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## Recovery of Zinc from Zinc Dross Using Pyrometallurgical and Electrochemical Methods

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RECOVERY of zinc from its dross was investigated using electrochemical and pyrometallurgical methods. Almost 100% pure zinc was obtained using these methods. A small bench scale system for the investigating the recovery of zinc using pyrometallurgical methods was developed. The system based on the evaporation of zinc (in the dross) at 1050°C and condensing zinc vapors into pure zinc in a separate crucible. Almost pure Zn (99.95%) is obtained using this method. Recovery of zinc was also performed using electrowinning from the acidified zinc sulfate bath and electrorefining of zinc dross anodes in the alkaline ammonia bath. In either method different parameters including current density and deposition time were addressed. Zinc powder of almost 100% purity was obtained.

**Keywords:** Zinc dross, Hot dip galvanizing, Electrorefining, Electrowinning, Pyrometallurgical techniques.

## Introduction

Over the past fifty years, the production of metallic zinc has increased by over three times with consumption. However, due to this increasing demand as well as numerous other economic issues, the production supply of primary zinc has been unable to keep pace with required zinc tonnage. At present, the scarcity of zinc is making many zinc consumers invest in secured zinc supply just to avoid a potential interruption in raw material [1-5].

Trpcevska, et al. [6] investigated metallic zinc recovery from the coarse-grained fraction of zinc ash from hot dip galvanizing using the pyrometallurgical process. A designed laboratory equipment which used centrifugal force and protective atmosphere of a nitrogen flow during the process of melting and separation of zinc was developed.

Hard zinc refining through removal of iron was successfully achieved by using different refining agents in the order; aluminum turnings > zinc powder > Al/Zn mixture [7]. Iron is present within hard zinc as intermetallic compounds. The addition of the refining agents to the hard zinc melt resulted in the formation of different oxides and intermetallic compounds as a slag of refining. A refined zinc containing 0.5 wt % Fe was obtained by adding 0.4 wt% Al at 700 ºC after 2h of refining.

Huajun, et al. investigated the electrorefining of zinc dross in $NH_3$–$NH_4Cl$ system [8]. In order to obtain a high recovery ratio of zinc dross and high current efficiency, an electrolyte composition of 4.0 M $NH_3$, 4.0 M $NH_4Cl$ and no more than 80 g $L^{-1}$ Zn(II) is suitable. The power consumption can be decreased by increasing the temperature of electrolysis.

Abdul Ghani et al. [9] have treated flue dust, produced as by-product during the production of steel from electric arc furnace, using sodium hydroxide as leaching medium. The optimum alkali concentration for selective leaching of zinc was found to be 4 M with a zinc recovery of 90 %.

Abdel-Aal et al. performed valuable research work on the extraction of zinc from Egyptian ores using sulfuric acid pressure leaching [10-12]. Under the optimum conditions employed, up to 99.25% of zinc extraction and 0.20% silica dissolution are obtained.

Research papers were also published on recovery of zinc and copper from brass slag,

and recovery of zinc from zinc plating mud, recovery of zinc from zinc-carbon batteries, Leaching kinetics of zinc residues augmented with ultrasound and zinc recovery from galvanizing dross by super-gravity separation [13-19].

Viswanath and George [20] studied the electrowinning of zinc from ammonia and glycerol with glycerol varying from 0 to 20% and ammonia from 0.5 to 1.5 N was studied. The zinc powder particles were spherical in shape with size varying from 2.63 to 79 μm, depending on the concentration of glycerol and the ammonia.

Das, et al. [21] proposed that the addition of perfluorocarboxylic acids increased the current efficiency, decreased power consumption and produced better surface morphologies during the electrowinning of zinc from acidic sulphate solutions. Perfluorocarboxylic acids were found to be better additives for zinc electrodeposition when Sb(III) was absent from the zinc electrolyte.

Sanjay [22] developed a process based on the direct electrowinning of zinc dross in an alkaline medium. In the electrolytic cell zinc dross was taken in a porous metallic container and used as the anode. It was found that at 5 A/dm$^2$ current density, the formation of zinc powder is possible with 1.23 kWh per kg of energy consumption. The impressed voltage was found 1.2 V with a current efficiency of 90%.

