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Production and Recycling of
Non-Ferrous Metals: Saving Resources
for a Sustainable Future

Lead • Nickel / Cobalt / Vanadium •
Minor Metals • General Pyrometallurgy •
General Hydrometallurgy • Strategies for Resource Savings •
HSE practices and Legal Aspects

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Treatment of a leach liquor after leaching EAF dust

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Keywords: Electric arc furnace dust (EAF dust), hydrometallurgy, zinc, recycling, waste

Abstract

The work deals with recovery of metallic zinc from leach liquor after leaching EAF dust. The leach liquor after leaching EAF dust in sulfuric acid solutions, containing Fe 3.34 g/L, Zn 4.17 g/L, Ca 0.6 g/L and other minor metals (Cd, Pb, Cu), was processed by iron precipitation, cementation of other accompanying metals and subsequent zinc electrowinning. Goethite and jarosite processes were used as iron precipitation step. Influence of a neutralizing agent (1 M NaOH, CaCO₃ and lime milk), pH (2.5 – 4) and temperature (80 and 95 °C) on the process efficiency was investigated. The aim was to determine optimal conditions at which residual iron concentration after precipitation does not exceed 30 µg/ml; i.e. the limit for iron concentration in an electrolyte for zinc electrowinning. It was observed, that pH value and neutralizing agent have the most significant effect on the iron precipitation efficiency. Lime milk was determined as the most suitable neutralizing agent, where the residual iron concentration (80 °C, pH 4) was below 1 µg/ml, and representing precipitation efficiency of 99.98 %. After iron precipitation step, cementation of other impurities (Cd, Pb, and Cu) by zinc powder was carried out. The influence of pH and temperature on the cementation efficiency was investigated. The results showed that almost 100 % Cd and 90 % Pb can be removed by cementation. In zinc electrowinning step, the influence of temperature (20, 40 and 60 °C) and current density (333 – 1000 A/m²) on the current efficiency was studied. Maximum recovery of zinc (almost 99 %) was obtained at 40 °C and current density of 333 A/m². At these conditions, zinc was recovered as a compact plate with a minimum of zinc being precipitated in dendritic form. Results of this study show possibility to recover high purity product by processing impure zinc containing solution coming from EAF dust leaching.

1 Introduction

EAF dust is an interesting source of secondary metals, especially zinc. Zinc content in EAF dust ranges from 8 to 33 wt.-%, depending on the quality of an input material (i.e. steel scrap) [1 – 9]. A source of zinc in the input material is mostly represented by galvanized steel scrap. In addition to zinc, EAF dust contains up to 50 wt.-% of iron, about 3 wt.-% of calcium, 2 – 4 wt.-% of lead and other metals in a minor content. For comparison, the zinc content in primary raw material varies from 5 to 15 wt.-% [10]. Differences in chemical composition of EAF dust mainly depend on the differences in the chemical composition of the steel scrap, as well as on the type of steel being produced. Flue dust from the carbon steel production is characterized by a high content of zinc, while flue dust and sludge from alloy steel production are characterized by a high content of Cr, Ni, Mg, Mn and others [11 – 16]. From the mineralogical point of view iron in EAF dust is present mainly in oxidic form, as magnetite – Fe_3O_4 , and/or as franklinite – ZnFe_2O_4 . Zinc is present in two mineralogical forms, as zincite – ZnO and as franklinite – ZnFe_2O_4 or complex franklinite (Zn, Mn, Ca, ...) Fe_2O_4 [2].

In Slovakia, approximately 7000 tpy of EAF dust containing iron and zinc is produced. It is considered as hazardous waste under the current European legislation. Therefore, benefits from processing and recycling of EAF dust are clear: reduction of landfilling hazardous waste, gain of iron-rich concentrate suitable as an input to the pig iron and/or steel production, gain of zinc and its compounds as commercial products, or obtaining other present metals.

Methods of EAF dusts processing can be divided into three main groups: pyrometallurgical, hydrometallurgical and combined procedures. Pyrometallurgical technologies (i.e. Waelz process, Primus, Fastmet and others [17 – 22]) are based on the reduction of steel dust, where the objective is obtaining iron concentrate and zinc oxide. The disadvantages of pyrometallurgical technologies are particularly high energy demand, low product quality, low flexibility and necessity of pretreatment input material (e.g. pelletizing). Economic feasibility is achieved when over 100,000 – 120,000 tpy is processed [23].

