World Applied Sciences Journal 13 (8): 1774-1780, 2011
ISSN 1818-4952

Effect of Distribution of Charge on the Separation and Purification of Metalic Zinc

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Abstract: The present work deals with the study of distribution of charge on the separation and purification of zinc. It involved the use of a Micro Controller based Electronic Switching System (MCDESS) for the separation of zinc from a mixture of samples. Mixtures of metal salts with varying concentrations were injected into a free electrolytic diffusion apparatus (FEDA). The micro cathodes surrounding the central anode were programmed with a Micro Controller based Electronic Switching System (MCDESS). The Micro Controller based Electronic Switching System (MCDESS) distributed the charge over the micro cathodes at time intervals of 100-900 μs or at the frequency of 2500 Hz to 278 Hz. The micro cathodes at applied 9.0 V, 1.8 A and 2500 Hertz electrodeposited with zinc metal, showing both separate and co-deposition. The purity of zinc was analyzed by Atomic Absorption Spectroscopy (AAS) and found to be 99.79±0.054 %.

Key words: Charge distribution · Conducting electrolyte · Digital thruster · Heavy metals · Purity · Separation

INTRODUCTION

Zinc was first recognized as a distinct metal in India and China prior to 1000 AD as reported by Morgan [1]. It received no attention as a metal in Europe until about 1500 AD. The use of Zinc in China dates from the 15th century, Paracelsus, in the 16th century, is said to have been the first European to recognize zinc as a distinct metallic element and to call it "Zinckum". In ancient India the production of zinc metal was very common. There are references of medicinal uses of zinc in the Charaka Samhita (300 BC). Zinc alloys have been used for centuries, as brass goods dating to 1000–1400 BC have been found in Israel. According to encyclopedia of chemistry an English metallurgist Libavius received in 1597 a quantity of zinc metal in its pure form [2]. Ulhman reported that, pyro metallurgical methods produced crude zinc that contained large amounts of impurities, the most important of which are Pb (0.7-3%), Fe (0.05-0.2%), Cd (0.3%) and also As in smaller amounts. [3].The purified solution of zinc chloride was treated by conventional methods of metal extraction to produce a high purity metal product as reported by Kekesi [4].

zinc purification demonstrated that anion exchange chromatography was a good method of separating elements that exhibited similar chemical properties as reported by Issihiki [5].To remove the chloride from the zinc solution by solvent extraction process using organic amine as an extracting agent dissolved in a suitable organic solvent was reported by Steintveit *et al.* [6] in 1974. Zinc and other valuable metals were then selectively stripped with sulphuric acid, generating a neutral solution of zinc sulphate suitable for zinc electro winning by Thorsen and Grislingas [7] in 1981. In 1975, Mori and Kiichi [8] also analyzed heavy metals in various samples. They separated zinc after extraction with nitric acid overnight. In 1978, There was a great revolution in the history of purification of Zinc when in 1981, Wandiga Shan [9] and coworkers analyzed samples of Kenyan rivers and lakes for zinc by flame atomic absorption spectrophotometry, differential pulse anodic stripping voltammetry and ion-selective electrodes. Biegler and Frazer [10] in 1986 studied the columbic efficiency of zinc electro winning in high-purity synthetic electrolytes. Measurements of columbic efficiency (QE) for zinc electro deposition were carried out under mass transfer-

