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Index at acceptance—70C3 + 5[LVIII(5)], 130G[XXXIII(7)].

PROVISIONAL SPECIFICATION

IMPROVEMENTS IN/OR RELATING TO RECOVERY OF ZINC VALUES FROM BY-PRODUCT ZINC COMPOUNDS.

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, RAJF MARG, NEW DELHI-1, INDIA, AN INDIAN REGISTERED BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES ACT (ACT XXI OF 1860)

The following specification describes the nature of this invention.

THIS IS AN INVENTION BY HANDADY VENKATAKRISHNAUDUPA, SCIENTIST, PENNAGARAM VYASA RAO VASUDEVA RAO, SCIENTIST AND RENGASWAMY VIJAYAVALLI, SENIOR SCIENTIFIC ASSISTANT, ALL INDIANS, ALL OF CENTRAL ELECTROCHEMICAL RESEARCH INSTITUTE, KARAIKUDI, S. RAILWAY,

This invention relates to improvements in or relating to recovery of zinc values from by-product zinc compounds such as the skimmings from galvanizing industry, wastes from the zinc oxide manufacturing plants and by-product zinc hydroxide or zinc oxide from the chemical industry.

Hitherto it has been found impossible to recover zinc values from the "galvaniser's ash" without incurring certain disadvantages such as iron contamination, difficulty of dissolution and deposition in order to avoid the impurities.

The object of this invention is to obviate these disadvantages by a novel electrolytic technique known as suspension electrolysis.

To these ends, the invention broadly consists in suspending the galvaniser's ash in a finely divided form (-150 mesh) in 5 to 40 per cent. strength of sodium hydroxide solution at a pre-determined ratio and electrolysis with mild steel, stainless steel or any other suitable anodes and mild steel, stainless steel, graphite, aluminium, titanium, or tantalum cathode. The nature of deposit obtained varies from a finely divided powder to a plate like sheet which can be easily peeled off the cathode substrate. The temperature at which electrolysis is carried out varies from 30°C to 60°C. The electrolysis is carried out in a container not attacked by concentrated sodium hydroxide solution. A mild steel tank would serve the purpose.

The finely divided galvaniser's ash (zinc content, 71 per cent.) is added periodically to replenish the deposited zinc. The deposit is removed at pre-determined intervals, washed free from alkali, dried and stored or cast into ingots.

The iron content of the zinc obtained was about 0.4 per cent. from an ash containing 4 per cent. iron and metallic zinc was above 97 per cent. The typical condition under which a sheet-like deposit was obtained is as follows :

Alkali concentration	. 20% by wt.
Current density	. 3.5 amp/dm ²
Cell voltage	. 2.5 V
Current efficiency	. 85 to 95%
Duration	. 7 hours
Thickness of deposit	. 0.5 mm
Weight of zinc obtained	. 29.0 g.

In a continuous electrolysis carried out by periodic addition of 300 g of ash, 199.0 g of zinc was recovered.

The following typical examples are given to illustrate the invention. In these experiments, 5 g of galvaniser's ash was added and electrolyzed.

Concentration of alkali (%)	Current density (amp/dm ²)	O-H voltage (volts)	Total current passed (a. p. hr)	Cathode efficiency (%)	Nature of deposit
5	5	2.75	2.92	55.0	Loose and powdery
10	5	2.75	3.6	72.1	Adherent and powdery.
20	5	2.45	2.92	75.8	Plate like
40	5	2.4	3.0	74.5	Powdery

The following are among the main advantages of the invention :

1. The galvaniser's ash and wastes from zinc oxide industry need not be separately converted to a soluble salt solution for electrolysis, thus avoiding the handling operations such as filtration, etc.
2. The impurities which are insoluble such as iron hydroxide, etc., remain in suspension without being included in the deposit and could occasionally be removed either by settling and decanting or by passing through suitable filters.
3. The process may be made continuous by suitable arrangement to feed the zinc compounds at pre-determined intervals.
4. Zinc metal can be obtained either in a plate-form or in a powder form by suitable adjustment of the experimental conditions.
5. Alkali could be used over and over again without any detrimental effect on the efficiency of recovery or purity of the metal obtained. Only the quantity of alkali lost in the handling operations will have to be made up.

