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## SOLVENT EXTRACTION REAGENT ENTRAINMENT EFFECTS ON ZINC ELECTROWINNING FROM WASTE OXIDE LEACH SOLUTIONS

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### ABSTRACT

*A considerable quantity of metal-containing waste oxide is generated by the primary metal industry, and under increased environmental regulation, treatment of these wastes has become viable. Hydrometallurgical processing is often appropriate and one potential treatment process for zinc oxide wastes involves leaching the oxide with an acid, purifying by a solvent extraction operation, and recovering metallic zinc by electrowinning the purified leach solution. The purification stage is frequently critical and the introduction of a solvent extraction organic reagent into the flowsheet can be detrimental to downstream processes. The object of this study was to determine the effect of organics, entrained during solution purification, on the zinc electrowinning efficiency.*

*Zinc solvent extraction / electrowinning studies were made using di-2-ethylhexyl phosphoric acid (D2EHPA), diluted with kerosene, as the organic extractant. The stripping of the zinc from the organic phase was achieved using spent electrolyte solution. Multiple stages of zinc solvent extraction tests were performed to produce a strong acid electrolyte suitable for electrowinning tests. The entrainment of the organic extractant, from the solvent extraction stage, in the loaded spent electrolyte was found to significantly affect the current efficiency, and to change the zinc deposit morphology and orientation with concurrent modifications to the polarization behavior of the system being noted. Purification of the contaminated electrolyte with activated carbon improved the current efficiency and deposit morphology, and decreased the polarization to acceptable levels.*

*The conclusions are that the detrimental effects of entrained organic can be eliminated, and that solvent extraction processing is a viable method for the treatment of zinc containing waste oxides.*

### Keywords

Solvent extraction, electrowinning, waste zinc oxides, organic entrainment

### INTRODUCTION

The unit operations and processes employed in extractive metallurgy often produce large amounts of waste materials such as flue dusts, fumes, slags, and leach residues. There is a growing need for recycling and extraction of metals from such materials, especially zinc bearing hazardous materials [1]. Recycling of zinc wastes is important and necessary to minimize existing and future waste materials and to recover valuable metals.

Currently, these materials are treated, initially, by pyrometallurgical processes to separate iron compounds from volatile elements (Zn, Cd, Pb, and others), which form condensed fumes. Such fumes may then be treated hydrometallurgically to recover the zinc and possibly the other metal values. The typical hydrometallurgical unit operations for treatment of fume to recover zinc, comprise leaching, purification of the leach pregnant solution by precipitation (cementation), and finally electrolysis to produce metallic zinc.

The purification of the leach solution is a crucial stage in the recovery of zinc from wastes, as the impurities present in waste materials differ markedly in type and concentration from those encountered when leaching ores. Thus it is appropriate to consider purification processes other than precipitation. Solvent extraction (SX) offers several attractive features as a process for leach solution purification. It has been shown to be highly selective and very efficient in many areas of metals processing.

Solvent extraction involves the selective mass transfer of metal species between aqueous solutions and organic reagents. There are four main types of organic extractants and typically the organic extractant is dissolved in a carrier or diluent to enhance dispersion of the reagent [2,3]. Cation exchange extractants are used for uranium, thallium, the actinides and the rare earths. An example of such an extractant is di-2-ethylhexyl phosphoric acid/kerosene, which can extract a variety of metals from sulfate solution, with selectivity being controlled by adjustment of solution pH. In addition the pH also controls the direction of the mass transfer of ions between phases. Equation (1) details the accepted reaction for metal (M) extraction by a cation exchange organic (R).



Once the extraction is complete, the aqueous phase is termed the raffinate, and contains the impurities that could be detrimental to subsequent metal recovery processes such as electrowinning. The desired metal ions should be present in the loaded organic phase, and to recover these ions, an aqueous stripping solution is contacted with the loaded organic producing a pregnant aqueous solution and a stripped organic phase for reuse. Thus the desired metal ions are selectively passed via the organic phase to an electrolyte suitable for electrowinning. Solvent extraction of copper and zinc are industrially proven technologies [4,5] for the purification of leach solutions produced from various ores. A process for the production of electrolytic zinc from secondary raw materials has been patented [6] and is being implemented.

The pregnant solution, produced by the stripping stage of solvent extraction, must be free from impurities to permit efficient metal recovery by electrowinning. It is considered that solvent extraction is an appropriate purification stage for leach solutions produced from waste materials, but there are possible difficulties for zinc recovery that must be examined. Since the active molecules of the organic extractant are ionic, and due to the intimate phase mixing in the loading/stripping stages, occlusion of the organic in the electrolyte is possible. This could detrimentally affect the electrolytic recovery of zinc in a subsequent process.

