



# EFFECT OF IRON ON ZINC ELECTROWINNING USING SULFATE SOLUTIONS<sup>1</sup>

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## Abstract

Electrolytic zinc used in galvanizing processes is obtained using zinc electrowinning from sulfate solutions. The presence of impurities in the electrolyte is a major problem for the zinc electrowinning industry. The impurities on zinc electrolysis can reduce the current efficiency and increase the energy consumption. In this work, the effect of iron on the zinc electrodeposition was studied using galvanostatic deposition and cyclic voltammetry. Contents of 5, 10, and 15 mg.L<sup>-1</sup> of iron were added in the electrolyte of zinc sulfate and in an industrial acid electrolyte. Using the industrial electrolyte, iron addition was detrimental to the zinc electrowinning, increasing the energy consumption and decreasing the current efficiency.

**Key words:** Electrolysis; Electrochemical methods; Energy consumption; Electrolytic zinc.

## EFEITO DO FERRO NA ELETRÓLISE DO ZINCO USANDO SOLUÇÕES SULFATADAS

### Resumo

O zinco eletrolítico usado nos processos de galvanização é obtido através da eletrólise do zinco a partir de soluções sulfatadas. A presença de impurezas no eletrólito é o maior problema para a eletrólise do zinco industrial. As impurezas na eletrólise do zinco podem reduzir a eficiência de corrente e aumentar o consumo de energia. Neste trabalho foi estudado o efeito do ferro na eletrodeposição do zinco, usando deposição galvanostática e voltamétrica cíclica. Foram adicionadas ao eletrólito de sulfato de zinco e a um eletrólito ácido industrial 5, 10 e 15 mg.L<sup>-1</sup> de ferro. No eletrólito ácido industrial a adição de ferro foi prejudicial para a eletrólise do zinco, aumentando o consumo de energia e diminuindo a eficiência de corrente.

**Palavras-chave:** Eletrólise; Métodos eletroquímicos; Consumo de energia; Zinco eletrolítico.

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## 1 INTRODUCTION

One of the greatest changes in the last forty years concerning the application of steel as a structural element was the use of zinc coated steels for situations, which demand a high corrosion resistance. Galvanized steels can be applied in all those industrial segments which normally use cold rolled steels such as the automotive industry, auto parts, metallic vehicle bodies, home appliances, electro-electronic equipment, steel furniture, civil construction and containers.<sup>[1]</sup>

One method of production of electrolytic zinc is electrowinning using sulfate solutions. Current is applied through insoluble electrodes and causes zinc deposition on cathode. Several parameters affect zinc electrolysis and its control.<sup>[2]</sup> The presence of impurities in the electrolyte is a major problem for the zinc electrowinning industry.<sup>[3]</sup> The synergistic interactions among impurities determine the quality of zinc deposit from the solution.<sup>[4]</sup>

Literature about the effect of impurities on zinc electrowinning is still scarce and restrict to specific impurities such as antimony,<sup>[5]</sup> nickel,<sup>[6]</sup> cadmium, iron and copper.<sup>[3]</sup>

A promising development in this area is the application of cyclic voltammetry and of electrochemical impedance spectroscopy to the investigation of the electrodeposition process.<sup>[3,7]</sup>

The aim of this work is to study the effect of iron in the zinc electrowinning process using electrochemical techniques such as galvanostatic deposition and cyclic voltammetry. Two electrolytes were studied: a zinc sulfate electrolyte, pH 5.05, and an acid industrial electrolyte of zinc sulfate with addition of 180 g.L<sup>-1</sup> of H<sub>2</sub>SO<sub>4</sub>. Considerations about the effect of pH and the presence of other species in solutions such as manganese, magnesium and cadmium were done.

## 2 MATERIALS AND METHODS

Two electrolytes of zinc sulfate were used. A solution of 60 g.L<sup>-1</sup> of zinc was prepared dissolving 262.8 g of ZnSO<sub>4</sub>.7H<sub>2</sub>O in 1000 mL of deionized water. Solutions containing 60 g.L<sup>-1</sup> of zinc and 5, 10, and 15 mg.L<sup>-1</sup> of iron was prepared using ZnSO<sub>4</sub>.7H<sub>2</sub>O, FeSO<sub>4</sub>.7H<sub>2</sub>O, and deionized water.