R. BHASKAR PAI,

Patent Officer.

Council of Scientific and Industrial Research,

Dated this 19th day of April, 1965.

COMPLETE SPECIFICATION

IMPROVEMENTS IN/OR RELATING TO RECOVERY OF ZINC VALUES FROM BY-PRODUCT ZINC COMPOUNDS.

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, RAJF MARG, NEW DELHI-1, INDIA, AN INDIAN REGISTERED BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES ACT (ACT XXI OF 1860)

The following specification particularly describes and ascertains the nature of this invention and the manner in which it is to be performed.

THIS IS AN INVENTION BY HANDADY VENKATAKRISHNA UDUPA, SCIENTIST, PENNAGARAM VYASA RAO VASUDAVA RAO, SCIENTIST AND RENGASWAMY VIJAYAVALLI, SENIOR SCIENTIFIC ASSISTANT, ALL INDIANS, ALL OF CENTRAL ELECTROCHEMICAL RESEARCH INSTITUTE, KARAIKUDI, S. RAILWAY.

This invention relates to improvements in/or relating to recovery of zinc values from by-product zinc compounds such as the skimmings from galvanizing industry,

wastes from the zinc oxide manufacturing plants and by-product zinc hydroxide or zinc oxide from the chemical industry.

Price : TWO RUPEES.

Hitherto zinc values from galvanizer's waste have been recovered by electrodeposition from an aqueous solution of the zinc values after a suitable purification steps to avoid any contamination.

It has been found very difficult to recover zinc from galvanizer's waste without incurring certain disadvantages such as iron contamination, difficulty of dissolution and deposition in order to avoid the impurities.

The object of this invention is to obviate these disadvantages by a novel electrolytic technique known as 'suspension electrolysis'.

The galvanizer's waste is ground to a very fine powder suspended in a solution of sodium hydroxide and electrolysed using suitable electrodes. By this process, zinc could be obtained either in the form of a sheet or powder, depending on the experimental conditions. The major impurities like iron do not go into solution in the electrolyte and precipitated as ferric hydroxide. Though the ferric hydroxide will also be in suspension with the zinc values, in the electrolyte, it has been established that the iron content of the deposited zinc is as low as 0.15 per cent. It has also been established that the cell can be run continuously by frequent addition of powdered material in order to maintain the concentration of zinc in the suspension at a desired level. The accumulation of ferric hydroxide in the suspension was not found to affect the percentage iron content of the deposit during the course of continuous electrolysis for 30 days.

This method of recovery offers the following advantages over other methods :

- (1) The material need not be taken in aqueous solution for the electrolysis.
- (2) No purification step is necessary before electrolysis and the powdered waste can be directly used for electrolysis.
- (3) During electrolysis, the impurities like iron are precipitated and do not co-deposit with zinc.
- (4) The process can be made continuous with frequent additions of the powdered waste.
- (5) By adjusting the conditions of electrolysis, zinc could be obtained either as a sheet or powder.
- (6) In view of the above, a few-unit processes involved in other methods are eliminated.

The present invention consists of a process for the recovery of zinc from by-product zinc compounds such as the skimmings from galvanizing industry, wastes from the zinc oxide manufacturing plants and by-product zinc hydroxide or zinc oxide from the chemical industry which comprises of electrolysis of a suspension of powdered waste in sodium hydroxide solution wherein the electrolysis can be carried out continuously by frequent addition of the powdered zinc waste and no prior purification step is necessary to eliminate the impurities. The zinc can be obtained in either plate-form or powder containing iron as low as 0.15 per cent.

The recovery efficiency was found to be 95 per cent at an average current efficiency of 85 per cent.

This invention relates to the recovery of zinc values from by-product zinc compounds as well as from skimmings and wastes obtained in the galvanizing industry by the suspension electrolysis. In this process, a suspension of powdered material in sodium hydroxide solution is electrolyzed between mild steel anodes and stainless steel cathodes. During electrolysis, the finely powdered material is kept in uniform suspension by effective stirring.