Tripthy et al [23] investigated the electrowinning of zinc from acidic sulfate path. The effects of perfluorobutyric acid, perfluoroheptanoic acid and perfluorooctanoic acid, in the presence and absence of antimony(III), on the cathodic current efficiency, power consumption and polarization behavior of the cathode during the electrowinning of zinc from acidic sulphate solutions were investigated. Addition of any of these perfluorocarboxylic acids increased the current efficiency, decreased power consumption and produced better surface morphologies.

Stanojevi, et al [24] investigated Zinc Dissolution Process in sulfuric acid. They proposed that acceleration of zinc dissolving is possible using copper cathode depending on the quality of electrical contact between copper electrode and zinc.

Guo et al [25] investigated zinc electrowinning from acidic chloride solutions. The effects of

catholyte flow velocity, temperature, current density, and zinc, HCl, NaCl and inhibitor TBACl have been investigated. Optimized electrowinning was obtained from solution containing 20 g/l Zn, 9 g/l HCl, 1 mol/l NaCl, and 50 mg/ TBACl and at a flow velocity of 100 cm/min and electrowinning at a current density of 300 A/m$^2$ at 35-40°C.

The effects of sodium laurayl sulphate on the electrowinning of Zinc from acidic sulphate solution were investigated by B. C. Tripathy, et al. [26] and the results were compared with glue as the addition agents. Results indicated that Addition of sodium laurayl sulphate increased the current efficiency, sodium laurayl sulphate and glue affected the cathodic polarization of zinc similarly.

The recovery of zinc metal from zinc ash, with a zinc content of about 76% by leaching and electrowinning has been studied. The presence of chloride has been identified as the major problem area. A catalytic anode based on the iridium dioxide ($IrO_2$) was used as the anode material to overcome this problem [27].

Rabah and Elsayed as well as Koros et al. [28,29] studied the factors affecting the performance and efficiency of the pyrometallurgical processes of zinc ash by using ammonium-chloride flux. The recovery efficiency amounts to 89% and 63% for zinc ash having particle size diameter of +1.25 mm and –0.4 + 0.315 mm, respectively, with 10% flux at 600°C.

Ratherford [30] studied a process called decantation filtration. A filter plate was attached on the top of the melting crucible. This gave a yield of 75% of the recoverable zinc at 600°C with a settling time of 37 min., and the remaining iron was 0.78%.

The aim of the present study is to recover zinc dross from Kandeel For Steel Co. The zinc dross was brought from a leading galvanizing company in Egypt, which uses hot dip continuous galvanizing process. The investigated sample was taken from the top-dross of the galvanizing bath. The sample contains a high percent of zinc metal (>90%), which associates the intermetallic $Fe_2Al_5$ dross. The work developed an effective technology for recycling zinc from zinc dross. The recovered zinc can be returned to the production cycle which would significantly reduce the cost of obtaining galvanized products.

## Experimental

### Materials and Reagents

The materials used in this study include zinc dross from Kandeel For Steel Co (raw material), aluminium, lead and copper sheets. Chemicals used include: sodium hydroxide, ammonium chloride, ammonia, gelatin, hydrochloric acid, sulfuric acid, hydrogen peroxide and acetone. All chemicals are analytical grade from Fluka.

### Apparatus

Adwa pH-meter model (AD8000), Denver electric balance model (Mxx-5001), Inductively couples plazma model (Perkinelmer optima 2000DV), programmable Chroma DC power supply model (62050P-100-100), scanning electron microscopy model (FEI-Netherland-INSPECTS 50), electric furnace and glass wares.

### Recovery of zinc from zinc dross using electrochemical methods

Zinc dross having a chemical composition: 96.18 wt% Zn, 2.06% Fe, 1.17 wt% Al, 0.017 wt% Si, 0.007 wt% Pb, 0.007 wt% Mg and 0.02 wt% Ca which resulted from hot dip galvanizing process was dissolved in conc. $H_2SO_4$ to give zinc sulfate solution (100 g Zn/l). In the present study 103.97 g of zinc dross was dissolved in 1 L of 3.76 M $H_2SO_4$ to acquire the desired concentration of zinc (100 g/l). Dissolution was performed at 55ºC for 1.5 h. Hydrogen peroxide (30 wt%) was added to the produced zinc sulfate lacquer so as to give 1% concentration at a pH of 5-5.5 in order to remove iron. The filtrated zinc sulfate solution was taken and used in the experiments of electrowinning and electrorefining of zinc.