Iron was added in a second electrolyte, used in an industrial plant of zinc electrowinning. Chemical characterization of this industrial acid electrolyte is shown in Table 1. Chemical analysis was performed using the technique of atomic absorption spectrophotometry. Contents of 5 mg.L<sup>-1</sup>, 10 mg.L<sup>-1</sup>, and 15 mg.L<sup>-1</sup> were added in the industrial electrolyte.

**Table 1** – Chemical composition of the industrial electrolyte

Element	Content (g.L <sup>-1</sup> )
Zn	51.59
Fe	8.06 .10 <sup>-4</sup>
Mn	1.36
Mg	7.27
Co	
Cd	0.06

Electrochemical tests of galvanostatic deposition were performed using a potentiostat/galvanostat AUTOLAB PGSTAT 30. Electrochemical cell was of three electrodes, an anode of Pb-Ag (1% wt), a cathode of aluminum and the reference

electrode was Ag/AgCl. Zinc was deposited using galvanostatic deposition, applying a current density of 500 A.m<sup>-2</sup>, which is used in the industrial plant of zinc electrolysis. The deposition time was 3 hours. Superficial area of cathode and anode was 1 cm<sup>2</sup>. Before testing, cathodes were polished with 600-mesh sandpaper, washed in distilled water and dried. The zinc was removed from the cathode surface with a stainless steel spatula. Zinc deposits were weighted in a precision balance for measurements of 0.0001 g.

Applying Faraday law, the theoretical mass of zinc was calculated:<sup>[8]</sup>

$$I = nF \frac{dn_i}{dt} = \frac{dQ}{dt} \quad (1)$$

where I is the electric current, in amperes, n is the moles of electrons, F is the Faraday constant, dni/dt represents the rate of reaction in moles per second, Q is the quantity of electricity or charge in coulombs (C), and t is the time. The theoretical zinc mass calculated was 184.71 mg. The current efficiency can be expressed by the ratio of deposited zinc mass and theoretical zinc mass.

Energy consumption (kWh/ton) was calculated using the expression:

$$\frac{Vm.8.4.10^5}{10.CE} \quad (2)$$

where Vm is the average potential (V), and CE the current efficiency (%).

Cyclic voltammetry tests were performed using a potentiostat/galvanostat AUTOLAB PGSTAT 30. Electrochemical cell was of three electrodes, an anode of aluminum, a counter electrode of platinum and the reference electrode was Ag/AgCl. Superficial area of cathode and anode was 1 cm<sup>2</sup>. The scan rate was 10 mV.s<sup>-1</sup>, and the potential range was from 0 to -1600 mV.

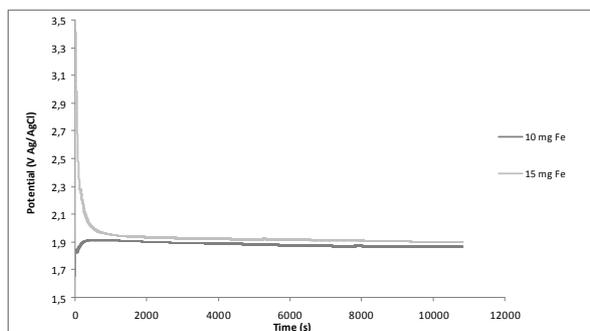
Characterization of the electrodeposited zinc was performed using scanning electron microscopy (SEM), and energy dispersive spectroscopy (EDS). Image analysis using Quantikov<sup>[9]</sup> software was carried out to evaluate porosity of the deposit.

### 3 RESULTS AND DISCUSSION

#### 3.1 Current Efficiency and Energy Consumption

##### 3.1.1 Electrolyte of acid solution

The evolution of potential versus time in an industrial acid electrolyte without iron addition and with addition of 5, 10, and 15 mg.L<sup>-1</sup> Fe during 3 hours of galvanostatic deposition was obtained, and curves of potential versus time for addition of 10, and 15 mg.L<sup>-1</sup> Fe in acid electrolyte are shown in Figure 1.



**Figure 1** -The evolution of potential versus time obtained using an industrial acid electrolyte with addition of 10, and 15 mg.L<sup>-1</sup> Fe during 3 hours of deposition.

Table 2 shows current efficiency and energy consumption obtained in electrolytes without Fe and with additions of 5 mg.L<sup>-1</sup>, 10 mg.L<sup>-1</sup>, and 15 mg.L<sup>-1</sup> of Fe.