There is little information in the published literature relating to the effect of the organic extractant on the zinc electrowinning process. One study [7] has suggested that small amounts of pure organic extractant may be a problem in zinc electrowinning. However, more information is required to determine the extent of the possible problem in the treatment of zinc waste oxides. This problem may well be significant when solution purification involves multiple stages of extraction and stripping.

There are several techniques commonly used to characterize and evaluate the electrolysis process in laboratory research. The methods include cell current efficiency, cyclic voltammetry, and studies of the morphology and orientation of the metal deposits by scanning electron microscopy and x-ray diffraction respectively.

Cyclic voltammetry is a useful technique for measuring zinc deposition polarization effects [8,9], and a typical test applies a controlled potential to an electrolysis cell and monitors the

current density. The resulting plot of potential against current density provides insightful information pertaining to the deposition of zinc.

The morphology and the orientation of the electrodeposited metal are of considerable technical importance [10]. The deposit is affected by the nature of the electrolyte, its hydrogen ion concentration, the nature and condition of the cathode blank, the electrolyte concentration, temperature, and impurities. It is possible to associate a given deposit morphology with a particular polarization or overpotential [11].

Activated carbon has been used as an absorbent in the extractive metallurgy industry for many years. Its porous structure and enormous surface areas accumulate and retain a broad variety of substances. A preliminary study of the effect of activated carbon in the purification of electrolytes, contaminated by organic extractant, has been reported [7].

In this study the results of preliminary solvent extraction/electrowinning tests, with pure zinc sulfate at high zinc concentrations, are presented. The tests were performed with the organic extractant, di-2-ethylhexyl phosphoric acid, with a kerosene diluent, and the loaded organic was stripped with a spent electrolyte produced in previous electrowinning tests. The loaded spent electrolytes were then electrowon to produce zinc metal.

The solvent extraction/electrowinning tests were conducted to determine the effect of organic entrainment in the electrolyte on the current efficiency, deposit structure and polarization behavior of zinc electrolysis. The results for the purification of the loaded spent electrolyte with activated carbon are also presented.

## EXPERIMENTAL

In order to investigate the possible effects of entrained organics on the subsequent electrowinning characteristics of zinc, it was necessary to determine the zinc loading capacities of the organic phase by solvent extraction. To prepare electrolytes suitable for electrowinning, it was also necessary to determine an extraction/stripping process to give solutions containing approximately 50g/l zinc.

### Electrolyte Preparation by Solvent Extraction

#### *a. Procedure*

The extraction and stripping stages of solvent extraction were undertaken in glass flasks utilizing ceramic stirrers, and small volumes of organic and aqueous phases, typically 50-300 cc. Phase separation, after mixing for 15 to 30 minutes, was achieved with thistle funnels, and all zinc analyses were determined using a Perkins Elmer 560 Atomic Absorption Spectrophotometer. The zinc content in the organic phase was analyzed by X-ray fluorescence, using a Portable X-ray Spectrograph "PORTASPEC" model 2510.

The organic extractant, D2EHPA was obtained from Albright & Wilson Americas, Inc., with a purity of approximately 95%. Kerosene, the diluent, was supplied by Ashland Chemicals Co. The aqueous solution was prepared from a 200 g/l zinc neutral solution made from French Process zinc oxide. Extractant concentrations of 15% and 20% were utilized in the tests. Sodium hydroxide from Fisher Scientific, or zinc oxide, were used to adjust the pH of the aqueous solution when necessary.

#### *b. Tests*

i) Organic Loading - A series of solvent extraction (SX) tests was performed with 20% D2EHPA in kerosene to determine the zinc loading in the organic phase as a function of aqueous feed concentration (g/l Zn) and final pH. The concentrations of the aqueous solution were 5, 20, and 51 g/l zinc at an initial pH of 3.0.