Electrowinning experiments were conducted in a one-liter Perspex cell. Two lead sheets having dimensions 7.5 cm x7 cm were used as anode whereas copper sheet having same dimensions was used as the cathode. The zinc sulfate solution was adjusted to contain 100 g Zn/l whereas conc. $H_2SO_4$ was added so that each liter contains 50 ml. Different current densities (30-90 mA/cm²) were tested. The testing period was 30 min. These experiments were used to determine the optimum current density that gives the highest efficiency.

Electrorefining of the anodes was performed in a Perspex glass cell (20 × 10 × 15 cm), the distance between the anode and cathode was fixed at 3 cm, to prevent problems with anodic sludge. Anodes 4x7x1 cm were made from zinc dross. The anodes were polished to give a bright surface using emery papers of grades 400, 800 and 1200 and degreased by immersion into acetone. The cathode was aluminium ($4 × 7 × 0.3$ cm). The electrolyte used was 4 M $NH_4Cl$, $4MNH_3$ and zinc sulfate as zinc with concentration of $1.2$ $gL^{-1}$. Gelatin was added into the electrolyte system at $0.1$ g $L^{-1}$. The chemicals, ammonia, ammonium chloride and gelatin were analytical reagents. Experiments were performed at room temperature.

### Bench scale system for pyrometallurgical zinc recovery

A small bench scale system for investigation of zinc recovery using pyrometallurgical methods was developed. The schematic presentation of this system shown in Fig. 1. The system consists of a furnace as a heating source. A silicon carbide crucible which could hold about 1 Kg zinc dross. The crucible was connected to a silicon carbide tube which can withstand high temperature as high as 1200ºC. The end of the silicon carbide tube is connected to a second crucible in which recovered zinc is received. Figure 2a represents the two crucibles connected by the SIC tube while Fig. 2b represents the heating crucible inside the furnace while the receiving crucible outside the furnace. This system was perfectly insulated using ceramic fiber from surrounding (Fig. 2c). Silicon carbide tube with sealed to both crucibles using thermal cement which succeeded in preventing any leakage during the experiment.
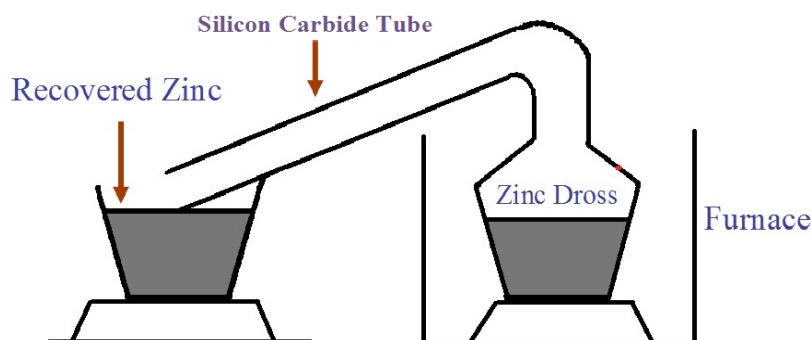


**Fig. 1. Schematic presentation of the pyrometallurgical system used for Zn recovery from Zn-dross.**

**Fig. 2. A photograph of the pyrometallurgical system used for Zn recovery and recovered zinc from Zn-dross.**

## Results and Discussion

The dross forms as a result of either oxidation of the zinc at the galvanizing bath surface or by intermetallic reactions with the iron in the bath. Iron is introduced by the steel substrates being coated. There are two types of galvanizing processes; general and continuous galvanizing. In both types, the zinc baths possess variable amounts of aluminum (0-0.05 wt% Al: General & 0.12-0.3 wt% Al: Continuous) resulting in different dross compounds being formed. Floating "top" dross (ash or skimming) for general galvanizing consist primarily of ZnO with varying amounts of $Zn_5(OH)_8Cl_2$ while the dross on the bottom of the bath is $FeZn_{13}$ ($\xi$). The top dross in continuous galvanizing is $Fe_2Al_5$ ($\eta$) and the bottom dross is $FeZn_{10}$ ($\delta$). However, during typical cleaning procedures to remove the accumulation of these drosses, a tremendous amount of "clean" metallic zinc is also extracted from the galvanizing pots. General galvanizing dross can contain between 40% to 80% metallic zinc while continuous galvanizing dross may contain up to 95% usable zinc [4].