Concerning the disadvantages and relatively small amount of dust produced in the Slovak Republic, hydrometallurgical processing can be considered as more appropriate procedure. Compared to pyrometallurgical processes, the main advantages of hydrometallurgical processing are: high flexibility, lower sensitivity to changes of the input material composition, lower investment and operating costs, lower energy consumption, high purity of the final product, etc. Hydrometallurgical processes are also considered as more environmental friendly. For EAF dust leaching, acid leaching agents are the most commonly used or investigated, especially H_2SO_4 , HCl , HNO_3 or acetic acid (CH_3COOH) [1, 2, 4, 5, 7, 8, 24 – 26]. For alkaline leaching of EAF dust, following leaching agents are usable: NaOH and $(\text{NH}_4)_2\text{CO}_3$ [25 – 31]. Some of the procedures have been developed and tested on a pilot scale; the most famous includes Ezinex [32], where alkaline leaching is used. The main aim of the leaching step is represented by dissolution of zinc into solution while iron remains in the insoluble residue. Output of the process is iron concentrate and zinc leach liquor intended for further pro-



cessing. However, zinc leach liquor can contain also small amount of iron and other impurities (Ca, Cd, Pb, etc.).

Due to presence of these impurities, obtained leach liquor has to be refined before zinc extraction. Zinc can be obtained from a refined solution by electrowinning as electrolytic zinc or by precipitation or crystallization as commercially valuable compounds. In the case of zinc electrowinning from refined leach liquor, the presence of impurities in the supercritical concentration can lead to a lower current efficiency, lower zinc quality and to contradictory process (oxidation and reduction) at the electrodes [33].

The basic methods for EAF dust leach liquor refining include precipitation and cementation steps. Precipitation is usually used as a method for iron removing; the limit iron concentration in leach liquor for subsequent zinc electrowinning is 20 – 30 $\mu\text{g/ml}$ [33]. In the case of acid sulfate solutions, iron can be precipitated as hematite – Fe_2O_3 , goethite – $\text{FeO}(\text{OH})$ and jarosite – $x[\text{Fe}_3(\text{SO}_4)_2(\text{OH})_6]$, where $x = \text{H}_3\text{O}^+$, Na^+ , K^+ or NH_4^+ [33 – 35].

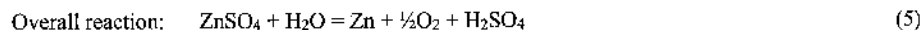
The precipitation processes differ by the temperature, pH, pressure, optionally the addition of agents promoting the formation of a particular phase. Hematite process takes place at temperature above 100 °C and at $\text{pH} > 2$. In the real commercial process, this process occurs at temperature above 180 °C, i.e. high-pressure process. In all developed processes of hematite precipitation, the first step is reduction of ferric ions to ferrous using $\text{SO}_2(\text{g})$ as a reducing agent, thereby promoting the precipitation of hematite. Obtained product can be theoretically used in cement production industry, in pig iron production, or as pigment, etc. The disadvantage of this process is its complexity (high pressure and temperature) and the high costs, which prevent its wider use in practice [33].

Goethite precipitation is carried out at $\text{pH} 2 – 2.5$, at 70 – 90 °C while oxygen is blowing into the solution due to oxidation of Fe^{2+} to Fe^{3+} . The amount of obtained precipitate is lower than in jarosite precipitation. The disadvantage is that goethite containing 40–45% Fe also contains a small amount of basic sulfate, which does not allow its use in the blast furnace [33].

Jarosite precipitation begins at $\text{pH} < 1$ and ends at $\text{pH} = 1.5 – 2$. Jarosite production and precipitation depends on the temperature. In practice, high temperature is used (95 – 180 °C) [33].

After separation of iron from zinc leach liquor, cementation of electropositive impurities such as cadmium, lead or copper is carried out. Cementation is necessary due to the fact that these metals can co-precipitate on the cathode together with zinc and thus affect its final purity. In this case, zinc powder is usually used for cementation of impurities [2]. Subsequently, after removal of these impurities and obtaining refined solution with high zinc content, zinc electrowinning can be applied.