Laboratory scale experiments: Rectangular cells made of PVC were used for the laboratory scale experiments. A 5 amp cell was run continuously for 35 days with periodic additions of the powdered waste at predetermined intervals. One stainless steel cathode and two mild steel anodes on either side of the cathode were used. The electrodes were tightly held in their positions by proper clip arrangement.

Large scale experiments: Experiments have been carried out with a 100 amp cell. The cell dimensions are given in Figs. 1 and 2 of the accompanying drawings. The cell (No. 4 in Fig. 5) was a cube of 38.0 cm length, breadth and height with two strips welded to two opposite sides so as to hold the connections and busbars and is made of mild steel. Three perforated mild

steel anodes (Nos. 6, 8 and 10 in Fig. 5) and two stainless steel cathodes (7 and 9 in Fig. 5) were connected to copper busbars 1 and 2 respectively. The busbars were fixed on to an insulating PVC strip (No. 3 in Fig. 5). The insulator in turn was fixed on to the two steel strips (No. 12(a) in Fig. 4) welded to the sides of the electrolysing tank. Two stirrers (No. 5 in the Fig. 5) were fitted diagonally opposite to each other and the rate of stirring was adjusted by adjusting the speed of the motor. The interelectrode distance was maintained at a constant value of 5 cm and the electrodes were held tight in their position by means of connecting clips (No. 12 in Fig. 4). The arrangement of each electrode is already shown in Fig. 4. The electrode plate was connected to a connecting strip by connecting screws (No. 11 in Fig. 4), which fitted into the clip tightly. The electrical contact for the electrode was provided by the copper busbars 1 and 2 in the Fig. 5. Electrode assembly is well represented in Figs. 3 and 5. The cross-sectional view of the cell is represented in Fig. 3. The anodes and cathodes are arranged alternating each other and are connected to common busbars No. 1 and 2 in Fig. 5 respectively. Fig. 5 represents the view from the top of the cell and indicates the positions of the electrodes and stirrers.

The following typical examples are given to illustrate the invention :

EXAMPLE 1.

In these experiments, 5 g. of galvanizer's ash was added to 300 ml of electrolyte and electrolysed :

Concentration of alkali (%)	Current density (amp/dm ²)	Cell voltage (volts)	Total current passed (amp-hr)	Current efficiency (%)	Nature of deposit
5	5	2.75	2.92	55.0	Loose and powdery
10	5	2.75	3.6	72.1	Adherent and powdery
20	5	2.45	2.92	75.8	Plate-like
40	5	2.4	3.0	74.5	Powdery

EXAMPLE 2.

In this, the cell was run continuously with periodic additions of the ash so as to maintain the zinc concentration in the electrolyte always at and above 50 per cent of the initial concentration :

Alkali concentration :	..	200 g/l
Current density :	..	5 amp/dm ²
Cell voltage :	..	2.5 volts
Current efficiency :	..	85—95%
Duration :	..	7 hours
Thickness of the deposit :	..	0.5 mm

Recovery efficiency for a continuous run of a cell for about a month was found to be 95 per cent.

EXAMPLE 3.

Electrolyte concentration :	..	200 g/l
Amount of material added :	..	100 g/l of solution
(a) Current density :	..	2.5 amp/dm ²
Duration :	..	5½ hours
Weight of deposit :	..	280 g.
Current efficiency :	..	83.62%
(b) Current density :	..	5 amp/dm ²
Duration :	..	5 hours
Weight of deposit :	..	555 g.
Current efficiency :	..	89.35%

The iron content of the deposit was found to be 0.15 to 0.2 per cent.

The following are the main advantages of the invention :

(a) The galvanizer's ash and wastes from zinc oxide industry need not be separately converted to a soluble salt solution for electrolysis, thus avoiding the handling operations such as filtration, etc.

(b) The impurities which are insoluble, such as iron hydroxide, etc., remain in suspension without being included in the deposit and could occasionally be removed either by settling or decanting or by passing through suitable filters.

(c) The process may be made continuous by suitable arrangement to feed the zinc compounds at predetermined intervals.