Chemical analysis of $ZnSO_4.xH_2O$ prepared from the dissolving dross in conc. $H_2SO_4$ was determined using ICP technique and shows 32.1 wt% $Zn^{+2}$, 0.99 wt% $Al^{+3}$, 0.53 wt% $Fe^{+2}$. It is noted that Fe content is much lower than the Fe content in the original dross. The decrease in iron content is related to the addition of hydrogen peroxide at a concentration of 1% to the produced zinc sulfate solution at mildly acidic pH (~5-5.5). The addition of $H_2O_2$ results in the precipitation of ferric sulfate. The filtrated zinc sulfate is taken for electrowinning and electrorefining experiments.

*Recovery of Zinc Using Electrowinning and Electrorefining Techniques*

Table 1 shows the working conditions as well as the current efficiency of zinc recovery using electrowinning experiments. The current efficiency equals the experimental weight gain divided by the theoretical weight gain multiplied by 100. The theoretical weight gain was calculated using Faraday's law shown below:

$$m = (It/F)(M/z) \qquad (1)$$

- $m$ is the mass of the substance liberated at an electrode in grams

- I is current in ampere

- t is the time in seconds

- $F = 96485$ C mol$^{-1}$ is the Faraday constant

- $M$ is the molar mass of the substance

- $z$ is the valence number of ions of the substance (electrons transferred per ion).

The maximum current efficiency of 90% is obtained at the lowest current density (40 mA/cm$^2$) keeping the time at 30 min and temperature at 25°C. The current efficiency is decreased by increasing current density and almost diminishes at the highest current density (90 mA/cm$^2$). The deposited zinc was striped by gentle scratching and analyzed using Inductively Couples Plazma (ICP) technique. The deposit chemical analysis shows 99.99 wt% Zn, 0.0003 wt% Al, 0.0007 wt% Fe. Fortunately, the deposit was almost 100% zinc with minute traces of Fe and Al.

Table 2 shows the effect of current on the efficiency of the electrorefining of zinc using the NH$_3$/NH$_4$Cl bath. Low efficiencies were obtained at low currents (26.8-53.6 mA/cm$^2$). At sufficiently high current (107 mA/cm$^2$) the

efficiency is largely increased up to 96.6%.

Table 3 shows the effect of time on the efficiency of Zn electrorefining was investigated at a fixed current density of 107 mA/cm$^2$. The time of the experiment was extended to 10 h while the cathode is taken every two hours, dried and reweighed to determine the amount of zinc deposited. This weight of zinc is divided by the theoretical weight of Zn calculated using Faraday's low as mentioned in equation 1. Table 3 shows that the efficiency of the electrorefining process is slightly decreased with time (2%) till 8 h and then markedly decreased at 10 h. The high cathodic current efficiency can be attributed to the elimination of hydrogen evolution in the ammoniacal ammonium salt system [10].

Figure 3 shows the morphology of zinc deposited in the electrowinning experiments using the sulfuric acid bath. The images reveal the presence of excavations in the deposit. These excavations have different sizes and are distributed along the surface. At high magnification, deposit takes the form of rectangular flakes condensed together.

**TABLE 1. The working conditions as well as the current efficiency of the electrowinning experiments. The time was fixed at 30 min.**

| Current Density, mA/cm$^2$ | Current, A | Wt of Cu cathode before testing/g | Wt of Cu cathode after testing/g | Wt gain/g | Theoretical wt gain/g | Current efficiency% |
|---|---|---|---|---|---|---|
| 40 | 0.288 | 0.6954 | 0.8535 | 0.1581 | 0.1756 | 90.03 |
| 50 | 0.36 | 0.6753 | 0.8517 | 0.1764 | 0.2196 | 80.33 |
| 60 | 0.48 | 0.8672 | 0.8940 | 0.2222 | 0.2928 | 75.89 |
| 70 | 0.56 | 0.5490 | 0.8080 | 0.2590 | 0.3416 | 75.82 |
| 80 | 0.64 | 0.9035 | 1.1430 | 0.2395 | 0.3904 | 61.35 |
| 90 | 0.72 | 0.8830 | 0.9126 | 0.0296 | 0.4392 | 6.74 |

