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ORIGINAL PAPER

Effects of [HMIM]HSO$_4$ and [OMIM]HSO$_4$ on the electrodeposition of zinc from sulfate electrolytes

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Abstract The effects of the organic additives 1-hexyl-3-methylimidazolium hydrogen sulfate ([HMIM]HSO$_4$) and 1-octyl-3-methylimidazolium hydrogen sulfate ([OMIM]HSO$_4$) on current efficiency (CE), power consumption (PC), polarization behavior of the cathode, deposit morphology, and crystallographic orientation during electrodeposition of zinc from acidic sulfate solution were investigated. The results were compared with those of a common industrial additive, gum arabic. Addition of these additives increases current efficiency, decreases power consumption, and improves the surface morphology at lower concentrations. Both the additives showed similar polarization behavior to gum arabic and the extent of polarization was in the order: gum arabic > [OMIM]HSO$_4$ > [HMIM]HSO$_4$. The nature of the electrode reactions was studied through measurements of Tafel slopes, transfer coefficients, and exchange current densities. Data obtained from X-ray diffractogram revealed that the presence of any of these additives did not change the structure of the electrodeposited zinc but affected the crystallographic orientation of the crystal planes.

Keywords Additives · Zinc deposition · Ionic liquids · Deposit morphology · Crystal orientation

1 Introduction

A contemporary method for zinc production is extraction from acid zinc sulfate electrolyte by electrolysis with aluminum cathodes and anodes made of lead-silver (1%) alloy [1]. However, electrolysis of zinc is extremely sensitive to the presence of certain metallic impurities [2–5] in the electrolyte augmenting simultaneous evolution of hydrogen during zinc ion electroreduction from aqueous solutions. Hydrogen evolution, which is an unwanted reaction during zinc electrodeposition, decreases cathodic current efficiency and increases power consumption, apart from occluding hydrogen into the zinc deposit, thereby increasing internal stress and producing pitted deposits.

To achieve high current efficiency and produce a leveled, smooth, and dense cathodic deposit, additives such as glue and gum arabic are most commonly used. Robinson et al. [6] have found that the general effect of glue was to refine the grain size and caused the basal plane platelets to become aligned perpendicular to the substrate. MacKinnon et al. [7] have reported that the changes in deposit characteristics in the presence of glue were related to the changes in zinc deposition overvoltage. In a separate study [8] they studied the effects of 15 impurities and their interaction with glue on zinc electrowinning from industrial acid sulfate electrolyte and found that the addition of glue to the electrolyte counteracted the detrimental effects of Sb and Ge on the current efficiency of zinc deposition. A decrease in current efficiency along with a reduction in crystallite size during zinc electrodeposition from acidic sulfate solutions in the presence of gum arabic has been reported [9, 10] and generally attributed to a strongly adsorbed additive layer at the cathodic surface. Similar observation was also made by Sato [11]. However, the search for better additives is continuing. Prion et al. [12] demonstrated that 2-butyne-1,4-diol increased the zinc current efficiency and improved the quality of the electrodeposited zinc. Hosny [13] investigated the current

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efficiency of synthetic zinc electrolytes containing additions of nonylphenoloxylethelene and found that this surfactant decreases zinc current efficiency and increases both cell voltage and energy consumption. Karavasteva et al. [14, 15] have reported the influence of the combined addition of nonylphenolpolyethylene glycol, dinaphthylamine-4, 4′-disulfuric acid, and polyethelene glycol on the electrolyte with high concentrations of impurities. They found that specific combinations of these surfactants decrease the negative effect of metal impurities on both the current efficiency and structure of zinc deposits. In addition, Tripathy and Das et al. have investigated the use of 2-picoline, 4-ethylpyridine, and 2-cyanopyridine [9, 10], sodium lauryl sulfate [16], triethylbenzylammonium chloride and cetyltrimethylammonium bromide [17, 18], and perfluorinated carboxylic acids [19] as alternatives to glue or gum arabic to improve the electrodeposition characteristics of zinc from acidic sulfate electrolytes. They observed that, except for 2-picoline, 4-ethylpyridine, and 2-cyanopyridine, which showed a reduction in current efficiency at all concentrations, all other compounds activated the Zn^{2+} electroreduction.