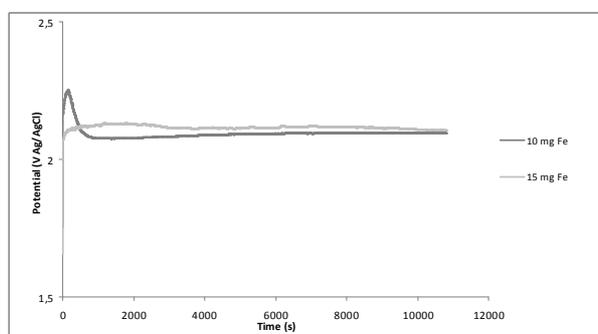
**Table 2** – Current efficiency and energy consumption of zinc electrowinning from acid solution

Acid electrolyte	Potential after 3 hours of electrolysis (mV <sub>Ag/AgCl</sub> )	Electrodeposited zinc mass (mg)	Theoretic zinc mass (mg)	Current efficiency (%)	Energy consumption (KWh/T)
Without Fe	1825.9	654.82	728.05	89.94	1705.28
Addition of 5 mg.L <sup>-1</sup> of Fe	1835.3	638.42	728.05	87.70	1757.89
Addition of 10 mg.L <sup>-1</sup> of Fe	1864.0	641.54	728.05	88.12	1776.86
Addition of 15 mg.L <sup>-1</sup> of Fe	1896.4	640.88	728.05	88.03	1809.55

Iron addition was detrimental to the zinc electrowinning, increasing the energy consumption and decreasing the current efficiency. Among the solutions with iron addition, the addition of 10 mg.L<sup>-1</sup> produced the highest current efficiency.

### 3.1.2 Electrolyte of zinc sulfate

The evolution of potential versus time obtained in zinc sulfate electrolyte without iron addition and with addition of 5, 10, and 15 mg.L<sup>-1</sup> Fe during 3 hours of galvanostatic deposition was obtained, and curves of potential versus time for addition of 10, and 15 mg.L<sup>-1</sup> Fe in acid electrolyte are shown in Figure 2.



**Figure 2** - The evolution of potential versus time obtained using a zinc sulfate electrolyte with addition of 10, and 15 mg.L<sup>-1</sup> Fe during 3 hours of galvanostatic.

The addition of iron in the zinc sulfate electrolyte did not change the zinc mass deposited on cathode, and did not change the current efficiency, as shown in Table 3.

**Table 3** – Current efficiency and energy consumption of zinc electrowinning from zinc sulfate solution

Zinc sulfate electrolyte	Potential after 3 hours of electrolysis (mV <sub>Ag/AgCl</sub> )	Electrodeposited zinc mass (mg)	Theoretic zinc mass (mg)	Current efficiency (%)	Energy consumption (KWh/T)
Without Fe	2068.5	182.42	183.11	99.62	1744.15
Addition of 5	2038.3	182.06	183.11	99.43	1721.96

mg/L of Fe					
Addition of 10	2094.4	182.46	183.11	99.65	1765.49
mg/L of Fe					
Addition of 15	2104.5	182.06	183.11	99.43	1777.91
mg/L of Fe					

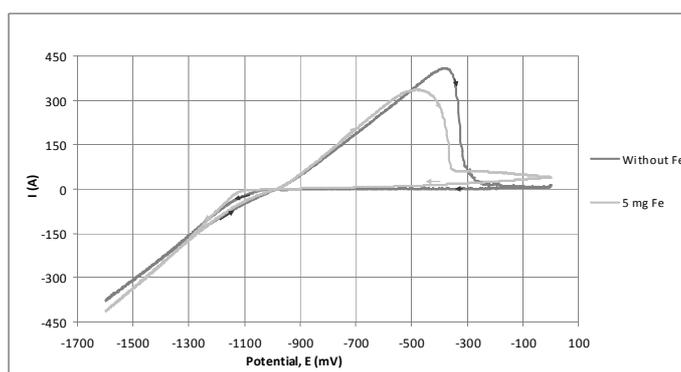
The addition of 5mg.L<sup>-1</sup> of Fe decreased the energy consumption and the addition of 10, and 15 mg/L of Fe increased energy consumption (Table 3).