**TABLE 2. The effect of current density on the efficiency of the electrorefining tests using the NH$_3$/NH$_4$Cl bath.**

| Current density mA/cm$^2$ | Time min | Al cathode weight before Zn deposition | Al cathode weight after Zn deposition | Zn Deposit weight | Theoretical weight of Zn deposit | % Efficiency |
|---|---|---|---|---|---|---|
| 26.78 | 60 | 11.1354 | 11.9177 | 0.7823 | 1.829 | 42.7 |
| 35.71 | 60 | 11.2890 | 12.4870 | 1.2086 | 2.400 | 50.0 |
| 53.57 | 60 | 11.5174 | 13.5775 | 2.0601 | 3.6500 | 54.7 |
| 107.14 | 60 | 11.6547 | 18.6817 | 7.0270 | 7.2746 | 96.6 |

**TABLE 3. Effect of time on the efficiency of the electrorefining of zinc from NH$_3$/NH$_4$Cl bath.**

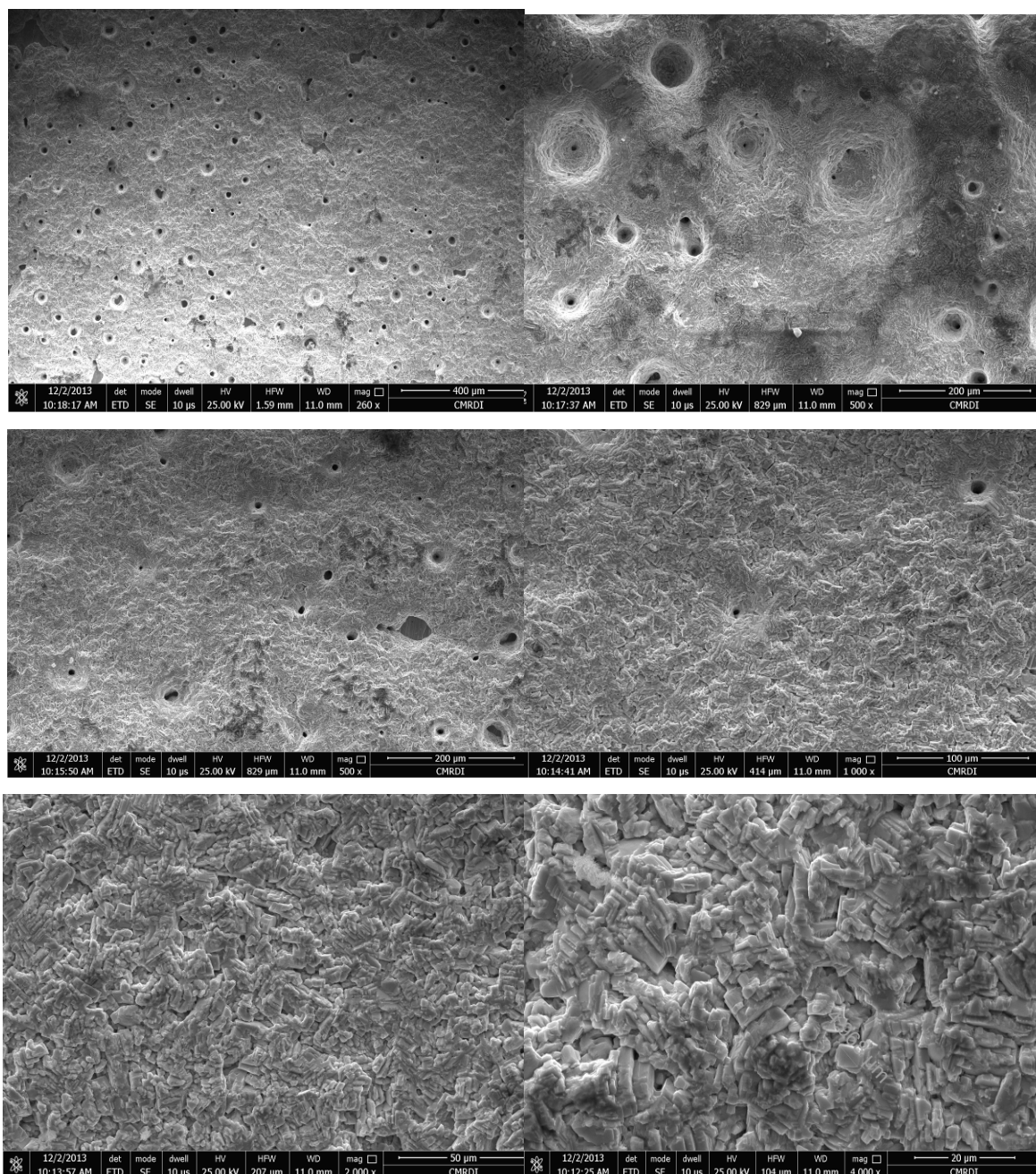| Current density mA/cm² | Time min | Al cathode weight before Zn deposition | Al cathode weight after Zn deposition | Zn Deposit weight | Theoretical weight of Zn deposit | % Efficiency |
|---|---|---|---|---|---|---|
| 107.14 | 120 | 33.8919 | 48.0330 | 14.1411 | 14.6380 | 96.60 |
| 107.14 | 270 | 33.8919 | 64.5650 | 30.6731 | 32. 9300 | 93.130 |
| 107.14 | 360 | 33.8919 | 75.4850 | 41.5931 | 43.9160 | 94.708 |
| 107.14 | 480 | 33.8919 | 89.1550 | 55.2631 | 58.5500 | 94.389 |
| 107.14 | 600 | 33.8919 | 98.8730 | 64.9811 | 73.1963 | 88.770 |