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controlled conditions using a rotating disc electrode in synthetic acidic zinc sulphate electrolytes. In 1987, Corradi *et al.* [11] determined trace impurity contamination in silicon wafers at different stages of processing. During this trace impurity contamination, Zn was determined in significant detection limits. Ault and Frazer [12] in 1988, observed the effects of certain impurities on zinc electro winning in high-purity synthetic solutions. The individual effects of lead, copper, nickel, cobalt and antimony on zinc electro winning were evaluated by measurements in high-purity synthetic solutions, free from additives. In 1991, Cladera and coworkers [13] estimated Zn by an automatic system. In this method, they adopted anodic stripping potentiometry using the oxygen dissolved in the sample as oxidant. According to Bond M.A [14] and other researchers during 1976-94, following metals were determined in Zinc electrolyte Cd, Cu, Pb, Sb, Co, Ni, Tl, As using techniques DPP, using techniques DPASV Pb, Tl using techniques DPASV [15] Cd using techniques Fourier transform admittance [16] Cd, Co, Cu, Pb, Ni, Hg using techniques solvent extraction, liquid chromatography, spectrophotometric and electrochemical detection [17] Cu, Cd using techniques ISEs [18] Cd, Cu, Sb, Pb using techniques DPASV [19] Cu, Cd, Co, Ni using techniques DPASV, DPAdSV, DPV [20]. Co using techniques DPASV techniques [21] Fe(II), Fe(III), total Fe using techniques DPP, DCP [22] Zn, using techniques acid DFA, spectrophotometric and ISE detection [23] Co using techniques DPAdSV [24] Ni using techniques DPAdSV [25] Total As using techniques. In all forms of adsorptive stripping voltammetry (AdSV), a metal complex was accumulated at the electrode surface by adsorption. Alan M. Bond [26] in 1999 reported that, 1000 years ago in China and India, sophisticated technology for zinc production had been developed only in western civilization in the 18th century when the metal was called Indian tin or calamine (a term now reserved for natural Zinc carbonate). Nasi [27] in 2004 investigated the metals which were deposited electrochemically from aqueous solutions, only iron group metals (Ti, V, Cr, Mn, Co, Ni) began to deposit on cathodes at potentials which were several tenths of volts smaller than their equilibrium potentials. Shar *et al.* [28] in 2005 purified Zn (II) by a new catalytic kinetic spectrophotometric method. This method was based on the decolar reaction of H_2O_2 with calcon catalyzed by Zn (II) in weak acid medium. Recently Moghaddam [29] in 2006, studied the purification of zinc ammonical leaching solution by cementation.

The present work deals with the study of distribution of charge on the separation and purification of zinc. It involved the use of a Micro Controller based Electronic Switching System (MCESS) for the separation of zinc from a mixture of samples.

MATERIALS AND METHODS

Reagents and Lab Ware: A super pure HNO_3 (14 mol dm^{-3}) used in this work was acquired by analytical-reagent grade HNO_3 under sub-boiling condition and 100mL beakers and volumetric flasks made of glass(Pyrex). In order to reduce blank value, all of these lab ware used in this work were carefully washed with 10% HNO_3 (v/v) and then washed repeatedly with deionized water.

Preparation of Standard Calibration Solution Preparation

Sample Preparation: Sample solutions were prepared with deionized water with the resistivity being more than 10^5 Ùm. Commercial reagent grade zinc chloride ($ZnCl_2$) was dissolved in a hydrochloric acid solution, prepared from commercial special grade HCl and dissolved in deionized water. After the anion exchange separation the highly purified $ZnCl_2$ solution was evaporated to dryness to obtain anhydrous $ZnCl_2$. The anhydrous salt was re-dissolved in 0.1NHCl and subjected to a specially designed Free Electrolytic Diffusion Apparatus (FEDA) and programmed at 278 Hertz and 2500 Hertz, respectively. Experiments were performed at room temperature; pH was adjusted at 10 by using basic buffer, KCl was used as a conducting electrolyte while supporting electrolyte solution contained 1.2 M NH_4OH + 0.2 M NH_4Cl (pH = 10.).

AAS Determination: Every micro cathode was weighed before and after the electro deposition using a sensitive analytical balance. Dissolved the scratching of deposited material in the extra pure conc. HNO_3. Made its dilutions up to 1000 times. Run these dilutions in AAS (Aanalyst 100). Compared the readings to the standards (Perkins Elmer [a] and Aldrich [b]). A graph between concentration and absorption was plotted to know the concentration of desired metal and then finally determined the percentage purity of metallic deposit on each micro cathode respectively.