The current efficiency obtained using the electrolyte of zinc sulfate without H<sub>2</sub>SO<sub>4</sub> addition, which was in the range of 99.5%, was higher than the current efficiency observed using the acid electrolyte, which was between 87.7% and 89.9%. For the electrolyte of zinc sulfate, the hydrogen reduction reaction did not compete significantly with zinc reduction. Overpotential of zinc cathode increases with a decrease of pH. Harrys <sup>[10]</sup> reported a quantitative analysis about pH variations near cathode during the electro deposition of a divalent metal. Harry <sup>[10]</sup> supposed that the rates of formation and dissociation of complex specie were high, and showed that alkalization of interface was due to OH<sup>-</sup> production in cathode, which can be inhibited if hydroxyls react to produce soluble compounds. Hydroxyl production can be due to water decomposition or oxygen reduction. Oxygen reduction is limited by the low concentration of oxygen in aqueous solution at room temperature and by the low diffusion coefficient.

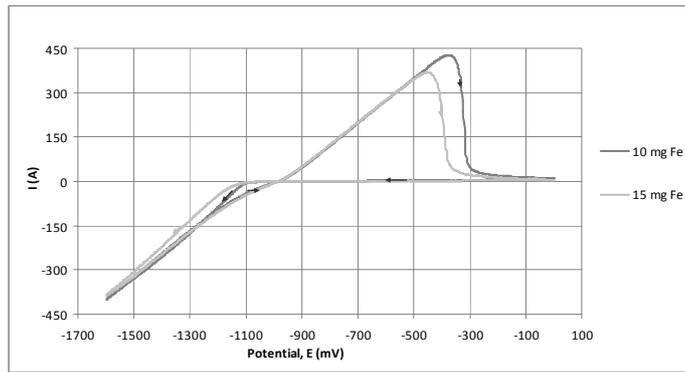
### 3.2 Cyclic Voltammetry

#### 3.2.1 Electrolyte of acid solution

Cyclic voltammetry experiments of zinc electrodeposition from 51.6 g.L<sup>-1</sup> Zn, and 180 g.L<sup>-1</sup> of H<sub>2</sub>SO<sub>4</sub> solution on aluminum substrate were carried out. The cathodic potential was varied in the range of 0 to -1600 mV(Ag/AgCl). The linear sweep curves that represent the onset of cathodic deposition and redissolution of deposited zinc are shown in Figure 3 for the solution without iron and with addition of 5 mg.L<sup>-1</sup> of Fe and in Figure 4 for the acid electrolyte with additions of 10 and 15 mg.L<sup>-1</sup> of Fe.



**Figure 3** – Linear sweep curves that represent the onset of cathodic deposition and redissolution of deposited zinc are shown for the solution without iron addition and with 5 mg.L<sup>-1</sup> Fe.



**Figure 4** - Linear sweep curves that represent the onset of cathodic deposition and redissolution of deposited zinc are shown for the acid electrolyte with additions of 10 and 15 mg.L<sup>-1</sup>.

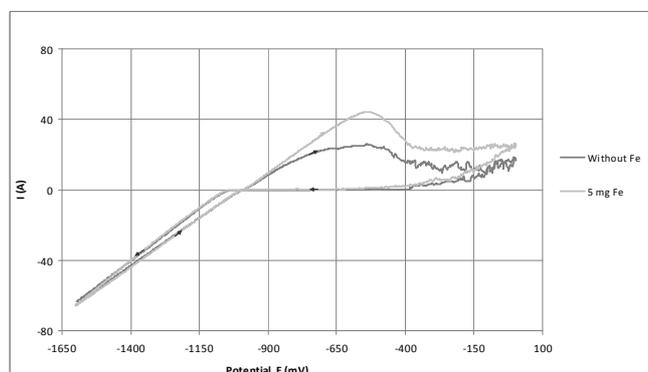
The cathodic curve can be attributed to the zinc deposition and hydrogen evolution reactions. Zinc deposition started at -1050 mV (Ag/AgCl). The anodic curves correspond to the dissolution of the deposited zinc. The current of anodic peak is higher for the electrolyte without iron and for the electrolyte with 10 mg.L<sup>-1</sup> of Fe, which also produced a higher zinc mass electrodeposited in the galvanostatic deposition than the solutions with 5 and 15 mg.L<sup>-1</sup> of iron. The electrolytes without iron and the electrolyte with 10 mg.L<sup>-1</sup> of Fe showed anodic peaks at a higher potentials than the peaks for electrolytes with 5, and 15 mg.L<sup>-1</sup> of Fe. The two points of interception of curves with axis of abscise (zero current) determine the segment of the nucleation hysteresis loop. This segment is called BC.