**Fig. 3. Surface morphology of zinc deposited from ZnSO4/H2SO4 bath.**

Figure 4 represents the morphology of zinc deposited using electrorefining method from the NH$_3$/NH$_4$Clbath at 107 mA/cm$^2$ and 2 h. Aggregates of zinc deposits were obtained. Each aggregate seems to consist of small particulates (flakes) tightly adhered to each other. The aggregates are obviously distinguished from each other and mostly of the same size. SEM images show that deposits formed using electrorefining method are more compact and adhered which allows the formation of high quantity of deposit in comparison to the electrowinning method.

It is clear that electrofinning technique excels the electrowinning technique regarding the ease of the process where electrorefining technique uses zinc dross as anode whereas in the electrowinning technique zinc dross must be dissolved prior to electrodeposition.
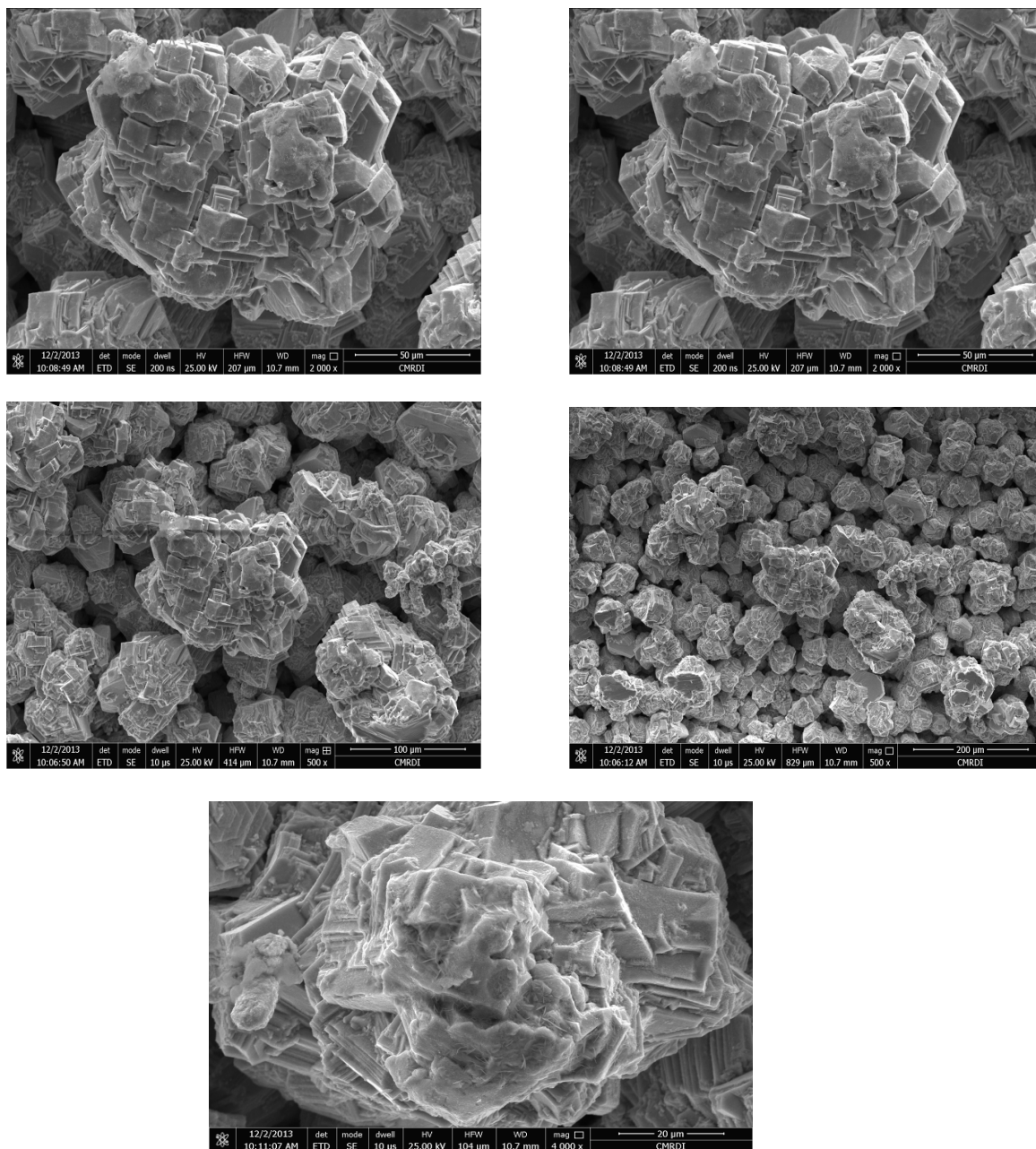


