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# Electrochemical studies of ionic liquid additives during the zinc electrowinning process

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#### ABSTRACT

1-Ethyl-3-methylimidazolium methanesulfonate ([EMIM]MSO<sub>3</sub>) and 1-butyl-3 methylimidazolium bromide ([BMIM]Br) were evaluated individually as additives compared to gelatin and a previously studied additive ([BMIM]Cl) in zinc electrowinning from a synthetic acidic sulfate electrolyte containing 8 g dm<sup>-3</sup> of Mn<sup>2+</sup> ions. A metallic impurity of 0.0055 mg dm<sup>-3</sup> of Sb<sup>3+</sup> ions was examined in combination with 1 and 3 mg of each additive. Galvanostatic measurements have been employed to investigate the cathodic and anodic potentials individually and also the current efficiency of the deposited zinc metal in the acidic sulfate electrolyte for 2 h at 50 mA cm<sup>-2</sup> and 38 °C. The effect of each additive on surface morphology and crystallographic orientation was studied using scanning electron microscopy (SEM) and X-ray diffraction (XRD), respectively. Lead impurities in the deposit have been measured by using an inductively coupled plasma (ICP). Among five different additive concentrations (1, 3, 5, 10 and 40 mg dm $^{-3}$ ) tested, results revealed that the addition of 1 and 3 mg dm $^{-3}$  of [EMIM]MSO<sub>3</sub> reduced the cell voltage by  $\approx$  15 and 10 mV, respectively, while [BMIM]Br at 1 and 3 mg dm<sup>-3</sup> reduced the cell voltage by  $\approx$  10 and 5 mV, respectively. Current efficiencies of 93.6%–94.4% have been obtained by adding 1–3 mg dm<sup>-3</sup> of [EMIM]MSO<sub>3</sub> or 1–3 mg dm<sup>-3</sup> of [BMIM]Br as compared to 92.8% obtained from the standard electrolyte. Maximum reduction of power consumption of  $\approx$  165 kW h ton<sup>-1</sup> was obtained from adding 3 mg dm<sup>-3</sup> of [EMIM]MSO<sub>3</sub> in the presence of Sb<sup>3+</sup> ions followed by a reduction of  $\approx$  154 kW h ton<sup>-1</sup> by adding 3 mg dm<sup>-3</sup> of [BMIM]Br. Potentiodynamic polarization and voltammetric studies indicate that polarization for zinc electrodeposition decreased in the presence of antimony. Apparently, the two additives have approximately similar polarization behaviors on the aluminum electrode in the acidic sulfate electrolyte.

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#### 1. Introduction

Zinc is considered as the fourth most widely used metal, following iron, aluminum and copper (Porter, 1991). Zinc is processed by many methods in order to obtain the metal in high pure state; among these methods, the electrowinning process is the most commonly used (Bodsworth, 1994). Electrowinning uses an electrolytic cell to reduce the zinc on an aluminum cathode and electric current is run through a lead anode. During the electrolysis of a zinc sulfate electrolyte, two main reactions are competing on the cathode: one is zinc reduction, and the other is hydrogen evolution reaction (HER) (Cachet and Wiart, 1990). On the anode, oxygen gas is produced through the overall electrochemical reaction;  $H_2O \rightarrow 2 H^+ + 2e^- + \frac{1}{2}O_{2(g)}E^o = 1.229 V$ . Approximately, 99% of the anodic current is used for oxygen evolution reaction (OER), consuming  $\approx 40\%$  of total cell voltage (Scott et al., 1987).

As far as lead anodes are used in electrowinning this process is very sensitive to the detrimental effect of Pb impurities and to the presented metallic impurities in the electrolyte such as: Sb, Fe, Cu, Co, and Ni. Low

\* Corresponding author. *E-mail address:* nabil.sorour.1@ulaval.ca (N. Sorour). concentrations of these impurities substantially negatively affect the zinc deposition process. This leads to a decrease of zinc current efficiency (CE), change in deposit morphology, cathodic polarization and even anodic polarization (Muresan et al., 1996). Although the steps used for purification are costly, the zinc electrolyte is usually contaminated with many metal ion impurities (Mackinnon and Brannen, 1977). Zinc deposits contaminated with lead were found to have characteristic morphologies and orientations as well as negative effect on current efficiency. The overpotential which depends on the amount of lead present in the zinc deposits and to the presence of other impurities such as antimony and nickel in the deposits cannot be neglected (Mackinnon et al., 1979). One of the considerable goals in zinc electrowinning is minimizing the power consumption. The two important factors which can determine the energy requirements are the current efficiency and cell voltage which are affected negatively by the presence of impurities (Alfantazi and Dreisinger, 2001). Accordingly, additives are used to reduce the negative effect on current efficiency, cell voltage and deposit morphology through their adsorption on the surface of the electrode (Mackinnon and Brannen, 1977).

Natural products and surfactants have always been the focus of attention as additives in the zinc electrowinning process. Among these







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**Table 1** Effect of gelatin, [EMIM]MSO<sub>3</sub> and [BMIM]Br on CE and PC in the absence and presence of Sb(III) during zinc electrodeposition for 2 h at 50 mA cm<sup>-2</sup>.