Insoluble lead anode and aluminum cathode can be used in zinc electrowinning process. Pure metallic zinc is deposited on the cathode with hydrogen gas releasing (1, 2). At the same time, water is dissociated on the anode, what leads to free hydrogen ions formation and subsequently to oxygen gas releasing (3, 4). Zinc deposition is affected by electrode potential of metal.



The main goal of this study is to recover metallic zinc from purified leach liquor coming from acidic leaching EAF dust. Optimal conditions of iron precipitation, cementation and zinc electrowinning were investigated. These experimental works were carried out in laboratory scale. According proposed optimal conditions experimental works will continue in pilot plant scale, where the optimal conditions will be verified or modified. This work is only a part of a project which deals with complex EAF dust processing by hydrometallurgical way. Apart from acidic leaching and metals recovery from solution, project includes alkaline leaching as well.

2 Experimental

2.1 Material and methods

The leach liquor from leaching EAF dust in sulfuric acid, with metals content listed in table 2, was used for the study.

Table 2: Content of main metals in the leach liquor from leaching EAF dust, AAS

	Fe	Zn	Cd	Pb
[$\mu\text{g/ml}$]	3391	4170	18.9	5.1

Based on the preliminary experiments, following neutralizing agents were chosen for iron precipitation: 1 M NaOH, CaCO_3 and lime milk. Experiments of iron precipitation were carried out in the apparatus fitted with a thermostat, stirring and supplying neutralizing agent. The pH was determined using the pH meter Hanna pH213 equipped with a temperature correction. During jarosite precipitation process, $(\text{NH}_4)_2\text{SO}_4$ was added to the neutralizing agent to support jarosite formation. This additive was chosen based on literature review.

Conditions of iron precipitation experiments are listed in table 3.



Table 3: Experiment conditions of iron precipitation

Neutralizing agent	1M NaOH, CaCO_3 , lime milk*
Volume (for 1 experiment)	400 ml
Time	120 min
Temperature	80; 95 °C
pH during goethite process	3; 4
pH during jarosite process	2.5; 3.5
Additive for jarosite formation	$(\text{NH}_4)_2\text{SO}_4$
Stirring rate	300 rpm

* Lime milk was prepared as a suspension of CaO in distilled water in a ratio 200 g CaO/1 l.

Cementation of lead and cadmium was carried out after iron precipitation. Zinc powder in an amount of 0.5 g per 250 ml of leach liquor was used as cementation agent. Cementation was carried out in a glass reactor (figure 1) with stirring (300 rpm) for 60 minutes at 25, 50 and 80 °C, at pH = 4, 5 and 6. The aim of these experiments was to determine the optimal conditions (pH and temperature) for the cementation.

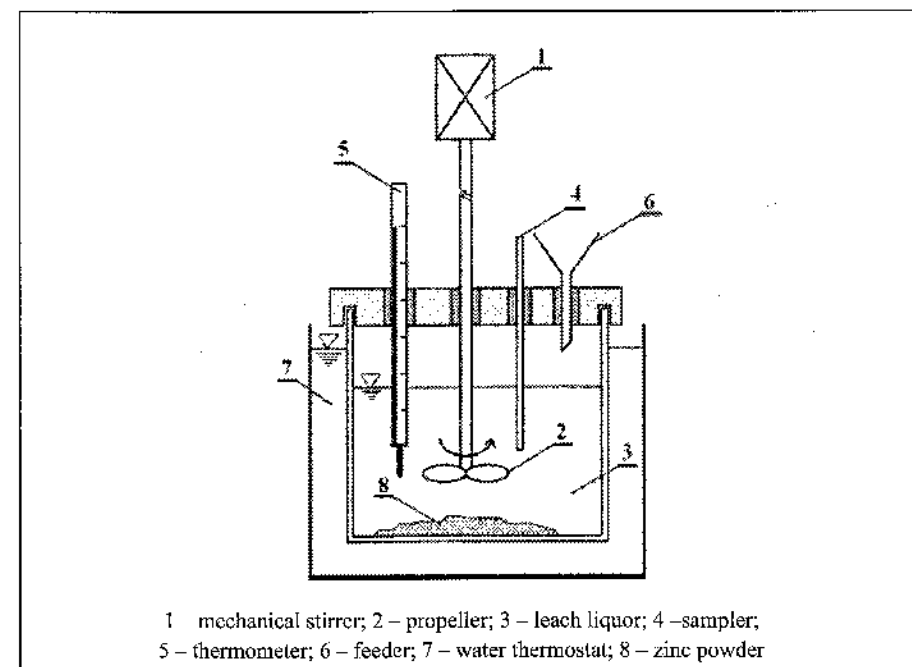


Figure 1: Schematic view of the apparatus for cementation [36]