After each contact of the aqueous and organic phases, the pH of the aqueous raffinate was adjusted with sodium hydroxide to the initial pH of 3.0, and again it was contacted with the loaded organic for a further 15 minutes. This procedure was repeated until the pH of the raffinate attained the desired level, and then the organic loading was determined.

ii) Multi Stage Solvent Extraction and Stripping Tests- To examine the effect of the number of organic/aqueous contacts on the subsequent electrowinning process, zinc sulfate solutions were subjected to two and four stage SX operations. The two stage extraction, illustrated in Figure 1, was performed with 300 cc of 15% D2EHPA organic solution, and 300 cc of 50.3 g/l of zinc solution at pH 3.0, being contacted for 30 minutes. After phase separation, the pH of the raffinate was adjusted to 3.0 with sodium hydroxide, and the extraction process was repeated with the loaded organic to produce a final raffinate pH of 1.9. In the second SX stage, a new aqueous solution (50 g/l zinc) was put in contact with the loaded organic from the first stage, and the same procedure was repeated until the equilibrium pH of 1.9 was achieved. In this way the maximum loading capacity of the 15% D2EHPA was attained.

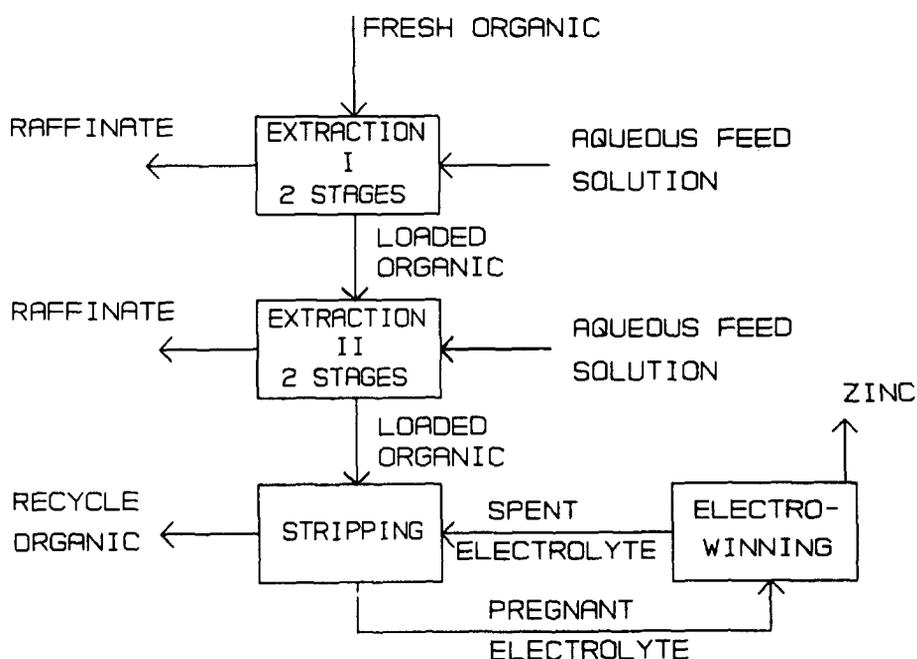


Fig.1 Flowsheet for the solvent extraction-stripping-electrowinning of zinc

For the stripping test, 200 cc of the loaded organic was contacted with 200 cc of spent electrolyte (approximately 160 g/l  $H_2SO_4$  and 38 g/l zinc) from a previous zinc electrowinning test. The stripping was undertaken for 30 minutes, and the procedure was designed to raise the zinc level of the electrolyte to approximately 50 g/l for subsequent electrowinning tests.

The cross-flow SX cascade method (Figure 2) was used to perform a four stage test with a 20% D2EHPA extractant in kerosene. In this method each stage behaved as an individual batch process with 30 minute contact, and an average aqueous to organic ratio (A/O) of 1.3. A single aqueous feed was contacted with successive fresh organic samples to permit an extraction of 90% to be obtained. The raffinate from each stage was adjusted to a pH level of 3.0 before being fed into the next extraction stage. The initial stripping solution contained 160 g/l  $H_2SO_4$ , and it was passed from one stripping stage to the next with an average A/O ratio of 1.4. In this manner the loaded electrolyte zinc level was raised to approximately 50g/l for electrowinning.

## MULTI-STAGE SOLVENT EXTRACTION

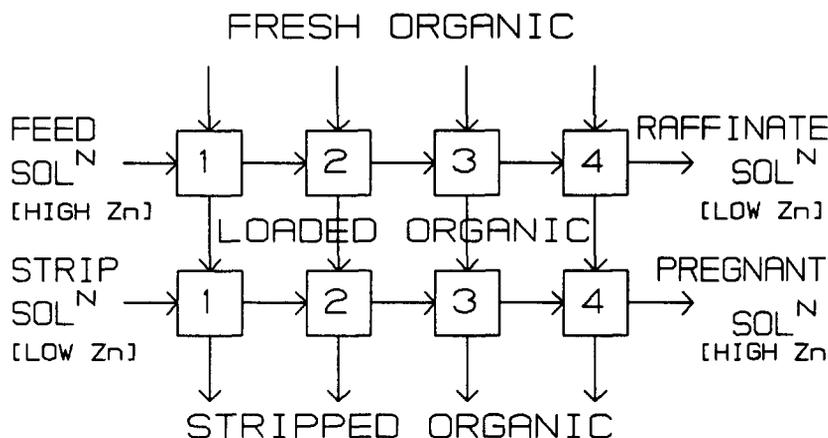


Fig.2 Flowsheet for the multi-stage solvent extraction-stripping and electrowinning of zinc for 20% D2EHPA

