



The effect of lead and some operating parameters on cathode contamination during zinc electrowinning



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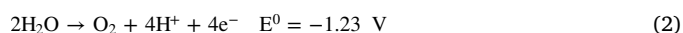
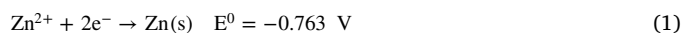
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ABSTRACT

The influence of various operating parameters such as lead concentrations as impurity, current density, acid concentration and temperature was investigated in acid zinc sulfate electrolyte. Nucleation overpotential (NOP) for various experimental conditions was determined using cyclic voltammetry technique. Scanning electron microscopy (SEM), X-ray diffraction (XRD) and Inductively coupled plasma (ICP) were employed to study the physical and chemical characteristics of zinc deposits. By adding lead to the electrolyte, the current efficiency (CE) was changed from 94.04% for the electrolyte without lead to 95.37% for the solution containing 0.2 mg L^{-1} Pb (1.33% increase). However, in the presence of higher lead concentrations ($0.2\text{--}0.8 \text{ mg L}^{-1}$), a gradual improvement of CE (0.94%) was found. The results revealed an increase of NOP up to 25 mV with the increase of lead concentration up to 0.8 mg L^{-1} in the electrolyte while the rise of acid concentration and temperature lowered the NOP for zinc deposition for 14 and 9 mV, respectively. The increase of current density from 45 to 60 mA improved CE from 94.59% to 95.41% while that of temperature from 35 to 45 °C, enhanced the CE from 94.63% to 95.64%. However, the increase of acidity (158 to 170 g) lowered the CE from 96.58% to 95.60%. The stirring rate up to 412 rpm did not significantly influence the nucleation overpotential values. However, increasing the scanning rate resulted in an increase of NOP values, indicating nucleation improvement and crystal rearrangement.

1. Introduction

Minimizing the energy consumption is one of the major goals of zinc electrowinning industry. The production of zinc is performed by its extraction from acid zinc sulfate electrolyte via electrolysis using aluminum cathode and lead or lead-silver alloy anodes. During the electrowinning process, zinc is deposited on the cathode and oxygen is evolved on the anode (reactions (1) and (2)). In the competition between the zinc and hydrogen ions reduction on the interface of electrolyte/electrode, the zinc reduction becomes the dominant cathodic reaction. Generally, around 90% of the cathodic current is consumed for the zinc deposition (Scott et al., 1987).



Major parameters affecting the zinc deposition and hence current efficiency and cell voltage are the nature and concentration of impurities, temperature, bath acidity, current density and additive levels. The electrolyte often contain the impurities such as Ni, Co, Sb,

Ge and etc. which are more electropositive than zinc. Impurities could influence the deposition process as follows: i) when the impurity is a more noble metal than zinc, it can be deposited easily on the cathode decreasing the purity of the produced zinc, ii) since the overpotential of hydrogen evolution on deposited impurities is lower than zinc, the impurities could promote the hydrogen evolution in place of zinc deposition leading to a decrease in the current efficiency and thereby increase the energy consumption and internal stress. Impurities could also change the morphology and produce pitted deposits due to redissolution of zinc deposits (Ichino et al., 1995; Zhang et al., 2009). Between several impurities existing in tank house solution, lead is an impurity, which its presence in trace levels is inevitable because lead is dissolved from the zinc ores and lead or lead-silver anodes during electrowinning process. Lead has been reported as an impurity that can show small positive (Ault and Frazer, 1988; Frazer, 1988; Ivanov, 2004) or negative (MacKinnon et al., 1979) effects on current efficiency (CE) depending on the electrolysis conditions, solution purity of added lead and method of the CE measurements (Frazer, 1988). The beneficial effect of lead on CE (the increase of current efficiency during zinc