Composition of obtained refined leach liquor was modified by evaporation to metal contents which are listed in table 4. The electrowinning was carried out in a laboratory electrowinning cell type EP-4. Aluminum cathode and lead anode were used during electrowinning. Zinc electrowinning was carried out at 20, 40 and 60 °C for 120 minutes. The distance between the electrodes was 30 mm and an active cathode area was 12 cm². In addition the effect of temperature, and current density ($J = 333, 500, 833 \text{ A/m}^2$) on the current efficiency was investigated.

Table 4: Metals content in the leach liquor before electrowinning

	Zn	Fe	Cd	Pb
[$\mu\text{g/ml}$]	50 000	1.8	0.05	0.55

Current efficiency was calculated based on the real and theoretical amount of deposited zinc. Theoretical amount was determined by Faraday's law:

$$\Delta m = \frac{I \cdot \Delta t}{z \cdot F} \cdot M \text{ [g]} \quad (1)$$

Where Δm is the amount of obtained substance, I is the electric current, t indicates the time, F is the Faraday constant ($F = 9.6485 \cdot 10^4 \text{ C} \cdot \text{mol}^{-1}$), z is the number of electrons necessary for deposition of one molecule of the substance, and M is the molar mass of the substance.

In order to determine purity of zinc and the amount of present impurities, the analysis of the deposited zinc metal was carried out. All liquid and solid samples were analyzed by atomic absorption spectrometry on a Varian AA-240

3 Result and discussion

3.1 Precipitation of iron

Figure 1 shows an effect of pH on iron precipitation during goethite and jarosite precipitation at 80 and 95 °C by various using precipitating agents. All achieved efficiencies and residual iron concentrations in leach liquor are listed in table 5.