Methodology: The methodology of this research work is same as it has been used for the separation of lead and cadmium as reported in our previous work, Ahmad *et al.* [30, 31].

RESULTS AND DISCUSSION

Analysis of Commercial Anhydrous Zinc Chloride by ICP: 2.724g anhydrous zinc chloride (commercially available) was dissolved in 100 ml of double deionized water to prepare 0.2 M $ZnCl_2$. The volume was made up to the mark with distilled water and analyzed by ICP (Perkin Elmer Optima 2100). The ICP results obtained are reported in Table 1.

Purification of Zinc by Using Free Electrolytic Diffusion Apparatus: The sample solution prepared above was then subjected into the anodic chamber of the Free Electrolytic Diffusion Apparatus. The micro-electrodes used were 0.5 cm wide and 5.0 cm long. The conducting and supporting electrolytic solution was prepared by taking 20 ml of 1.2M NH_4OH + 0.2 M NH_4Cl was also added as a supporting electrolyte. The voltage was adjusted at 9.0 V while the current was 1.8 A. The process was continued for 60 minutes at a pH of 10.0.

The above experiment was repeated at frequencies of 278 Hz and 2500 Hz. The Zinc from Copper deposited on their respective pre weighed micro electrodes depending upon their electrode potential and ionic mobility along with micro electrode based switching system frequency. Both the Zinc and Copper were deposited on micro electrodes indicating their spectrum on 450 microelectrodes. A total of 450 micro electrodes were used in these experiments. In order to find out the percentage purity of Zinc from Copper on the respective electrodes 450, it was difficult to analyze all the micro electrodes by AAS ((Perkin Elmer AAnalyst 100), so micro electrodes were then grouped (sub grouped) based on the visibility of spectrum. Each micro electrode was then weighed on an Analytical balance and a sample solution of a group of 45 micro electrodes was then prepared. A total of 45 samples were thus obtained from spectrum and analyzed by AAS (Perkin Elmer AAnalyst 100). TheAAS conditions for the required base metal are shown in Table 2.

Preparation of Standards: Standards were prepared by dissolving 1g of pure zinc metal in 30 ml of 5M hydrochloric acid, followed by dilution with deionised water to prepare 1000 ml stock solution. Then further standards of 1, 2, 3, 4 and 5 ppm were prepared using this stock solution. Absorbance of these standards and the deposited sample was recorded at the wavelength (λ) of 213.9 nm on the atomic absorption spectrophotometer (Perkin Elmer Aanalyst 100). The values were also

Table 1: ICP analysis of commercial anhydrous zinc chloride

Sr.No.	Element	Conc. (ppm)	Conc. (ppm) at 278 Hz	Conc. (ppm) at 2500 Hz
1	Na	0.11	0.1	0.001
2	K	0.1	0.01	0.01
3	Co	0.12	0.1	0.01
4	Cd	0.1	0.01	0.01
5	Mg	0.2	0.01	0.01
6	Ca	0.09	0.01	0.01
7	Si	0.18	0.11	0.01
8	Pb	0.2	0.01	0.01
9	Zn	93.2	99.49	99.79
10	Cu	5.2	0.04	0.05
12	Fe	0.3	0.01	0.01
13	Ca	0.1	0.02	0.01
14	Ni	0.1	0.01	0.01

Table 2: AAS conditions Zinc

PERKIN ELMER	AANALYST 100
Oxidant	Air
Fuel	Acetylene
Fuel: Oxidant	3:06
Spectral Bands Pass	1.0 nm
Fuel Pressure	15 PSI
Lamp Current	5.0 mA
Flame Speed	160 cm/s
Flame Temperature	2250°C
wavelength (λ) for Zinc	213.9 nm
wavelength (λ) for Copper	327.4 nm