Ionic liquids (ILs) are organic salts that melt below 100 °C. Unlike traditional solvents, ILs are comprised entirely of ions. The interest in ILs stems from their potential as green solvents because of their chemical and thermal stability, nonflammability, very low or negligible vapor pressure, high ionic conductivity, and wide electrochemical potential window [20, 21], which makes them potentially attractive alternatives for volatile organic solvents. Recently, a large number of studies have been reported on the applications of ILs as additives. The majority of these studies have focused on ILs used as lubricant additives [22, 23], organic synthesis and catalytic reactions [24, 25], mobile-phase additives in liquid chromatography [26], and the effect of ILs in the detection of paraoxon [27], etc.

In our previous report [28], 1-butyl-3-methylimidazolium hydrogen sulfate ([BMIM]HSO_4) and its mixture with gelatine had a pronounced inhibiting effect on Zn^{2+} electroreduction and both are found to be efficient as leveling agents. Shi et al. [29] investigated the corrosion inhibition performance of a series of new imidazoline ILs in acidic solution and found that increasing the carbon chain length of the alkyl connecting with $N_{(3)}$ of imidazoline ring increases their inhibition performance. Thus, it was thought worthwhile to study the effect of two newly synthesized imidazoline ILs namely, 1-hexyl-3-methylimidazolium hydrogen sulfate ([HMIM]HSO_4) and 1-octyl-3-methylimidazolium hydrogen sulfate ([OMIM]HSO_4) on the kinetics and mechanism of the cathodic process as well as upon the quality of zinc deposits for zinc deposition from a sulfate electrolyte. The results are compared with data for gum, arabic which is widely used in industrial zinc electrowinning as a leveling agent.

2 Experimental details

2.1 Reagents

Zinc electrolyte was prepared from AnalaR zinc sulfate ($ZnSO_4 \cdot 7H_2O$) and analytical-grade H_2SO_4. Reagent grade gum arabic was obtained from Chemical Reagent Co, Shanghai, China. The 1-hexyl-3-methylimidazolium hydrogen sulfate ([HMIM]HSO_4) and 1-octyl-3-methylimidazolium hydrogen sulfate ([OMIM]HSO_4) were synthesized in the laboratory as mentioned elsewhere [30, 31]. The synthetic products were characterized by infrared (IR) (Bio-Rad FTS-40, sample dispersed between KBr plate) and nuclear magnetic resonance (NMR) (Bruker Avance DPX-300) spectroscopy as described below.

2.1.1 IR spectroscopy

The IR spectroscopic study was used to investigate the purity of the synthesized additives. The results are listed below:

(i) [HMIM]HSO_4—IR (KBr): v_{max}/cm^{-1} 3149.3 (s, aromatic C–H stretch), 2939.1 and 2863.9 (m, aliphatic C–H stretch), 1562.1 and 1457.9 (s, C=C stretch), 1168.7 (s, C–N stretch), and 1051.0 (s, HSO_4 stretch).

(ii) [OMIM]HSO_4—IR (KBr): v_{max}/cm^{-1} 3139.7 (s, aromatic C–H stretch), 2923.7 and 2854.3 (m, aliphatic C–H stretch), 1560.2 and 1457.9 (s, C=C stretch), 1178.3 (s, C–N stretch) and 1052.9 (s, HSO_4 stretch).

2.1.2 NMR spectroscopy

H NMR spectroscopy helps in analyzing the type of protons attached to different groups of the additives studied; the corresponding spectral data (300 MHz; $CDCl_3$; Me_4Si) are given in Table 1.