## Zinc Electrowinning

### a. Procedure

The electrolyte for the standard test was prepared by adding sulfuric acid to a neutral solution made from French Process zinc oxide giving a concentration of 50 g/l Zn and 150 g/l H<sub>2</sub>SO<sub>4</sub>. The electrolytic cells consisted of a 250 cc beaker covered with a plexiglass lid which had slots to mount the electrodes. Aluminium cathodes with a deposition area of 2 cm x 2 cm were used. The cathodes were polished with 600 grit emery paper, cleaned with distilled water in an ultrasonic bath, rinsed with deionized water, and dried with hot air. Conventional Pb-Ag anodes were used, and the distance between electrodes was 2.5 cm.

All electrolysis tests were run at a current density of 50 mA/cm<sup>2</sup> in a constant temperature bath at 35°C. At the end of electrolysis the cathode was removed from the cell, rinsed and dried. The deposits were then stripped and weighed, and the current efficiency was calculated from the theoretical and deposited weights.

Samples of the electrolytes produced by solvent extraction, and possibly containing dissolved organics, were also treated with activated carbon, prior to electrowinning. The procedure was to add 3 g of activated carbon into the electrolyte and to stir the mixture for 30 minutes at room temperature. The mixture was then filtered to remove the activated carbon and provide an electrolyte for the electrowinning tests. Two types of activated carbon were used; a) a granular form for electrolytes loaded from 15% D2EHPA, and b) a powder form for electrolytes loaded from 20% D2EHPA.

### b. Tests

i) Standard - To determine baseline operations, the standard electrolyte was electrowon to permit a standard current efficiency to be calculated. In addition electrowinning tests were also undertaken with additions of 25-100ppm D2EHPA, 20%D2EHPA and kerosene to the standard electrolyte.

ii) Multi-stage solvent extraction - The loaded stripping solutions from the two stage and four stage SX treatments were electrowon to determine the effect of organic contacts, and hence possible organic entrapment, on the current efficiency of subsequent zinc deposition.

iii) Electrolyte purification - Samples of electrolytes from i) and ii) were treated with activated carbon in an attempt to remove any entrained organic phase. The resulting purified solutions were then electroplated.

## Zinc Deposit Evaluation

### *a. Procedure*

The deposits were examined by scanning electron microscopy (SEM) to determine surface morphology. X-ray diffraction techniques were used for identification of grain orientation.

Cyclic voltammetry tests were performed to evaluate the effect of the organic extractant on the polarization behavior of zinc. The electrolytic cell consisted of three electrodes: an aluminum cathode with a deposition area of 2 cm x 2 cm was used as the working electrode, a platinum mesh as auxiliary electrode, and a saturated calomel electrode (SCE) as a reference. The tests were conducted using a Petrolyte Potentiodyne Analyzer (Model M-4100). The potential was cycled between -0.9 to -1.25 volts versus SCE at a potential sweep rate of 5 mV/s.

## RESULTS AND DISCUSSION

### Solvent Extraction

To permit a comparison of untreated electrolytes and electrolytes "purified" by solvent extraction, several tests were undertaken to identify appropriate conditions for the SX treatment.

#### *a. Organic Loading*

The results of the extraction tests for the aqueous solutions containing 5 - 51 g/l zinc, at different pH levels, are presented in Table 1 and in Figure 3. It can be seen that the extractant appears to have a loading capacity of approximately 15 g/l zinc, and that extraction increases with increasing raffinate pH for all zinc feed concentrations. The loading capacity of the extractant obviously limits high extraction rates to the lower zinc feed concentrations.