Table 3: Separation of Zinc from Copper by using FEDA

	At 278 Hz		At 2500 Hz	
MCG No.	PP of Cu +SD	PP of Zn +SD	PP of Cu +SD	PP of Zn +SD
1	95.87±0.005	4.13±0.0456	94.99±0.036	5.01±0.0154
2	95.01±0.006	4.99±0.0648	91.25±0.046	8.75±0.032
3	94.39±0.00	5.61±0.0365	88.36±0.025	11.64±0.036
4	92.1±0.004	7.9±40.0125	85.48±0.031	14.52±50.032
5	90.45±0.065	9.55±0.0325	81.69±0.0321	18.31±0.045
6	88.21±0.032	11.79±0.032	79.89±0.036	20.11±0.0654
7	85.54±0.025	14.46±0.045	77.25±0.0458	22.75±10.325
8	82.36±0.036	17.64±0.036	75.21±0.036	24.79±0.0621
9	79.69±0.014	20.31±0.036	72.56±0.254	27.44±0.0785
10	75.69±0.036	24.31±0.036	70.01±0.014	29.99±0.0125
11	72.45±0.014	27.55±0.01485	68.25±0.025	31.75±0.0356
12	70.64±0.0368	29.36±0.087	65.23±40.036	34.77±0.0324
13	68.23±0.074	31.77±0.098	62.36±0.0987	37.64±0.0896
14	65.21±0.096	34.79±0.65	60.01±0.062	39.99±0.0621
15	62.36±0.098	37.64±0.65	56.58±10.0312	43.42±0.0369
16	60.23±0.065	39.77±0.015	53.48±0.0632	46.52±0.0156
17	58.39±0.032	41.61±0.25	50.02±0.0145	49.98±0.0325
18	55.41±0.064	44.59±0.14	46.38±0.0215	53.62±0.018
19	52.26±0.0643	47.74±0.15	42.69±0.0365	57.31±0.0563
20	48.36±0.0125	51.64±0.215	40.05±0.0156	59.95±0.0325
21	45.21±0.0452	54.79±0.1863	36.99±0.0321	63.01±0.014
22	44.25±0.0156	55.75±0.14	32.74±0.698	67.26±50.025
23	41.01±0.0258	58.99±0.13	29.69±0.0254	70.31±0.014
24	36.36±0.0345	63.64±0.12	25.44±0.0321	74.56±0.005
25	35.09±0.045	64.91±0.101	23.11±0.02558	76.89±0.006
26	31.02±0.0856	68.98±0.09	20.99±0.065	79.01±0.004
27	28.69±0.045	71.31±0.087	17.54±0.0326	82.46±0.0098
28	25.99±0.046	74.01±0.005	15.1±0.036	84.9±0.0250
29	23.45±0.15	76.55±0.06	12.35±50.0245	87.65±0.0078
30	21.65±0.28	78.35±0.006	9.36±0.0178	90.64±0.0065
31	20.01±0.36	79.99±0.04	7.25±0.314	92.75±0.0045
32	18.69±0.0780	81.31±0.141	4.54±0.015	95.46±0.0012
33	15.01±0.0698	84.99±0.002	2.99±0.036	97.01±0.0036
34	12.23±0.087	87.77±0.004	1.87±0.078	98.13±0.0039
35	11.69±0.063	88.31±0.033	1.11±0.0315	98.89±0.048
36	10.65±0.018	89.35±0.0254	0.95±0.0315	99.05±0.0034
37	9.36±0.0578	90.64±0.0365	0.81±0.0369	99.19±0.015
38	8.65±0.0126	91.35±0.0145	0.74±0.068	99.26±0.031
39	7.54±0.0356	92.46±0.025	0.63±0.045	99.37±0.068
40	5.35±0.0164	94.65±0.069	0.54±0.039	99.46±0.0065
41	3.58±0.0321	96.42±0.078	0.48±0.0147	99.52±0.069
42	2.47±0.025	97.53±0.038	0.35±0.035	99.65±0.0678
43	1.01±0.156	98.99±0.046	0.31±0.031	99.69±0.0126
44	0.71±0.015	99.29±0.036	0.29±0.092	99.71±0.0369
45	0.51±0.009	99.49±0.064	0.21±0.081	99.79±0.054

MCG NO: Micro Cathode Group Number; SD: Standard Deviation; PP: Percentage Purity;

compared to the Aldrich and Perkin Elmer standards for calibration. The results of 45 samples obtained though 450 micro electrodes are reproduced in Table 3.