(d) Zinc metal can be obtained either in a plate-form or in a powder form by suitable adjustment of the experimental conditions.

(e) Alkali could be used over and over again without any detrimental effect on the efficiency of recovery of purity of the metal obtained. Only the quantity of alkali lost in the handling operations will have to be made up by adding fresh alkali.

To summarise, zinc can be recovered from by-product zinc compounds and skimmings and wastes obtained in the galvanizing industry by the process known as suspension electrolysis'. As the name implies, a suspension of finely powdered material in sodium hydroxide solution is electrolysed between mild steel anodes and stainless steel cathodes. During electrolysis, zinc alone goes into solution and gets deposited on the cathode whereas the impurities such as iron get precipitated. Though the iron hydroxide also remains in suspension, it is not included in the deposit during electrolysis. The zinc can be deposited either as a sheet or a powder, depending on the conditions of the experiment. The deposited zinc was found to contain very little amount of

iron as impurity which may be as low as 0.15 per cent. The process can be made continuous by frequent addition of the powdered material at predetermined intervals. In this process of recovery some of the unit processes such as precipitation and filtration of the impurities, etc., are eliminated and the material can be directly fed into the electrolysis tank without an intermediate step of purification. When the electrolysis was carried out using a sample of galvanizer's waste containing 4 per cent iron as impurity, it was found that the iron content of the deposited zinc was found to be only 0.4 per cent.

We claim :

1. A process for the electrolytic recovery of Zinc values from by-product zinc compounds such as skimmings from galvanizing industry, wastes from the zinc oxide manufacturing plants and by-product zinc hydroxide or oxide from the chemical industry, which comprises electrolysis of a suspension of powdered waste in sodium hydroxide solution, wherein the electrolysis can be carried out continuously by frequent addition of the powdered waste.

2. A process as claimed in Claim 1 wherein the conditions of electrolysis are suitably adjusted as illustrated in Example 1 to obtain zinc either in the form of a sheet or powder.

3. A process as claimed in Claim 1 or 2 wherein the alkaline electrolyte is used for electrolysis over and over again by adding the quantity of alkali lost in the handling operations.

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Council of Scientific and Industrial Research.

Dated this 10th day of February, 1966.