No peak corresponding to Fe deposition is observed, probably because Fe does not adhere to the Al substrate and it is physically flushed away from the electrode surface by hydrogen evolution.

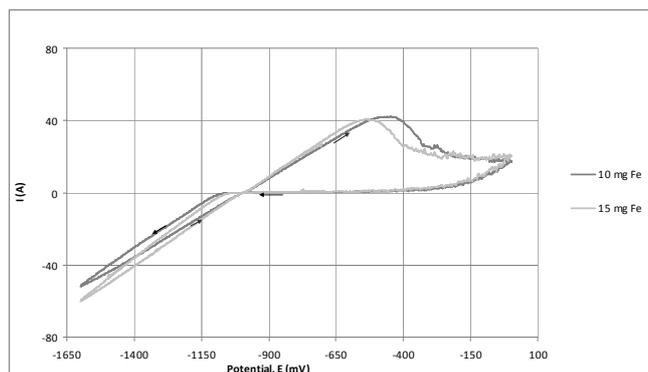
Figure 3 shows that for the acid electrolyte without iron, the BC portion decrease, indicating an acceleration of the cathodic process. The addition of iron to the acid electrolyte produces an inhibition of the zinc deposition, which can be confirmed by the results of electrodeposited zinc mass as shown in Table 2.

### 3.2.2 Electrolyte of zinc sulfate

The linear sweep curves that represent cathodic and anodic reactions for the zinc sulfate electrolyte are shown in Figures 5 and 6.



**Figure 5** - Linear sweep curves that represent the onset of cathodic deposition and redissolution of deposited zinc are shown for the zinc sulfate solution without iron addition and with 5 mg.L<sup>-1</sup> Fe.



**Figure 6** - Linear sweep curves that represent the onset of cathodic deposition and redissolution of deposited zinc are shown for the zinc sulfate electrolyte with additions of 10 and 15 mg.L<sup>-1</sup>.

The electrolyte without iron addition showed a lowest current density of anodic peak. A tendency of formation of a new anodic peak is observed at - 50 mV(Ag/AgCl). The EDS analysis of the electrodeposited zinc showed the presence of iron in deposited zinc, for the electrolysis using the zinc sulfate with 10 and 15 mg.L<sup>-1</sup> Fe.

The BC region in the voltammograms shown in Figures 5 and 6 are similar in length, indicating a same facility of the cathodic process. The values of electrodeposited zinc mass of the zinc sulfate electrolytes without iron, and with 5, 10, and 15 mg.L<sup>-1</sup> Fe are similar, and are according to these results.

### 3.3 Chemical Analysis of Electrolyte

#### 3.3.1 Acid electrolyte

Zinc concentration of the acid electrolyte decreased from 51.6 g.L<sup>-1</sup> to 48.0 g.L<sup>-1</sup> after the zinc electrolysis. Iron concentration decreased in solution after the electrolysis for the electrolyte without iron addition and with addition of 15 mg.L<sup>-1</sup> of iron (Table 4).

**Table 4** – Chemical analysis of electrolyte for acid solution

Element (g.L <sup>-1</sup> )	Without Fe	5mg.L <sup>-1</sup> Fe	10 mg.L <sup>-1</sup> Fe	15 mg.L <sup>-1</sup> Fe
Zn – before electrolysis	54.05	50.27	48.19	53.84
Zn – after electrolysis	53.92	46.95	47.83	54.16
Fe – before electrolysis ( 10 <sup>-3</sup> )	0.81	2.09	2.95	3.69
Fe – after electrolysis ( 10 <sup>-3</sup> )	0.63	2.21	2.99	3.53
Mn- before electrolysis	1.33	1.35	1.28	1.49
Mn – after electrolysis	1.39	1.05	1.18	1.45

Manganese contents in the acid electrolyte with addition of 5 and 10 mg.L<sup>-1</sup> of iron decreased after the electrolysis process. Manganese is deposited on anode according to the reaction:



### 3.3.2 Zinc sulfate electrolyte

Zinc concentration of the zinc sulfate electrolyte decreased from 59 g.L<sup>-1</sup> to 57 g.L<sup>-1</sup> after the zinc electrolysis (Table 5). Iron concentration in the electrolyte did not vary significantly after the electrolysis.