Effect of Charge Distribution on the Purity of Zinc: The ICP analysis of the Zinc Chloride shows that it contains only 68 % pure Zinc while Cu is the main impurity present in as shown in Table 1. In FEDA there was a set of pre weighed 300 micro cathodes. In the start percentage purity of Cu on first ten micro cathodes was calculated to be 95.87 ± 0.005, while the percentage purity of Zinc at 278 Hz was found to be 4.13 ± 0.0456. While at 2500 Hz, the percentage purity of Cu on first five micro cathodes was 94.99 ± 0.036, while Zinc purity was 5.01 ± 0.0154 as given in detail in Table 3.

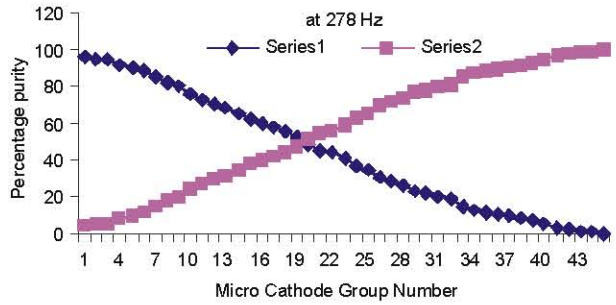

Fig. 1: Comparison of Percentages purities of Zinc from Copper at 278 Hz.
(Series 1 for Copper and series 2 for Zinc)

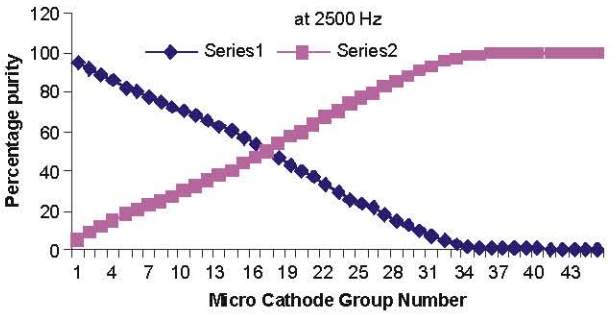

Fig. 2: Comparison of Percentages purities of Zinc from Copper at 2500 Hz.
(Series 1 for Copper and series 2 for Zinc)

At 278 Hz, an alloy of Zinc and Copper with varying composition formed at the micro cathodes 180-210 as shown in figure 1. While at 2500 Hz, the Zinc -Copper alloy formed on the earlier group of micro cathodes (150-180) as shown in figure 2. This proved that at a higher frequency (2500 Hz) Zinc -Copper alloy formation took place earlier due to enhanced ionic mobility of cations. At the last ten micro cathodes the percentage purity of Cu decreased to 0.51 ± 0.009, while the percentage purity of Zinc increased to 99.49 ± 0.064, at low frequency (278 Hz). At higher frequency (2500 Hz), Zinc separates from copper in the purified form (99.79 ± 0.054), as given in detail in Table 3.

While the literature is silent about the purification of zinc by the distribution of charge over micro cathodes and few other methods for the recovery of zinc have been reported.

In 1991, Cladera, A and coworkers [13] estimated Zn by an automatic system adopting anodic stripping potentiometry. The system relied on the use of a PC-compatible computer for instrumental control and data acquisition and processing. The reported method for carrying out purification procedure was cementation with zinc dust, because the residue from this process was metallic cement, which was treated in the process for recovering the metals as also reported by Perales in 1993 [32].