2.2 Electrolysis

Small-scale galvanostatic electrolysis was performed in absence and presence of various amounts of additives by chronopotentiometric measurements, employing a 250 cm^3 plexiglass cell. A pure vertical planar aluminum sheet (>99.95%) and two parallel Pb–Ag (Ag, 1%) plates were used as the cathode and anode, respectively. The inter-electrode distance was 2.5 cm and the working temperature was 40 ± 1 °C. The electrolytic solution contained 55 g dm^{-3} zinc and 150 g dm^{-3} sulfuric acid and the

Table 1 Proton chemical shift (ppm) of synthesized ionic liquids

H	$[HMIM]HSO_4$	$[OMIM]HSO_4$
1H, s, Im	10.83	10.81
1H, s, Im	7.26	7.26
1H, s, Im	7.20	7.26
2H, s, NCH_2–	4.25	4.25
1H, s, NMe	4.07	4.07
1H, s, HSO_4	3.87	3.11
2H, m, NCH_2CH_2–	1.85	1.87
2H, m, $N(CH_2)_2CH_2$–	1.23	1.24
2H, m, $N(CH_2)_3CH_2$–	1.19	1.20
2H, m, $N(CH_2)_4CH_2$–	0.83	1.19
3H, m, NHe	0.80	–
2H, m, $N(CH_2)_5CH_2$–	–	0.82
2H, m, $N(CH_2)_6CH_2$–	–	0.81
3H, m, NOc	–	0.79

specific experimental procedures were similar to as described previously [16]. In all cases, the current density was held constant at 400 A m^{-2} during the deposition time of 2 h. Zinc was deposited on both sides of cathode onto a total area of 4.5 cm^2. After electrolysis, the cathode was removed from the cell and washed thoroughly with distilled water and dried. The current efficiency was calculated by weight according to Faraday's law.

2.3 Electrochemical measurements

All electrochemical measurements were carried out by using a CHI760C electrochemical workstation from Shanghai CH Instruments Company in China.

Electrochemical studies were based on the analysis of cyclic voltammetric measurements and potentiodynamic polarization tests. A conventional three-electrode cell was used for these experiments. All the measurements were preformed at 25 °C under atmospheric condition. The working electrode was made from high-purity (>99.95%) aluminum and sealed with epoxy resin, with an effective area of 0.28 cm^2. A graphite rod was used as counter-electrode, and a saturated calomel electrode (SCE) as the reference electrode. Cyclic voltammetric experiments were carried out by initiating scans at constant scan rate of 10 mV s^{-1} from the initial potential of -0.70 V to the final potential of -1.30 V. Potentiodynamic polarization studies were carried out after the cyclic voltammetric tests; the potential was scanned from about -1.05 V to -1.30 V with constant scan rate of 5 mV s^{-1}. All potentials were recorded with respect to the saturated calomel electrode (SCE). Before each experiment, the working electrode was polished successively with fine-grade Emery papers, degreased with anhydrous alcohol in an ultrasonic bath for 10 min, washed with twice-distilled water, and finally dried.

2.4 Deposit examination

The surface morphology of the deposits was examined by scanning electron microscopy (SEM) using a Tescan VEGA II XMH microscope. Rigaku D/max 2200 X-ray diffractometer was used to examine the sections of the deposits by X-ray diffraction (XRD) to determine their orientations relative to American Society for Testing and Materials (ASTM) standard for zinc powder.

3 Results and discussion

3.1 Cathode current efficiency and power consumption

The effects of $[HMIM]HSO_4$ and $[OMIM]HSO_4$ on current efficiency (CE) and power consumption (PC) during zinc electrodeposition from the acidic sulfate bath were studied in the concentration range 1–50 mg dm^{-3} and the results were compared with those of gum arabic (Table 2). As can be seen, the initial addition of $[HMIM]HSO_4$ and $[OMIM]HSO_4$ improves the CE, but drops steadily at higher