Additive/mg dm <sup>-3</sup>	Sb(III)/mg dm <sup>-3</sup>	Cell voltage/ V	CE/ %	PC/ kW h ton <sup><math>-1</math></sup>
		$\pm 0.07\%$	$\pm 0.1\%$	$\pm 0.003\%$
SE				
0	0	2.898	92.8	2560
0	0.0055	2.873	88.7	2655
Gelatin				
1	0	2.904	91.1	2613
3	0	2.909	90.6	2632
5	0	2.922	89.1	2688
10	0	2.928	85.3	2814
40	0	2.937	77.5	3107
1	0.0055	2.885	94.1	2513
3	0.0055	2.895	93.7	2533
[EMIM]MSO3				
1	0	2.883	93.9	2518
3	0	2.888	94.4	2508
5	0	2.900	93.8	2534
10	0	2.904	93.1	2557
40	0	2.930	91.6	2622
1	0.0055	2.870	93.9	2505
3	0.0055	2.873	94.6	2490
[BMIM]Br				
1	0	2.888	93.6	2529
3	0	2.893	94.3	2515
5	0	2.915	92.8	2575
10	0	2.928	92.4	2598
40	0	2.938	90.7	2655
1	0.0055	2.877	93.8	2514
3	0.0055	2.880	94.4	2501

additives, animal glues and Arabic gums showed a positive influence on the CE and deposit orientation in the presence of traces of Sb<sup>3+</sup> ions in the industrial electrolyte (Kerby et al., 1977; Mackinnon et al., 1990a, 1990b). The effects of saponin alone and in combination with antimony and glue have been investigated; saponin alone decreased the CE and was weakly polarized but in combination with glue + antimony at low concentrations resulted in an increase in CE (Mackinnon et al., 1990a, 1990b). Addition of sodium lignin sulfonate alone into the industrial electrolyte at a range of 3–10 ppm had no negative impact on CE, nor on the zinc electrowinning process (Alfantazi and Dreisinger, 2003). Also many organic additives have been deeply studied in the zinc electrowinning process; Zhang et al. (2009a, 2009b) have reported the beneficial effect of triethyl benzyl ammonium chloride (TEBACI) and malonic acid on CE and cell voltage in the presence of Ni as impurity. Quaternary ammonium bromides in forms of cetyltrimethylammonium bromide (CTABr) and tetrabutyl ammonium bromide (TBABr) were studied by Tripathy et al. (1999), as they increased the CE and reduced the power consumption in the presence of antimony. Mathieu et al. (1988) have investigated the effect of 2-butyene-1,4-diol and reported its significant effect on improving current efficiency.  $Al_2(SO_4)_3$  and the horse-chestnut tree extract (HCE) showed their beneficial effects on the deposit quality, being good leveling agents (Muresan et al., 1996).

Recently, ionic liquids have been used in many chemical and hydrometallurgical applications due to their chemical and physical properties, as they are salts where the ions are poorly coordinated, leading to being liquids below the boiling point and even at room temperature (Wasserscheid and Keim, 2000). Ionic liquids consist of an organic cation and inorganic or organic anion; they have a wide range of solubility and miscibility. For example, some of them are hydrophobic while others are hydrophilic; most of them are non-flammable and nontoxic (Johnson, 2007; Singh and Kumar, 2008), Jonic liquids are widely used in liquid-liquid extraction and electrodeposition of some metals due to their low melting point and the thermal degradation properties which are important in the electrochemical media (Rout et al., 2013). Also, as a medium in the electrodeposition of aluminum on a stainless steel cathode (Yue et al., 2009), they are used as an organic solvent in electroplating of a range of metals impossible to deposit in water due to hydrolysis e.g. Al, Ti, Ta, Nb, Mo, and W (Endres et al., 2008). Ionic liquids in the form of 1-butyl-3-methylimidazolium hydrogen sulfate ([BMIM]HSO<sub>4</sub>) showed their effects on the kinetics of oxygen evolution as an additive during the zinc electrowinning process (Zhang and Hua, 2011). [BMIM]HSO<sub>4</sub> is found to have good influence in increasing current efficiency, reducing power consumption and producing smooth and compact zinc deposits similar to that obtained from gelatin (Zhang and Hua, 2009). In other previous studies (Sorour et al., 2015; Hunt et al., 2006), the influence of the ionic liquid salt 1-butyl-3methylimidazolium chloride ([BMIM]Cl) as an additive and its electrochemical activity have been studied and its good ionic conductivity and effect in reducing power consumption have been showed. This study investigates the effect of two different ionic liquid salts, 1-ethyl-3-methylimidazolium methanesulfonate ([EMIM]MSO<sub>3</sub>) and 1-butyl-3-methylimidazolium bromide ([BMIM]Br) as additives compared to gelatin and ([BMIM]Cl) on CE, cell voltage, morphology and electrochemical activity during the electrowinning process. The current ionic liquid additives are chosen in this research paper in order to study the effect of a different anion (Br<sup>-</sup>) compared to the previously studied one (Cl<sup>-</sup>) presented in [BMIM]Cl (Sorour et al., 2015), and also to examine the effect of a different ionic liquid ([EMIM]MSO<sub>3</sub>) which could show a good performance among the examined additives.



Fig. 1. Effects of gelatin, [EMIM]MSO<sub>3</sub> and [BMIM]Br on CE: (a) in the absence of Sb(III) and (b) in the presence of 0.0055 mg of Sb(III) during zinc electrodeposition for 2 h at 50 mA cm<sup>-2</sup>.

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