**TABLE 1 Organic loading as a function of zinc feed concentration and final pH**

Feed [Zn] g/l	Raffinate pH	Raffinate [Zn] g/l	Organic [Zn] g/l	Extraction %
5	1.1	2.2	2.99	56
5	1.2	1.6	3.4	68
5	1.7	0.7	4.4	86
5	1.9	0.5	4.6	89
5	2.0	0.3	4.8	94
20	1.1	16.0	4.4	22
20	1.2	14.4	6.0	29
20	1.4	11.6	8.7	43
20	1.6	11.2	9.2	45
20	2.1	5.5	14.9	73
51	1.0	45.9	5.1	10
51	1.2	43.9	7.1	14
51	1.4	42.9	8.3	16
51	1.9	36.2	14.7	29

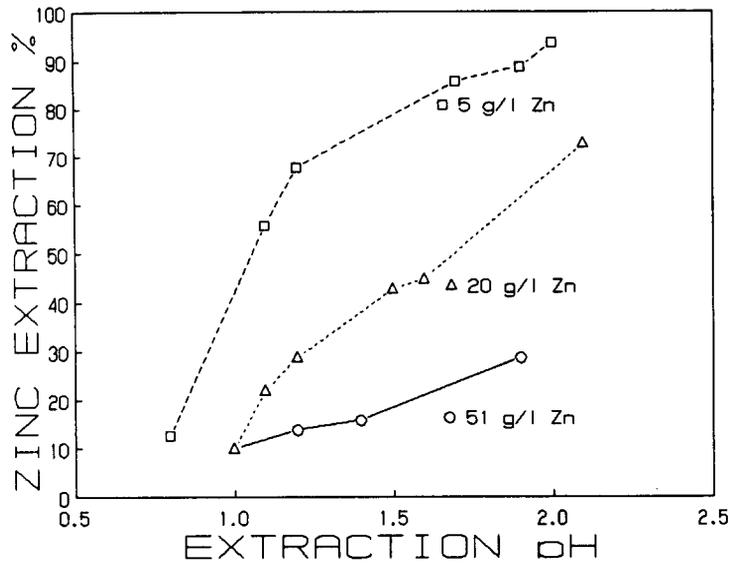


Fig.3 Percent extraction of zinc as a function of the equilibrium pH at different aqueous concentrations

As D2EHPA essentially exchanges hydrogen for zinc ions during the extraction, it is expected that higher extractions will be evident as pH increases. To enable effective extraction of zinc from aqueous feed solutions containing zinc levels in excess of 15 g/l, multiple stage solvent extraction must be utilized.

#### b. Multi-stage solvent extraction

The results of the two and four stage extraction and stripping tests are given in Tables 2 and 3. It can be seen, in Table 2, that the organic phase is 73% loaded in a single stage, and that the zinc recovery is relatively low. The second stage of extraction increased the loading a further 20%, but the increase in zinc recovery was marginal. The stripping efficiency of the loaded organic, by spent electrolyte containing 37.8g/l zinc, is shown in Table 3 to be 94%, with a residual zinc level in the stripped organic of 0.7 g/l. However the loaded electrolyte zinc concentration was 47.6 g/l, which was suitable for subsequent electrowinning assessment.

TABLE 2 Multi-stage treatment - extraction

Stage	Feed [Zn] g/l		Product [Zn] g/l		Extraction %
	Aqueous	Organic	Aqueous	Organic	
I	50.3	0.0	39.0	11.0	22
II	50.3	11.0	47.1	13.9	5
I	52.5	0.0	42.4	15.2	19
II	42.4	0.0	31.2	15.1	26
III	31.2	0.0	18.2	14.9	42
IV	18.2	0.0	6.2	14.8	66

**TABLE 3 Multi-stage treatment - stripping**

No. of Stages	Feed [Zn] g/l		Product [Zn] g/l		Stripping %
	Aqueous	Organic	Aqueous	Organic	
2	37.8	13.9	47.6	0.7	94
4					
I	0.0	15.2	9.5	0.4	97
II	9.5	15.1	19.9	0.4	98
III	19.9	14.9	31.5	0.4	97
IV	31.5	14.8	42.9	0.5	97

In the four stage extraction process, the single stage loading of the organic phase can be seen (Table 2) to be virtually 100%, but the zinc recovery is still very low. However with four extraction stages, the concentration of the raffinate phase was reduced to 6.22 g/l zinc, with a final zinc recovery of 66%. Table 3 shows that the stripping process gave a loaded aqueous phase containing 42.9g/l zinc, with an average stripped organic analysis of 0.45 g/l zinc. The average stripping efficiency exceeded 97%.

It is apparent that the extraction efficiency and loaded aqueous zinc concentration can be improved with additional stages, but the two and four contact tests were adequate to provide the feed electrolytes to investigate the effect of entrained organic on zinc deposition.