Table 2 Effect of additives on current efficiency during zinc electrodeposition

Additives (mg dm^{-3})	Current efficiency (%)	Cell voltage (V)	Power consumption (kWh t^{-1})
Blank	89.3	2.89	2,655
$[HMIM]HSO_4$			
1	91.2	2.80	2,518
2	92.9	2.88	2,543
5	92.6	2.88	2,551
10	90.1	2.89	2,631
50	82.8	2.90	2,873
$[OMIM]HSO_4$			
1	92.2	2.88	2,562
2	90.9	2.91	2,626
5	88.2	2.92	2,716
10	85.8	2.93	2,801
50	76.7	2.94	3,144
Gum arabic			
1	89.2	2.90	2,667
2	86.5	2.92	2,769
5	83.8	2.96	2,897
10	81.7	2.98	2,992
50	71.8	2.99	3,416

concentrations. This increase can be attributed to suppression of hydrogen evolution [19] and corrosion inhibition on the dissolution of the deposited zinc during zinc electrodeposition by blocking the active sites through cathodic adsorption of additives. In the absence of additive, the CE is ~89% whereas at 2 mg dm^{-3} [HMIM]HSO$_4$ the CE was ~92.9% and then fell to 82.8% at 50 mg dm^{-3}. The observed trend of CE with increasing [OMIM]HSO$_4$ concentration is similar to that for [HMIM]HSO$_4$, where a CE of 92.2% is obtained at 1 mg dm^{-3} but a reduction occurs at higher [OMIM]HSO$_4$ concentrations. It is clear that the CE decreases with initial addition of gum arabic and further decreases at higher concentrations. Similar observation was also made by Das et al. [15], who contrasted the effect of 2-picoline and gum arabic on zinc electrowinning from acidic sulfated electrolyte. The decreases in CE with increasing additives concentrations are well documented [14–19] and have been generally attributed to the blocking of the active sites of the cathode surface by superfluous adsorption of the additives, which results in an increase in the interfacial viscosity, causing a decrease in the diffusivity of zinc ions and slowing the deposition rate, thereby inhibiting electrocrystallization of zinc.

Moreover, it can be seen from Fig. 1 that CE drops off with increasing additive concentrations in the order: gum arabic > [OMIM]HSO$_4$ > [HMIM]HSO$_4$. The order probably reflects increasing adsorption at the electrode surface with increasing molecular size and hence molecular mass [19, 32]. Change in PC followed the opposite order to that of CE (Table 2). Addition of [HMIM]HSO$_4$ at lower concentrations, e.g., 1 mg dm^{-3} reduced PC by ~137 kWh t^{-1}. However, at 50 mg dm^{-3}, extra PC of ~218 kWh t^{-1} was needed for the electrodeposition process. Similar results were obtained with [OMIM]HSO$_4$ (Table 2). However addition of gum arabic even at 1 mg dm^{-3} increased the PC by ~12 kWh t^{-1}, and increasing the concentration of gum arabic further increased the PC during the electrodeposition process.

