligible effect on the CE of electrowinning in the alkaline electrolyte due to the fact that lead exhibits high hydrogen overvoltages. This result is consistent with that obtained in the typical acid process. The cathodic contamination by lead is counteracted by the diffusion limiting current density. During the electrowinning process, diffusion control is rapidly achieved and the Zn^{2+} electrowinning, which is not controlled by the limiting current, tends to be predominant. This mechanism can be explained by the following equation:

$$\omega(Pb) = \frac{i_{dl}(Pb)\,M(Pb)}{i(Zn)\,M(Zn)\,CE}$$

where
M (Pb) is the molar mass of lead,
M (Zn) is the molar mass of zinc,
CE is the current efficiency of electrowinning.

Table 2: Effects of Pb^{2+} on current efficiency, cathodic contamination, cell potential and particle size.

Pb^{2+} (mg)	Size (µm)	STD (µm)	Current efficiency (%)	Cathodic contamination (%)	Cell potential (V)
20	6.3	7.17	99.10	0.06	2.55-2.65
40	5	5.33	98.88	0.12	2.68-2.78
60	4.3	4.07	98.76	0.18	2.7-2.85

The cyclic voltammetric curves for zinc electrowinning from alkaline solution containing 25 g/L Zn are depicted in Fig. 8. The BCD region corresponds to the electrowinning process, whereas the BEA region concerns the anodic dissolution of previously obtained zinc.
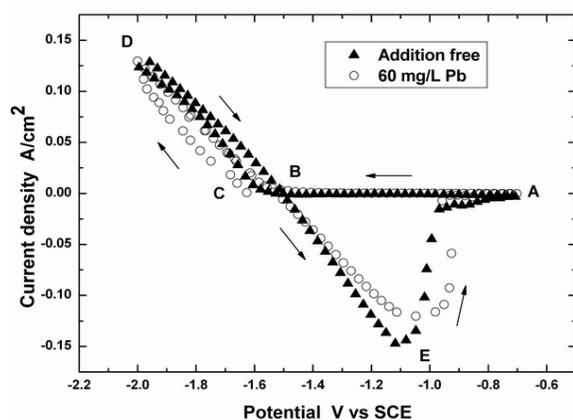
Figure 8: Cyclic voltametry curves recorded for zinc in 200 g/L NaOH in the presence of lead

With the addition of Pb, the BCD region reflects a larger nucleation overpotential than the case without Pb, confirming the size distribution analysis. In the reverse scan, the anodic peak is shifted toward more positive potential, showing the more noble character of the zinc obtained due to the presence of Pb. Hence, Pb was found to inhibit the process of zinc dissolution under the conditions studied.

CONCLUSION

Optimum conditions for zinc electrolysis in alkaline solution were determined as:
- Current density 1000 to 1200 A/m^2
- Electrolyte temperature between 30 and 40 °C
- Zn concentration 25 to 35 g/L
- NaOH concentration 200 to 220 g/L
- Lead level between 20 and 40 mg/L.

A zinc electrolysis current efficiency higher than 97% and a size distribution less than 10 µm were achieved under the above conditions. The negative effect of Pb on the purity of zinc powder was counteracted by the high current density and low amount of additive and high purity zinc powders (99.5 - 99.8 % Zn) were obtained.

ACKNOWLEDGMENTS

NOMENCLATURE

BOF	basic oxygen furnace	
Co	the concentration of Zn ion at the surface of the electrode	kg/m^3
Cs	the bulk concentration of Zn ion	kg/m^3
CE	current efficiency	%
D	diffusion coefficient	m^2/s
E	decomposition voltage	Volt
E_{ir}	the potential measured in the experiment	Volt
$E_{O2/H2O}$	the equilibrium potential of O_2	Volt
E_r	the equilibrium potential on the cathode	Volt
$E_{Zn(OH)_4^{2-}/Zn}$	the equilibrium potential of $Zn(OH)_4^{2-}$	Volt
EAF	electric arc furnace	

F	Faraday's constant	Coulomb
i_d	the apparent cathodic current density	Ampere
i_{dl}	the diffusion limiting current density	Ampere
K	the stoichiometric coefficient of Boltzman	J/K
M	molar mass	g/mol
R	gas constant	J/(mol*K)
r	the radius of ionic solvation	m
STD	standard deviation	
T	temperature	K
Z	number of moles	Moles

Greek Letters

δ	the thickness of the diffusion layer	m
η_d	diffusion over-potential	Volt
η	the kinetic viscosity of the leach solution	$N{\cdot}s/m^2$
ω	mass percent	%
γ	activity coefficient	

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