### Electrowinning of Loaded Electrolytes

#### *a. Standard*

The electrowinning of a standard electrolyte solution, containing 50 g/l zinc, produced a current efficiency of 93.4% (Table 4), and this figure is utilized as a base for comparison of subsequent tests.

**TABLE 4 Electrowinning of loaded electrolytes**

Electrolyte	[Zn] g/l	Current Efficiency
Standard	50.0	93.4
Two Stage	47.8	82.8
Four Stage	50.0	86.2

Preliminary indications of the effect of entrained organics on electrowinning current efficiency, are given in Table 5. It is evident that 100ppm additions of D2EHPA significantly reduced the electrowinning current efficiency, and that kerosene did not have an effect. Therefore, it appears that only the organic extractant D2EHPA has a negative effect on the electrowinning process.

#### *b. Multi-stage*

The results for the electrowinning of the loaded electrolyte produced by two stage solvent extraction are also summarized in Table 4. It can be seen that the current efficiency has decreased from 93.4%, for the standard electrolyte, to 82.8% for the two stage electrolyte. Although the zinc contents of the two electrolytes are slightly different, the major difference is the contact of the second electrolyte with the organic extractant.

**TABLE 5 Electrowinning of electrolytes doped with organics**

Organic Addition ppm	Current Efficiency %		
	D2EHPA	D2EHPA/Kerosene	Kerosene
0	92.7	92.6	92.7
25	87.5	91.7	92.7
50	85.9	88.8	92.7
100	85.1	85.6	92.7

The loaded electrolyte from the four stage SX tests with 20% D2EHPA gave a current efficiency of 86.2% (Table 4). Thus it is apparent that multi-stage contacting reduced the current efficiency and that two to four stages of contact produced results roughly equivalent to those determined for the standard electrolyte doped with 100ppm D2EHPA extractant (Table 5).

### *c. Purified Electrolyte*

Activated carbon was used to remove any organics entrained in the electrolytes treated by SX. The results are presented in Table 6, and it can be seen that the current efficiency increased for the two and four stage extractions after electrolyte purification with activated carbon. However, the removal of the entrained organic in the electrolyte, after four stripping stages, was more difficult than for the two stage extraction. The activated carbon treatment had no effect on the deposition of zinc from the pure electrolyte.

**TABLE 6 Electrowinning of purified electrolytes**

Electrolyte	Current Efficiency %
Standard	93.4
Two stage	91.2
Four Stage	89.1

### **Cyclic Voltammetry Tests**

Cyclic voltammetry techniques were used to evaluate the polarization behavior of the standard electrolyte, and the electrolytes contaminated with organic extractant.

#### *a. Standard electrolyte*

A typical voltammogram obtained for zinc deposition from the standard electrolyte is shown in Figure 4. The potential is initiated at point A, and the curve ABC indicates hydrogen evolution on the aluminum substrate. The curve CD represents the simultaneous evolution of hydrogen and deposition of zinc onto the aluminum cathode. At point D, the applied potential is reversed. The curve DB shows the polarization overpotential associated with zinc deposition onto freshly deposited zinc, and line BE represents the crossover potential. Finally the curve EF corresponds to the anodic dissolution of the previously deposited zinc.

Polarization curves showing the effect of 100ppm additions of D2EHPA and kerosene to the standard electrolyte, on zinc deposition, are presented in Figures 5 and 6. The D2EHPA caused a polarizing effect, which increased as the level of D2EHPA was increased. The addition of kerosene did not effect the polarization behavior, but a polarization effect was evident (Figure 7) for the 20% D2EHPA/kerosene mixture. Thus the detrimental effect of D2EHPA on the electrowinning process is supported by the polarization data. No changes in the anodic region were observed with any of the organics tested.