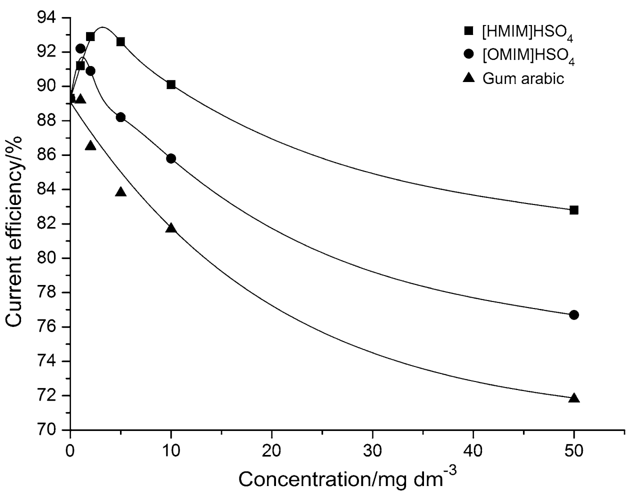

Fig. 1 Effect of additives on current efficiency during zinc electrodeposition

3.2 Polarization studies

The effect of [HMIM]HSO$_4$, [OMIM]HSO$_4$, and gum arabic on electroreduction of Zn^{2+} ion on aluminum electrode from the acidic sulfate bath in the concentration range 1–10 mg dm^{-3} was investigated using cyclic voltammetry and potentiodynamic polarization techniques. The nucleation overpotential (NOP), which is the difference between the nucleation potential (E_{nu}) and the cross overpotential, is discussed in the previous report [28]; the NOP data can be determined from the cyclic voltammograms [16]. NOP is regarded as an indicator of the extent of polarization of a cathode, and high NOP values indicate strong polarization of the cathode [33]. As can be seen in Table 3, the additives were found to have a strong effect on the NOP of zinc electrodeposition on aluminum electrode, which shifted towards more negative potentials with increasing additive concentration in the order: gum arabic > [OMIM]HSO$_4$ > [HMIM]HSO$_4$ (Fig. 2); for example, addition of 5 mg dm^{-3} of [HMIM]HSO$_4$ increased the NOP from 118 to 146 mV. [OMIM]HSO$_4$ alone increased the NOP slightly more than [HMIM]HSO$_4$; addition of 5 mg dm^{-3} increased the NOP from 118 to 171 mV, and the NOP increased to 176 mV with addition of 5 mg dm^{-3} gum arabic. In addition, it is clear that the cathodic process area and the cathodic peak current decrease along with the

Table 3 Effect of additives on nucleation overpotential (NOP) during zinc electrodeposition from acidic sulfate solution

Additives	Concentration (mg dm^{-3})	$-E_{nu}$ (mV)	−NOP (mV)
Blank	0	1,170	118
[HMIM]HSO$_4$	1	1,172	120
	2	1,192	140
	5	1,198	146
	10	1,219	167
[OMIM]HSO$_4$	1	1,173	121
	2	1,197	145
	5	1,223	171
	10	1,234	182
Gum arabic	1	1,181	129
	2	1,199	147
	5	1,228	176
	10	1,245	193

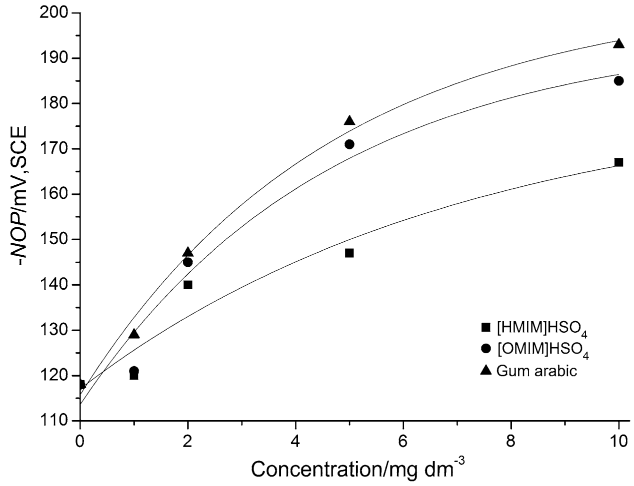

Fig. 2 Effect of additive concentration on nucleation overpotential

increase in NOP, denoting an inhibition of electrocrystallization (Fig. 3). This is generally attributed to the strong adsorption of the additives on the surface of the electrode. The extent of absorption appears to be in the order: gum arabic > [OMIM]HSO_4 > [HMIM]HSO_4. The order reflects the adsorbability of the additives studied and is consistent with their effect on the deposition process (Table 2). This can also be concluded from the influence of the additives on the NOP and the cathodic electroreduce process.

The cathodic linear sweep polarization curves for zinc eletrodeposition on aluminum electrode from acidic zinc sulfate solutions in absence and presence of 5 mg dm^{-3} [HMIM]HSO_4, [OMIM]HSO_4, and gum arabic are presented in Fig. 4. It can be seen that the trace addition of these additives to the solution markedly increases the electroreduction potential of Zn^{2+} ion and the polarization is dependent of the nature of the additives. The cathode polarization is highest for gum arabic and lowest for [HMIM]HSO_4, and this result is consistent with the discussion in the previous section.