**Fig. 4. Surface morphology of zinc deposited from NH3/NH4Cl bath.**

*Recovery of Zinc Using Pyrometallurgical Technique*

The treatment process is described as follows: Zinc dross was placed in the heating crucible and the cover was tightly closed over the crucible with thermal cement to prevent any leakage. The furnace temperature is heated up to 600°C which is slightly above the melting temperature of Zn. This heating step is continued for sufficient time (30 min was enough) to allow the complete melting of the dross. The temperature is then raised above 900°C which typically the vaporization temperature of Zn. At this step, Zn dross melt is turned into Zn vapor leaving impurities, especially Fe which has much higher melting temperature. Zn vapors which pass through the silicon carbide tube and subjected to cooling (note that the longest part of silicon carbide is placed in open air). Upon cooling, Zn vapors are turned into Zn melt that poured into the second crucible where it is solidified and samples were taken for chemical analysis. Many trials have been carried out to determine the sufficient time for the whole dross to be evaporated. Time of 1.5 h was sufficient after complete melting of the zinc dross at 600°C. After finishing distillation, the system is left for cooling and then separated into its original components by removal of binding thermal cement. Distilled zinc either formed in the condensation (receiving) crucible or the SIC tube was collected (Fig. 2d), weighed and chemically analyzed. Remaining ash in the heating crucible was also taken for chemical analysis (Table 4).

Table 5 represents the results of pyrometallurgical Zn recovery at different temperatures. Almost pure Zn (99.95%) is obtained using this method. The recovery of Zn and its percent was largely dependent on temperature. No zinc was obtained below 900°C. Zinc recovery of 52% was obtained at 950°C. Further increase in temperature up to 1000°C significantly increase zinc recovery up to 73%. The highest recovery of 82% was obtained at 1050°C which can be related to the high amount of heat energy gained at this temperature.

The effect of time on the percent recovery of zinc was investigated while the temperature was raised and fixed at 1050°C after complete melting of the zinc dross at 600°C. Table 6 shows the results. 1 h was insufficient to give the desired percent recovery where only 68% recovery was obtained. Increasing the time up to 1.5 h increases the percent recovery up to 82%. Further increase of time towards 2 h and 2.5 h does not bring a pronounced change in percent recovery where 82.5% and 82.7% recovery were obtained. ICP Chemical analysis of produced zinc and impurities of Fe and Al is shown in Table 6. The percent purity of the recovered zinc is 99.95%. It is also interesting to see that the chemical analysis of the residue in the distillation crucible represents 5.64% Fe, 43.79 Al and 0.11% Zn. The developed pyrometallurgical system is quite simple giving the facility for investigation of important parameters that affect the pyrometallurgical recovery of zinc. The most important parameters are temperature and time.