Two major manufacturing processes have emerged since 1999. The first was a thermal process where, initially, the zinc sulfide ore (most common source of zinc) was roasted in air to produce the crude oxide. ZnO was then reduced with carbon at high temperatures and the resultant metal vapour condensed to the solid metal. Unfortunately, due to the production of harmful greenhouse gases and a zinc product which was only about 98% pure, this type of zinc production had its drawbacks. The second process was the electrolytic one where, initially, the zinc ore was roasted at high temperatures and then dissolved into solution before the zinc ions were electrolyzed to metallic zinc at a cathode.

While other important process impurities such as copper and cadmium were deposited close to their equilibrium potentials. This difference in metal deposition caused problems in the purification and use of different reagents. Recently Moghaddam.J [29] in 2006, studied the purification of zinc ammonical leaching solution by cementation. He determined optimum conditions with experimental design using Taguchis method. The total optimum purification conditions obtained from this study were, series 1 for 45 min at 35 °C having concentration of 1.6 g/dm3 and 450 rpm, respectively. In these conditions, many impurities were removed.

Effect of Voltage: In the production of pure zinc at high voltage (18-30 V), the conductivity of electrolyte increased but the evolution of hydrogen gas at the micro cathodes also increased. The rapid reduction of hydrogen ions at the micro cathodes not only increased the temperature of the apparatus but also affected the pH of the conducting electrolyte. The experiments were performed at a voltage of 9.0 V at which less evolution of hydrogen took place. Deposition is usually overlapped by the reduction of hydrogen ions during electro deposition of less noble metal. This normally lead to a large increase of the pH value close to the electrode surface and to a change of the deposition behavior and the properties of the deposits. The hydrogen reaction had therefore was minimized and this was normally done by buffering the electrolyte. Zinc was also produced by electrolysis of aqueous solution using Pb-Ag as anode Al as anode having no diaphragm at 35 °C and 3.2-3.6 V.

Distance Between Cathodes and Anode: The distance between a cathode and an anode plays very important role during electro deposition. Usually decreasing their distance decreased the resistivity with an accompanying increase in conductivity. However a small distance (2-3 cm) between the micro cathodes and anode was not suitable for the separation of cations present in our mixtures, usually resulting in co deposition. Good separation of Zinc from the impurities was possible when the micro cathodes and anode were at a distance of 8.1 cm.

Distance Between Micro Cathodes: In these experiments a total of 450 micro cathodes were arranged around the cylindrical platinum anode in a circular fashion. Each micro cathode was 1.0 mm apart. It was found that micro cathodes at this distance show good separation of Zinc from the matrix. When the distance between micro cathodes is increased to more than 1 mm then co deposition of Zinc and impurities took place on all micro cathodes.

Conducting and Supporting Electrolytes: To enhance the electrical conductivity for purification of Zinc Merck grade KCl (1.0 M) was used as a conducting electrolyte. The supporting electrolyte used for these experiments consisted of (i) 1.2 M NH_4OH + 0.2 M NH_4Cl (pH = 10.0). This supporting electrolyte was used at negative electrode potential, which was necessary for these experiments. The reported [33] supporting electrolytes(i) 0.4 M Sodium tartarate + 0.1 M NaH tartarate (ii) 0.4 M KCN + 0.2 M KOH

Concentration of Sample: It was found that a higher concentration of sample mixture (1.0 M) caused the co deposition of Zinc and impurities on all available micro cathodes. The rapid release of Zinc ions in the conducting electrolyte did not allow their complete separation. For good results 1 ml of sample mixture (0.2 M) was diluted 20 times with deionized water. The diluted sample mixture diffused into the outer conducting electrolyte drop wise.

Conclusion The observed optimum conditions for the Zinc purity were the use of a supporting electrolyte solution containing 1.2 M NH_4OH + 0.2 M NH_4Cl (pH = 10.0). 1.0 M KCl was used as a conducting electrolyte. Each microcathode was 1 mm apart. The micro cathodes and anode were at a distance of 8.1 cm and at applied 9.0 V and 1.8 A, when the charge was rotated at 2500 Hz.

ACKNOWLEDGEMENT

Authors specially thank to Higher Education Commission of Pakistan for providing Financial assistance (PIN 063-121117-ps3-016).

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