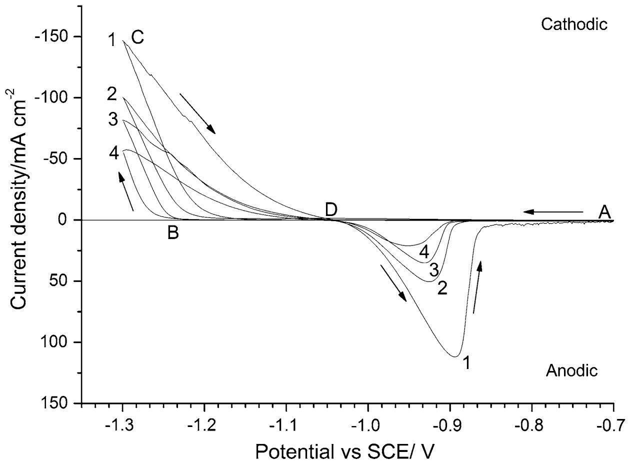

Fig. 3 Cyclic voltammograms of acidic zinc sulfate solutions in the absence and in presence of 5 mg dm^{-3} different additives: (1) blank, (2) [HMIM]HSO_4, (3) [OMIM]HSO_4, and (4) gum arabic

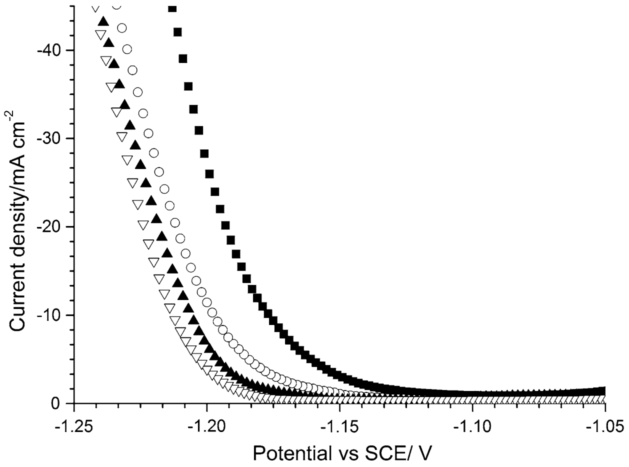

Fig. 4 Effect of different additives on the cathodic polarization during zinc deposition on aluminum: (■) blank, (○) 5 mg dm^{-3} [HMIM]HSO_4, (▲) 5 mg dm^{-3} [OMIM]HSO_4, and (▽) 5 mg dm^{-3} gum arabic

The kinetic parameters Tafel slope, b (mV $decade^{-1}$), transfer coefficient α, and exchange current densities, i_0 (mA cm^{-2}) for zinc electrodeposition on aluminum electrode were calculated from their respective cathodic linear sweep polarization curves using the same method as described earlier [18, 28]. The results are given in Table 4.

Table 4 Effects of additives on the kinetic parameters during zinc electrodeposition from acidic sulfate solution

Additives (mg dm^{-3})	Tafel slope (mV per decade)	Transfer coefficient, α_c	Exchange current density, i_0 (mA cm^{-2} ($\times 10^{-2}$))
Blank	120	0.49	6.5
[HMIM]HSO_4			
1	130	0.45	5.9
2	128	0.46	5.2
5	126	0.47	4.4
10	125	0.47	4.3
[OMIM]HSO_4			
1	126	0.47	5.1
2	123	0.48	3.4
5	129	0.46	3.3
10	128	0.46	2.6
Gum arabic			
1	140	0.42	5.3
2	135	0.44	3.7
5	128	0.46	3.5
10	125	0.47	2.5

It is found that the addition of [HMIM]HSO_4, [OMIM]HSO_4 or gum arabic does not have any significant effect on the Tafel slopes and transfer coefficients, indicating that they do not control the charge transfer reaction and have no effect on the symmetry of the electron transfer reaction, respectively. However, the presence of additives has an inhibiting effect on the kinetics of the zinc discharge process, reflected in the decrease of the i_0 values, which may be attributed to the