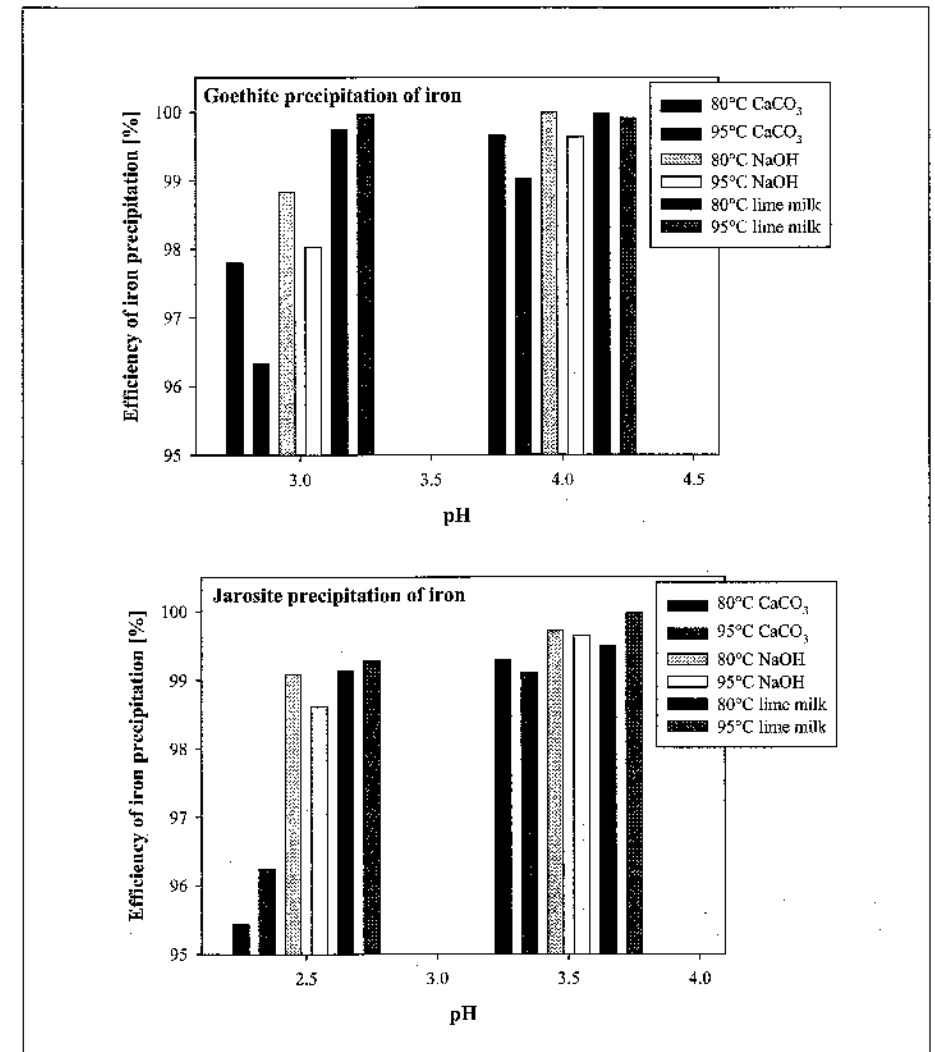


Figure 1: Efficiencies of iron precipitation during goethite and jarosite process

Table 5: Efficiencies of iron removal and residual iron concentrations in leach liquors

pH	80 °C		95 °C	
	c_{Fe} [$\mu\text{g/ml}$]	η [%]	c_{Fe} [$\mu\text{g/ml}$]	η [%]
Goethite precipitation by CaCO_3				
3	74.51	97.8	124.6	96.33
4	11.72	99.65	30.81	99.01
Goethite precipitation by NaOH				
3	39.26	98.84	66.89	98.03
4	9.68	99.99	12.51	99.63
Goethite precipitation by lime milk				
3	8.43	99.75	1.18	99.97
4	0.68	99.98	3.06	99.91
Jarosite precipitation by CaCO_3				
2.5	155	95.43	127.3	96.25
3.5	24.04	99.29	30.73	99.1
Jarosite precipitation by NaOH				
2.5	31.3	99.08	47.13	98.61
3.5	9.81	99.71	12.25	99.64
Jarosite precipitation by lime milk				
2.5	29.07	99.14	24.05	99.29
3.5	17.34	99.49	0.58	99.98

Iron precipitation efficiency was mainly influenced by pH and the type of neutralizing agent, while higher temperatures did not result in higher precipitation efficiency. As it was mentioned above, limit for iron concentration in leach liquor intended for zinc electrowinning is around 20–30 $\mu\text{g/ml}$ [33]. A higher concentration of iron in solution causes reduction of hydrogen pressure and leads to decreasing of overall current efficiency of zinc electrowinning. The final iron concentration in the leach liquor complies with the limit. The maximum removal of iron (almost 100 wt.-%, figure 1) was achieved by using lime milk as neutralizing agent, wherein the residual iron concentration in leach liquor was less than 1 $\mu\text{g/ml}$ at pH = 4. This neutralizing agent seems to be the most suitable in term of amount of removed iron as well as in term of high speed of filtration after iron precipitation.













3.2 Cementation of impurities

The results of cementation have shown a great possibility for cadmium removing (almost 100 wt.-%). At same time, it is possible to remove almost 90 wt.-% of lead. Additionally, if residual iron is present in leach liquor during cementation, it can be reduced under limit concentration. Optimal conditions for cementation of impurities were as follows: $t = 50\text{ }^\circ\text{C}$, $\text{pH} = 4 - 5$, 60 minutes.

3.3 Zinc electrowinning

Current efficiencies of zinc electrowinning at all temperatures and current densities are given in table 6, together with the pictures of the obtained zinc.

Table 6: Zinc deposits obtained by electrowinning and achieved current efficiencies

[°C]	Current density [A/m^2]			
	333	500	833	1000
20	 95.98 %	 95.90 %	 97.95 %	 83.62 %
40	 98.98 %	 95.90 %	 91.80 %	 84.02 %
60	 83.67 %	 71.23 %	 86.10 %	 70.31 %

* The percentages in correspond to the current efficiency under the listed conditions

Zinc was deposited as a compact sheet with partial formation of dendrites, figure 2, depending on the used current density.