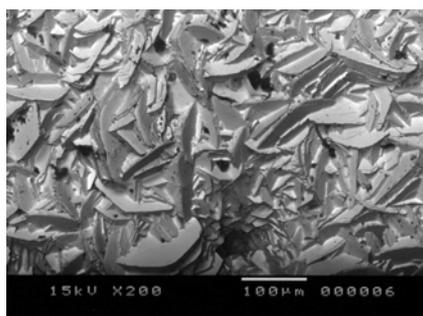
**Table 5** – Chemical analysis of electrolyte of zinc sulfate

Element (g/L)	Without Fe	5mg/L Fe	10 mg/L Fe	15 mg/L Fe
Zn – before electrolysis	59.28	60.80	59.49	60.89
Zn – after electrolysis	57.38	58.87	60.54	56.08
Fe – before electrolysis ( 10 <sup>-3</sup> )	0.11	0.52	1.38	2.21
Fe – after electrolysis ( 10 <sup>-3</sup> )	0.31	1.03	1.83	4.17
Mn- before electrolysis ( 10 <sup>-3</sup> )	0.04	0.06	0.09	0.07
Mn – after electrolysis ( 10 <sup>-3</sup> )	0.05	0.06	0.07	0.08

## 3.4 Morphology

### 3.4.1 Acid electrolyte

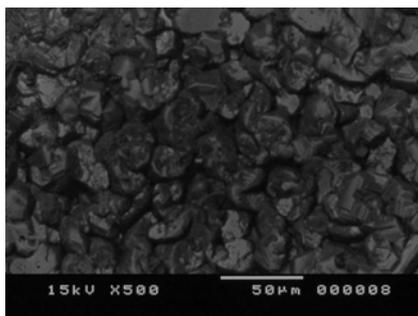
Morphology of electrodeposited zinc using acid electrolyte showed hexagonal crystals of zinc, deposited as multilayers, as shown in Figure 7, which presents a micrograph of surface of electrodeposited zinc using an acid electrolyte with 15 mg.L<sup>-1</sup> Fe.



**Figure 7** - Micrograph of surface of electrodeposited zinc using an acid electrolyte with 15 mg.L<sup>-1</sup> Fe. EDS analysis of the electrodeposited zinc showed the presence of iron on deposited zinc using the acid electrolyte without iron addition (0.25% wt Fe ), and with 10 mg.L<sup>-1</sup> Fe addition (0.11% wt Fe). The acid industrial electrolyte contained iron as impurity before iron addition, according to Table 1. The electrodeposited zinc using the acid electrolyte without iron addition showed a higher mass than the deposited zinc obtained with acid electrolyte with addition of 5 and 15 mg.L<sup>-1</sup> Fe. The porosity of electrodeposited zinc did not change, or decreased with iron addition in acid electrolyte as shown in Table 6.

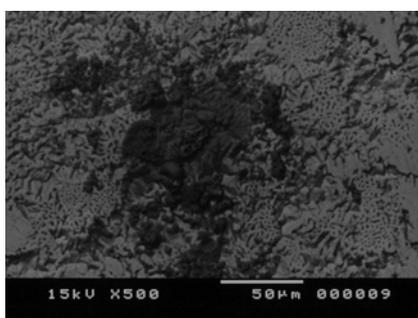
### 3.4.2 Zinc sulfate electrolyte

Morphology of the electrodeposited zinc consisted of circular nodules of several diameters, as shown in Figure 8, which presents a surface of the electrodeposited zinc using a zinc sulfate electrolyte without iron addition.



**Figure 8** - Morphology of the electrodeposited zinc consisting of circular nodules of several diameters obtained using the zinc sulfate electrolyte without iron addition.

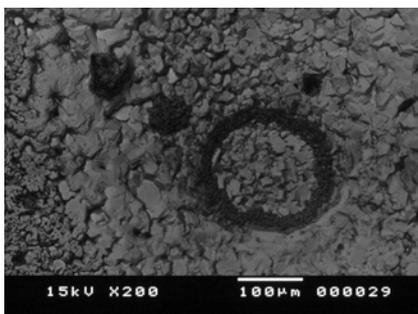
Hexagonal crystals were not identified in deposits obtained using the zinc sulfate electrolyte without H<sub>2</sub>SO<sub>4</sub> addition. Electrodeposited zinc using a electrolyte of Zn 60 g.L<sup>-1</sup> showed a content of sulfur of 2.5% (wt) in a general surface analysis. Some sample surfaces showed dark regions with concentration of sulfur of 11.7% wt (Figure 9).