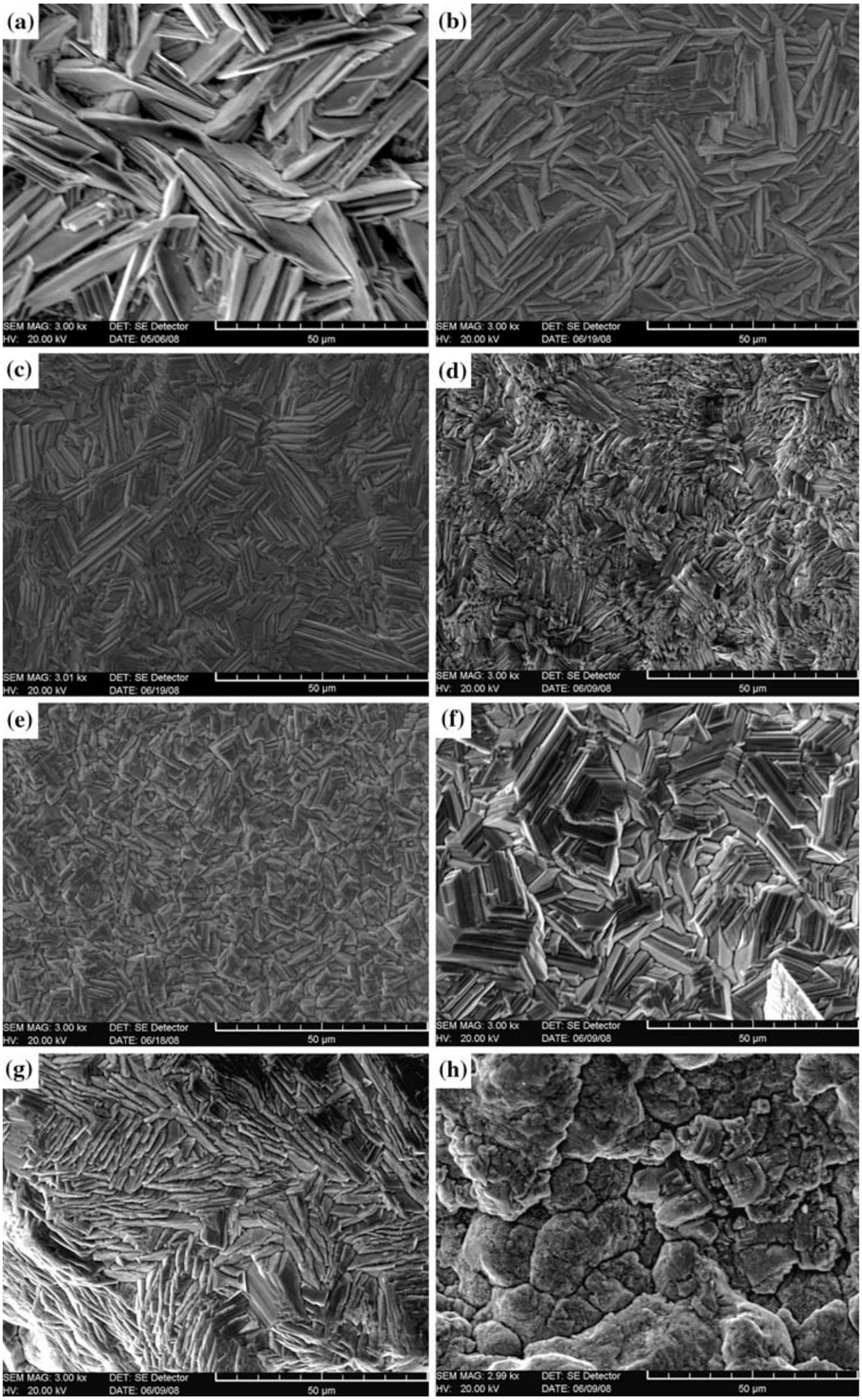

Fig. 5 Scanning electron micrographs of zinc deposits: **a** blank, **b** 1 mg dm^{-3} [HMIM]HSO_4, **c** 10 mg dm^{-3} [HMIM]HSO_4, **d** 1 mg dm^{-3} [OMIM]HSO_4, **e** 10 mg dm^{-3} [OMIM]HSO_4, **f** 1 mg dm^{-3} gum arabic, **g** 5 mg dm^{-3} gum arabic, **h** 10 mg dm^{-3} gum arabic

adsorption of the additives on the cathode surface, thereby lowering the rate of electron transfer reaction. The inhibition enhancement due to increasing additive concentration could be based on the electrostatic cation adsorbate altering the double-layer structure and decreasing the rate of an electrochemical reaction [33].