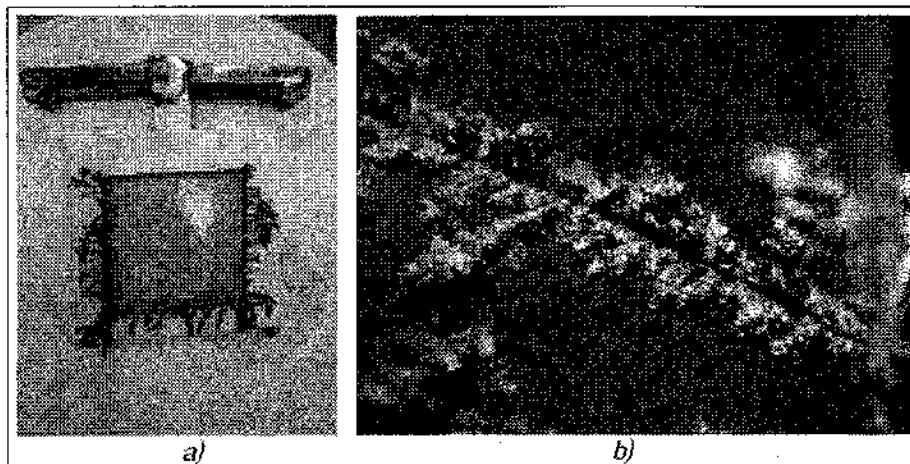


Figure 2: a) Zn cathode with dendritic deposit, b) dendrite at a magnification of 100 x

Results show that the highest current efficiency (over 95 %) was achieved at 20 and 40 °C, at current densities 333 and 500 A/m². The highest current efficiency (almost 99 %) was achieved at 40 °C and current density of 333 A/m². From table 6 also results that under conditions with higher current efficiencies, the formation of dendrites was the minimum.

Current efficiencies 92 – 99 % can be considered sufficient, since the literature indicates [23, 33] that economically viable zinc electrowinning process should achieve a current efficiency of 90 %.

Chemical analysis of obtained metallic zinc confirmed a high purity of final product (more than 99.5 %). Content of impurities in the final product was as follows: 0.06 wt.-% of iron, 0.03 wt.-% of cadmium, 0.08 % of copper and 0.02 wt.-% of lead.

4 Summary

The work deals with the recovering metallic zinc from the leach liquor coming from acidic leaching of EAF dust. Due to the fact that EAF dust composition depends on input material with various metals content, the composition of EAF dust is highly individual. It follows that leach liquor coming from EAF dust leaching has various composition as well. For that reason, it is impossible to apply a universal technology for its processing. Studied EAF dust is characterized by high zinc and iron content as zincite and/or franklinite and by a presence of other non-ferrous metals, such as lead and cadmium, etc. The presence of these impurities must be taken into account during leaching and subsequent leach liquor treatment.

The main aim during EAF leaching is zinc conversion into solution. However, except zinc, obtained leach liquor contains also significant amount of other impurities (Fe, Pb, Cd,...) which can have negative effect on the purity of deposited zinc and processing costs. Iron removal step was per-

formed by well-known precipitation techniques (jarosite and goethite). Goethite precipitation with using lime milk as a neutralizing agent showed the best results in terms of precipitation efficiency. At optimal conditions (pH = 4, 80 °C), residual iron concentration was less than 1 µg/ml.

Subsequently, iron-depleted leach liquor was subjected to the cementation with zinc powder. Under optimal conditions: $t = 50$ °C, pH = 4 – 5, and time of 60 minutes, nearly 100 % of Cd and 90 % of Pb was removed. Last step was zinc electrowinning, where the highest current efficiency of almost 99 % can be achieved after 120 minutes, at 40 °C and 333 A/m². In this case, metallic zinc with high purity (more than 99.5 %) was obtained.

Obtained metallic zinc is a product with high added value. Zinc price is currently 2312 USD/tonne (to 29.09.2016) according the London Metal Exchange. However, for the treatment of zinc leach liquor a preparation of other commercial products, e.g. zinc sulfate or zinc oxide can be appropriate as well. These products could be economically more interesting as metallic zinc, mainly in terms of high energy consumption during zinc electrowinning. Research and development of alternative technology for the treatment of leach liquor from EAF dust leaching is subject for further study.

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