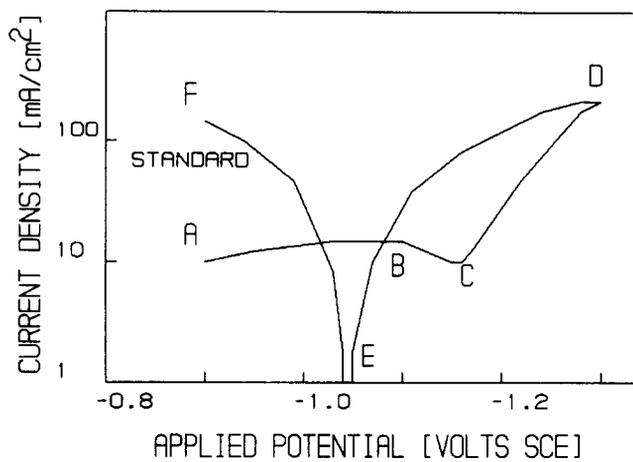


Fig.4 Cyclic voltammogram for non-addition electrolyte

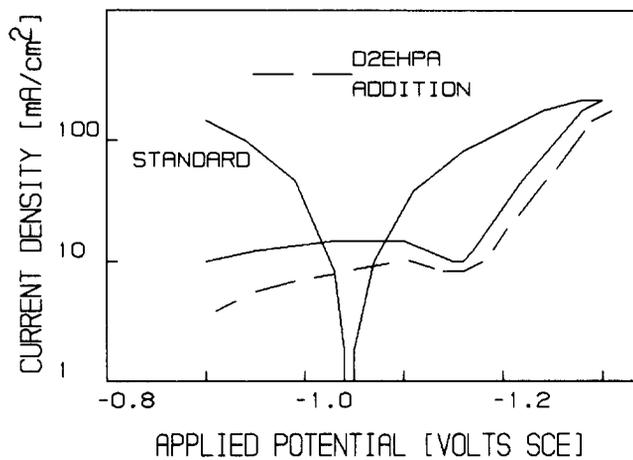


Fig.5 Cyclic voltammogram for electrolyte containing 100 ppm D2EHPA

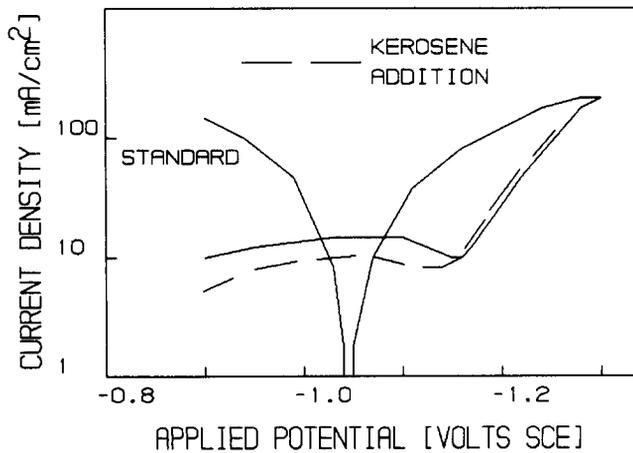


Fig.6 Cyclic voltammogram for electrolyte containing 100 ppm kerosene

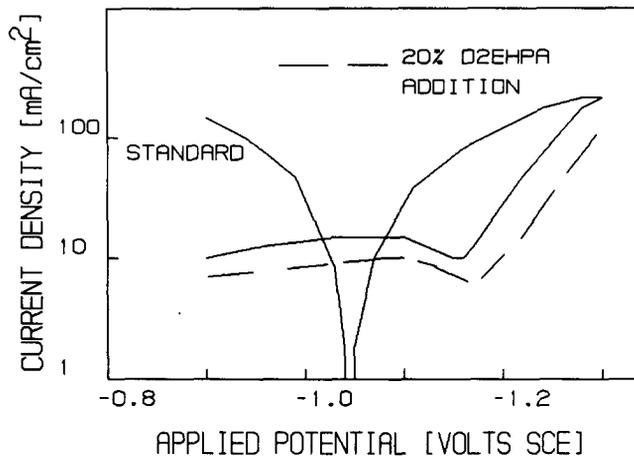


Fig.7 Cyclic voltammogram for electrolyte containing 100 ppm 20% D2EHPA

#### b. Multi-stage Tests

The polarization curve (Figure 8) for the electrolyte, loaded in the four stage extraction, shows significant polarization, and this also supports the detrimental effect of organics on the zinc deposition process. Again the level of polarization is roughly equivalent to that caused by a 100 ppm addition of D2EHPA to the standard electrolyte.

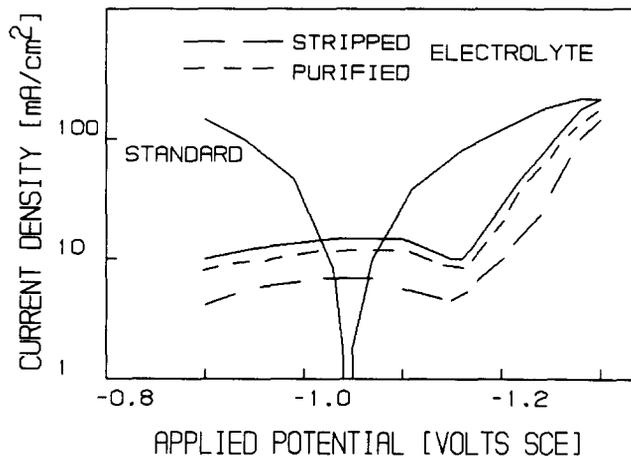


Fig.8 Cyclic voltammogram for stripped electrolyte (four stages) and purified electrolyte with powder activated carbon

#### c. Purified Electrolyte

It is evident from Figure 8 that the carbon purification of the four stage extraction electrolyte has reduced the degree of polarization. The voltammogram is not identical to that for the standard electrolyte, but it is close to it. This further supports the improved efficiency determination detailed above.