3.3 Effect of additives on deposit morphology and orientation

The zinc deposits were examined using scanning electron microscopy and X-ray diffraction to determine the surface morphology and crystallographic orientations. Typical SEM photomicrographs are shown in Fig. 5 and the crystallographic orientations of zinc deposits in absence and presence of different concentrations of additives are given in Table 5.

The investigated additives showed marked effect on the surface quality of the zinc electrodeposits. The zinc deposit obtained from addition-free electrolyte is bright but not smooth and consists of hexagonal platelets of moderate size with (101) (201) (100) (112) crystal orientations (Fig. 5a) [28]. Introducing the additives into the solution even at 1 mg dm^{-3} did not affect the shape of the crystals but decreased the size of the platelets. Increase in additives concentrations reduced the platelet sizes further, giving smooth and compact deposits (Fig. 5b–g). However with 10 mg dm^{-3} gum arabic clustered-type deposit was obtained (Fig. 5h). The order of preferred crystal orientations at 1 mg dm^{-3} of each additive resulted in (101) (100) (102) (112) for [HMIM]HSO$_4$, (101) (110) (112) (103) for [OMIM]HSO$_4$, and (101) (103) (100) (002) for gum arabic. With increase in additive concentrations the order of preferred crystal orientation did not change much, and in all cases (101) was found to be the most preferred orientation irrespective of the nature of the additives.

Overall, the deposits obtained with the [HMIM]HSO$_4$ and [OMIM]HSO$_4$ additives were found to be more leveled and compact than those with gum arabic. Such changes were also reflected in the trends seen in CE and PC.

4 Conclusions

The effect of [HMIM]HSO$_4$, [OMIM]HSO$_4$, and gum arabic on zinc electrodeposition from acidic sulfate solutions has been investigated and the conclusions drawn from the results are summarized as follows:

- [HMIM]HSO$_4$ and [OMIM]HSO$_4$ are found to be better additives than gum arabic with respect to their effect on CE, PC, and deposit quality.
- [HMIM]HSO$_4$ is found to be the best additive, reducing the PC by ~137 kWh t^{-1}.
- Although all the additives affected the polarization behavior of the cathode in similar ways, gum arabic is the strongest cathode polarizer.
- All these additives affected the deposit quality by reducing the platelet sizes and hence resulting in smooth and compact morphology. However [HMIM]HSO$_4$ and [OMIM]HSO$_4$ were found to be better than gum arabic.
- All these additives do not change the overall structure of the zinc deposits but affected the preferred crystal orientations. However, (101) plane was found to be the most preferred, independent of the nature of the additives.

Table 5 Crystallographic orientations of zinc deposits in absence and presence of different additives

Additives	Concentration (mg dm^{-3})	Crystallographic orientations (*hkl*) and peak intensity ratio (I/I_{max}), %							
		(002)	(100)	(101)	(102)	(103)	(110)	(112)	(201)
Blank	0	1	13	100	4	1	2	11	20
[HMIM]HSO$_4$	1	7	16	100	10	7	6	10	8
	2	6	20	100	16	5	11	9	5
	5	5	15	100	23	4	4	13	7
	10	7	10	100	39	12	9	17	–
[OMIM]HSO$_4$	1	10	13	100	10	16	42	25	–
	2	11	10	100	11	8	31	37	–
	5	12	10	100	29	14	9	32	–
	10	8	10	100	28	9	6	5	–
Gum arabic	1	15	18	100	12	20	8	9	–
	2	8	7	100	29	28	15	8	–
	5	19	13	100	29	22	8	11	7
	10	16	12	100	7	8	7	7	5

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