**TABLE 4. Chemical analysis of Zn formed after lab scale pyrometallurgical experiments performed at 1050°C.**

| Metal % | %Fe | %Al | %Zn |
|---|---|---|---|
| Zn produced in the receiving crucible | 0.028 | 0.015 | 99.95 |
| Zn found in the SiC tube | 0.053 | 0.04 | 99.91 |
| Powder in the receiving crucible | 0.18 | 0.11 | 99.7 |
| Ash remained in the heating crucible | 0.11 | 43.79 | 5.64 |
| | The remaining percent is for slag | | |

**TABLE 5. Effect of temperature on the percent recovery of zinc using pyrometallurgical technique.**

| Temperature/°C | Time/h | Wt of Zn Dross/g | Wt of Zn produced/g | % of Zn Recovery |
|---|---|---|---|---|
| 900 | 1.5 | 1000 | - | - |
| 950 | 1.5 | 1000 | 520 | 52 |
| 1000 | 1.5 | 1000 | 730 | 73 |
| 1050 | 1.5 | 1000 | 820 | 82 |

**TABLE 6. The effect of time on the percent recovery of zinc using pyrometallurgical technique.**

| Temperature/ºC | Time/h | Wt of Zn Dross/g | Wt of Zn produced | % of Zn Recovery |
|---|---|---|---|---|
| 1050 | 1.0 | 1000 | 680 | 68 |
| 1050 | 1.5 | 1000 | 820 | 82 |
| 1050 | 2.0 | 1000 | 825 | 82.5 |
| 1050 | 2.5 | 1000 | 827 | 82.7 |

## Conclusions

In the electrowinning experiments, the highest current efficiency was obtained for the lowest current density (40 mA cm$^{-2}$). In contrary, the efficiency of zinc recovery in the electrorefining experiments was increased with the increase of current up to 6 A where the efficiency is largely increased.

The recovery of Zn using lab scale pyrometallurgical technique was largely dependent on temperature. No zinc was obtained below 950ºC. The highest recovery was obtained after 1.5 hour at 1050ºC which can be related to the high amount of heat energy gained at this temperature.

Comparing electrochemical methods (electrowinning, electrofinning) to the pyrometallurgical method reveals the following facts:

- Electrochemical methods give almost pure zinc (100 % pure zinc).
- Electrochemical methods take a long time for complete zinc electrodeposition.
- Large scale electrochemical method requires large space for the deposition path and also consumes large amounts of electricity.
- Pyrometallurgical method is a direct method while electrochemical methods require a big effort for the preparation of electrodeposition path.

Pyrometallurgical method is considered quick, requires relatively small space and considered economic regarding energy consumption because the furnace can work with natural gas.

## Acknowledgment

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## استرجاع الزنك من مخلفات مصانع الجلفنة باستخدام الطرق الكهروكيميائية والحرارية

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تناول البحث دراسة استرجاع الزنك من مخلفات مصانع الجلفنة بالطرق الكهروكيميائية و الحرارية، حيث تم الحصول على زنك نقي بنسبة ١٠٠٪ تقريبا. وقد تم تطوير نظام معملي مصغر لاستعادة الزنك بالطرق الحرارية حيث يسخن الزنك إلى درجة التبخر فى بوتقة داخل فرن ثم يتم سحب هذا البخار وتكثيفه فى بوتقة خارجية. وقد تم الحصول على زنك نقاوة ٩٩٫٩٧٪ بهذه الطريقة. و أيضا تم دراسة استرجاع الزنك باستخدام تقنية الترسيب الكهربي من حوض كبريتات الزنك الحامضية والتى نتجت عن إذابة الزنك الغير نقى في حامض الكبريتيك المركز. كما تم تنقية الزنك باستخدامه كأنود في حوض الأمونيا القلوي وترسيبه كهربيا على كاثود من الالومنيوم. في كلا الطريقتين تمت دراسة العوامل المختلفة من كثافة التيار والوقت اللازم للترسيب كما تم الحصول على بودرة الزنك بنقاوة ١٠٠٪ تقريبا.