## Deposit Morphology and Orientation

### *a. Standard Electrolyte*

The zinc deposit morphology obtained from the addition free electrolyte, in the absence of organic extractant, is characterized by hexagonal platelets, which are illustrated in the SEM photomicrograph given in Figure 9. The orientation of the grains in this deposit is {002}, {101}, and {103}.



Fig.9 SEM photomicrograph for a deposit obtained with addition free electrolyte

With an addition of 50 ppm of pure D2EHPA, the zinc deposit morphology altered dramatically, and consisted of small platelets with rounded edges. The orientation was now {101}, {002}, and {103} (Figure 10), and it is evident that the organic addition had a significant effect on the deposit morphology.



Fig.10 SEM photomicrograph for a deposit obtained with electrolyte containing 50 ppm D2EHPA

### *b. Multi-stage Tests*

The deposit obtained with the loaded electrolyte, after four stages of extraction, also presented distinct differences in morphology. The finer grained deposit exhibited pitted platelets with rounded edges (Figure 11), and a orientation of {101}, {002} and {103}.



Fig.11 SEM photomicrograph for a deposit obtained from the electrolyte treated by four stage solvent extraction

### *c. Purified Electrolytes*

When the four stage loaded electrolyte was purified with activated carbon, the zinc deposit morphology (Figure 12) can be seen to be intermediate between the standard deposit (Figure 9) and the four stage deposit (Figure 11). It appears that further purification is needed to totally remove the effects of entrained organics.

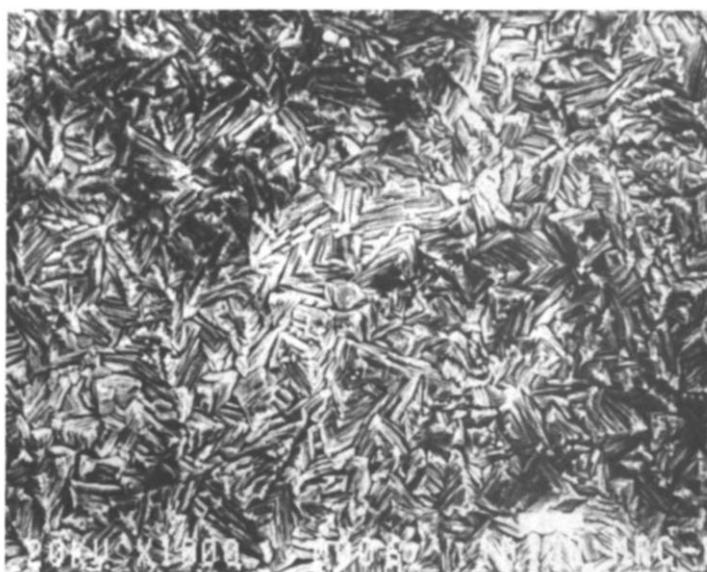


Fig.12 SEM photomicrograph for a deposit obtained from the electrolyte treated by SX and carbon purification

## CONCLUSIONS

1. It has been shown that the D2EHPA was capable of extracting zinc from aqueous solution with maximum loading capacity of the organic, at 20% D2EHPA, being 15 g/l zinc. Multi-stage processing was found to be capable of enabling appropriate levels of zinc extraction.
2. The loaded zinc organic was easily stripped with a strong acid solution of spent zinc sulfate from an electrowinning process. The average stripping efficiency was 94%, and the electrolyte was found to be suitable for electrowinning.
3. The organic extractant entrained in the spent electrolyte had an adverse effect on the electrowinning process, as evidenced by decreases in current efficiency, and by changes in the polarization behavior, and deposit morphology and orientation. Kerosene, the carrier used to dilute D2EHPA, did not affect the zinc electrolysis.
4. The purification of the electrolyte, contaminated with D2EHPA, using activated carbon, resulted in improved current efficiency. However, more than one purification stage with activated carbon may be necessary.

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