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deposition) could be attributed to the fact that contrary to majority of impurities, the overpotential of the hydrogen evolution on lead is higher than that of zinc (Kuhn et al., 1972). In another word, when Pb is adsorbed on zinc, it generates lessened activation of hydrogen evolution with increasing cathodic polarization and enhances the formation of H_{ad} on the zinc deposit (Ichino et al., 1995). Therefore, the production of the hydrogen evolution on lead sites is lowered leading to more consumption of the current by zinc deposition reaction and increasing of current efficiency. Frazer (1988) mentioned that the most of lead's beneficial effects must occur at $\leq 2 \text{ mg L}^{-1}$ Pb. He indicated also that the level of lead $[\text{PbCl}_2 \text{ solution}] \leq 5 \text{ mg L}^{-1}$ shows only a small ($\leq 1\%$) beneficial effect on CE. Previous studies on zinc electrowinning have shown a definite correlation between morphology the nature and concentration of impurities (MacKinnon et al., 1979; MacKinnon and Brannen, 1977; Lamping and O'Keefe, 1976). MacKinnon and Brannen (1977) showed that the zinc morphology and crystalline orientation are extremely sensitive to lead concentrations as low as 1 mg L^{-1} . MacKinnon et al. (1979) examined the effect of Pb contamination under chemical form of lead acetate (0, 1, 3 and 9 mg L^{-1}), lead sulfate (3 and 9 mg L^{-1}) and lead oxide (3 and 9 mg L^{-1}), on the morphology and orientation of zinc deposits. They reported that the morphology and orientations of zinc deposits are dependent on the lead content present on deposit.

Although, the influence of impurities and additives has been a popular subject studied by many researchers for several years, the direct effect of parameters such as temperature, current density and acid concentration were less discussed in literature. Scott et al. (1988) are between the researchers who investigated the influence of temperature, zinc and acid concentration on current efficiency for a zinc electrowinning cell using a mathematical model. They developed and validated their model for both steady state and dynamic simulations. Alfantazi and Dreisinger (2001) and Guillaume et al. (2007) were also studied the zinc/acid concentrations on the current efficiency, energy consumption and morphology of metallic zinc. Alfantazi and Dreisinger (2001) found an increase of the current efficiency with the decrease of acid concentration. They also reported a decrease in the percentage of ohmic drop with the increase of acid concentration. Guillaume et al. (2007) showed a decrease in zinc deposition yield with the increase of acid concentration. The deposition time and acid concentration was examined using an analytical electrochemical cell based on the Hull cell by McCollm and Evans (2001). They found some decrease in current efficiency with increasing of acid concentration. The effect of temperature and current density was studied on cathodic current efficiency, specific energy consumption and physical properties of the zinc deposits in the presence of $[\text{BMIM}]\text{H}_2\text{SO}_4$ as additive by Zhang et al. (2009). They reported an increase of the current efficiency and decrease of specific energy consumption with increasing of temperature.

Electrochemical techniques such as cyclic voltammetry, polarization curves and impedance measurements accompanied with current efficiency calculations, morphology and crystallographic orientation studies are between the most common methods used to investigate the zinc electrodeposition during electrowinning.

The aim of this work is to study the lead behavior at very low concentrations as function of temperature, current density and sulfuric acid concentration on zinc contamination. In order to study the effect of lead on zinc deposit contamination, Pb^{2+} concentration was varied between 0.05 up to 0.8 mg L^{-1} in the electrolyte, which corresponds to the operational practice of certain zinc electrowinning conditions. To our knowledge, this is the first time that the contamination of cathode during zinc electrowinning is deeply examined in the presence of several trace levels of lead ($< 1 \text{ mg L}^{-1}$). The influence of three variables of temperature, current density and acidity on cathodic zinc deposit was investigated in the presence of $0.15 \text{ mg L}^{-1} \text{ Pb}^{2+}$ in the bath.