No. 99591

COMPLETE SPECIFICATION

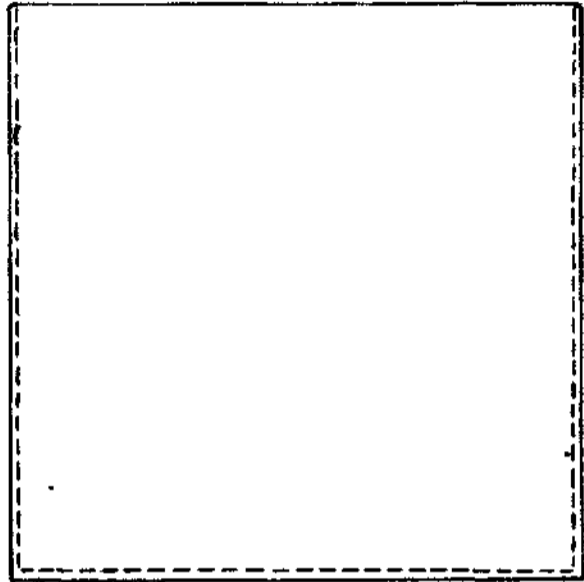


FIG. 1

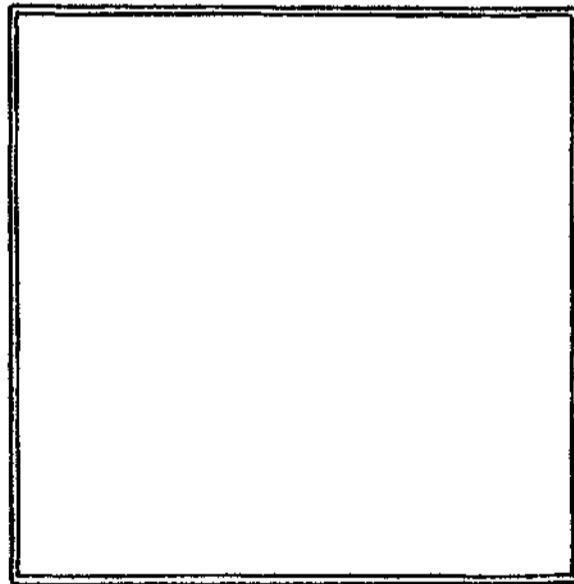


FIG-2

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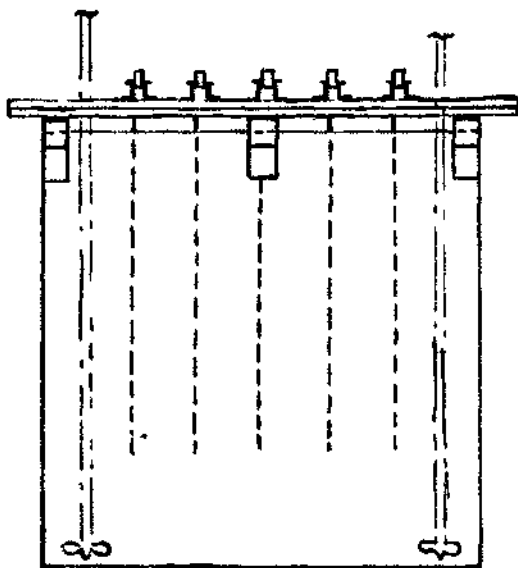


FIG-3

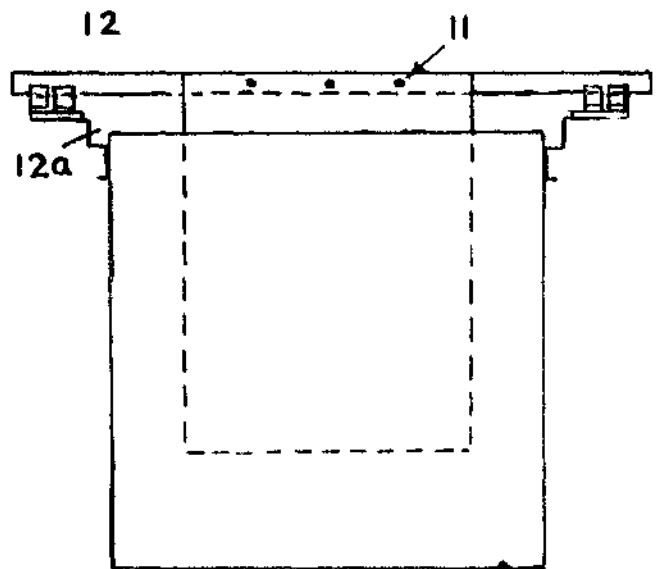


FIG-4

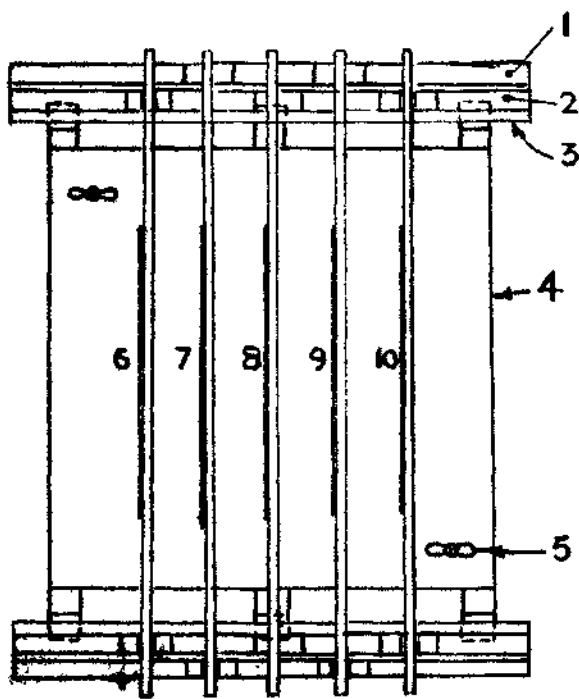


FIG-5

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