**Figure 9** - Surface of electrodeposited zinc obtained using a zinc sulfate electrolyte without iron addition, showing dark regions with enrichment of sulfur

The electrodeposited zinc with 5 mg.L<sup>-1</sup> Fe showed two different zinc electrodeposits. One zinc sheet was deposited, detached, and another zinc sheet was deposited. The edges of the first sheet showed cracks, and enrichment in sulfur (12% wt). The first sheet showed a content of sulfur of 1.5% wt, 0.9 % of Al, and a porosity of 0.75%. The second sheet showed a higher porosity than the first sheet (1.62%), a higher content of aluminum (2.1% wt), and a lower content of sulfur (1% wt).

The electrodeposited zinc with 10 and 15 mg.L<sup>-1</sup> Fe showed the presence of iron in deposit surface (0.94 and 0.10% wt, respectively). Figure 10 showed a surface of electrodeposited zinc using the 60 g.L<sup>-1</sup> Zn electrolyte with 15 mg.L<sup>-1</sup> Fe. Some circular areas of a higher depth were observed on zinc surface (Figure 10).



**Figure 10** - Surface of electrodeposited zinc using the 60 g.L<sup>-1</sup> Zn electrolyte with 15 mg.L<sup>-1</sup> Fe.



One result of the galvanostatic deposition of zinc using a electrolyte of  $60 \text{ g.L}^{-1}$  Zn with  $10 \text{ mg.L}^{-1}$  Fe was the deposition of two zinc sheets. The first sheet showed a porosity of 31.5% and a content of 5% wt of sulfur. The second sheet showed a porosity of only 0.33%, and 3.1% of sulfur.

The iron addition increased the porosity of electrodeposited zinc, using a  $60 \text{ g.L}^{-1}$  Zn electrolyte, according Table 6.

**Table 6** – Porosity of the zinc electrodeposited

Electrolyte	Porosity (%)
60 g/L Zn Without Fe	0.33
60g/L Zn with 5 mg/L of Fe	0.75
60 g/L Zn with 10 mg/L of Fe	0.73
60 g/L Zn with 15 mg/L of Fe	0.53
Acid electrolyte	0.65
Acid electrolyte with 5 mg/L of Fe	0.63
Acid electrolyte with 10 mg/L of Fe	0.38
Acid electrolyte with 15 mg/L of Fe	0.56

#### 4 CONCLUSIONS

The current efficiency obtained using the electrolyte of zinc sulfate without  $\text{H}_2\text{SO}_4$  addition, which was in the range of 99.5%, was higher than the current efficiency observed using the acid electrolyte, which was between 87.7% and 89.9%.

Using the acid electrolyte, iron addition was detrimental to the zinc electrowinning, increasing the energy consumption and decreasing the current efficiency.

The addition of iron in the zinc sulfate electrolyte did not change the current efficiency.

The addition of  $5 \text{ mg.L}^{-1}$  of Fe in the zinc sulfate electrolyte decreased the energy consumption and the addition of 10, and 15  $\text{mg.L}^{-1}$  of Fe increased energy consumption.

The results of cyclic voltammetry indicated that the addition of iron to the acid electrolyte produces an inhibition of the zinc deposition, which can be confirmed by the results of electrodeposited zinc mass.

The addition of 5, 10 and 15  $\text{mg.L}^{-1}$  of iron in zinc sulfate electrolyte produced a higher current density of anodic peaks in cyclic voltammograms, and a tendency of formation of a new anodic peak was observed at potential of - 50 mV(Ag/AgCl).

Morphology of electrodeposited zinc using the acid electrolyte showed hexagonal crystals of zinc, and the morphology of the zinc deposited using a  $60 \text{ g.L}^{-1}$  Zn electrolyte consisted of circular nodules.

EDS analysis of the electrodeposited zinc showed the presence of iron on deposited zinc using the acid electrolyte without iron (0.25% wt Fe ), and with  $10 \text{ mg.L}^{-1}$  Fe (0.11% wt Fe).

The electrodeposited zinc with the  $60 \text{ g.L}^{-1}$  Zn electrolyte with 10 and 15  $\text{mg.L}^{-1}$  Fe showed the presence of iron in deposit surface (0.94 and 0.10% wt, respectively).

The iron addition increased the porosity of electrodeposited zinc, using a  $60 \text{ g.L}^{-1}$  Zn electrolyte.

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