2. Experimental

2.1. Electrolyte composition and electrolysis parameters

The basic electrolyte (BE) contained $60 \text{ g L}^{-1} \text{ Zn}^{2+}$ and $170 \text{ g L}^{-1} \text{ H}_2\text{SO}_4$. Lead was added to the electrolyte in several concentrations of $0.05, 0.1, 0.15, 0.2, 0.4, 0.8 \text{ mg L}^{-1}$ as lead acetate ($\text{Pb}(\text{CH}_3\text{COO})_2$). All chemicals were supplied from Lab Mat–Canada. The electrodeposition was conducted using standard solution (basic solution with 0.15 mg L^{-1} lead ion) at 40°C under 52.5 mA cm^{-2} during 2 h. In order to investigate the influence of current density, acidity and temperature, these parameters were varied as follows: a) the current density was changed from 45 to 50 and 60 mA cm^{-2} , b) three concentrations of sulfuric acid of 158, 165 and 170 g L^{-1} were studied, c) the electrolyte temperature was varied from 35 to 40 and 45°C and maintained by employing a water thermostated double-wall cell. All experiments were performed using a standard electrolyte (STD) containing $60 \text{ g L}^{-1} \text{ Zn}^{2+}$, $170 \text{ g L}^{-1} \text{ H}_2\text{SO}_4$ and $0.15 \text{ mg L}^{-1} \text{ Pb}^{2+}$. Three electrodes including Pt as auxiliary, Al as working and silver chloride $[\text{Ag}, \text{AgCl}/\text{KCl}_{sat}; 0.197 \text{ V (SHE)}]$ as reference electrodes were mounted in a set-up using a Teflon holder to perform electrochemical tests. Both cathode and anode were casted in polyester resin with an exposed surface area of 1 cm^2 . Before each electrochemical experiment, the surface of the working electrode was manually polished with 240 and 600 SiC papers. The electrode was then rinsed by distilled water and ethanol and dried by a drier. Each test was performed at least two times. The close duplicates were considered for each experimental condition. The standard deviation in duplicates or triplicates of measured voltages, current efficiencies and lead contamination values are given as example in Table 2.

2.2. Electrochemical methods

Galvanostatic tests were conducted at several current densities of 45, 50, 52.5 and 60 mA cm^{-2} during 2 h. The solution was magnetically stirred at 60 rpm. A GAMRY® PC4/300 potentiostat was used to log potential and current values. The current efficiency (CE) was calculated according to the following equation:

$$\text{CE}(\%) = \frac{WFn}{ItM} \times 100 \quad (3)$$

where W presents deposit weight (g), F is the Faraday number (C mol^{-1}), n is the electron number, M is the molecular weight (g mol^{-1}), I is the applied current (A) and t is the deposition time (s).

The potentiodynamic polarization tests were performed over a range of potential (-1.0 to -1.3 V) using a potential sweep rate of 10 mV s^{-1} under atmospheric condition, with slight agitation rate of 60 rpm. Cyclic voltammograms were carried out over a wide range of potential (-1.3 to -0.7 V) at 40°C in atmospheric conditions without agitation using a potential sweep rate of 10 mV s^{-1} . The voltammograms were initiated at a potential of -1.30 V , scanned in the positive direction, and then reversed at -0.70 V in the negative direction. For the studies of stirring rate effect on Zn nucleation using cyclic voltammetry technique, the solution was stirred at 60, 100 and 412 rpm. In the case of the scanning rate studies, various scan rates of 1, 5 and 10 mV s^{-1} were considered. Both potentiodynamic polarization and voltammetry measurements were conducted using a potentiostat (Gamry Reference 3000-GAMRY USA).

2.3. Deposit examination

The surface characterization was performed using scanning electron microscopy (SEM) using JEOL JSM-840a. A Siemens D5000 diffractometer using monochrome CuK_α radiation at 40 kV and 30 mA was used for X-ray diffraction characterization with different angles varied up to 80° at 0.02° per 1.2 s. Lead content of the zinc deposits was

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