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Hydrometallurgical process for zinc recovery from electric arc furnace dust (EAFD) Part I: Characterization and leaching by diluted sulphuric acid

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ABSTRACT

The present paper is the first of a series of two articles dealing with the development of an integrated process for the recovery of zinc from electric arc furnace dust (EAFD), a hazardous industrial waste generated in the collection of particulate material during steelmaking process via electric arc furnace. Part I presents the EAFD characterization and its leaching process by diluted sulphuric acid, whereas Part II deals with the purification of the leach liquor and the recovery of zinc by solvent extraction/electrowinning. The characterization of the examined electric arc furnace dust was carried out by using granulometry analysis, chemical analysis, X-ray diffraction (XRD), thermogravimetric/differential thermal analysis (TG/DTA) and scanning electron microscopy (SEM). The leaching process was based on the Zn extraction with diluted sulphuric acid from EAFD under atmospheric conditions and without using any preliminary treatment. Statistical design and analysis of experiments were used, in order to determine the main effects and interactions of the leaching process factors, which were: acid normality, temperature and solid to liquid ratio. The zinc recovery efficiency on the basis of EAFD weight reached 80%. X-ray diffraction and scanning electron microscopy were used for the characterization of the leached residues.

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1. Introduction

Electric arc furnace dust (EAFD) is one of the most critical wastes encountered in steelmaking industries. During the meltdown of scrap, volatile components are fumed off and are collected with particulate matter in the off-gas cleaning system [1,2]. During the metal fusing process, the electric arc furnace (EAF) can reach temperatures of 1600 °C, or even higher, and many components of the charge, including iron, zinc, and lead, are volatilized and entered the vapour phase. A large quantity of dust is generated when the vapour is cooled and collected.

When galvanized scrap is used in the EAF, most of the zinc from the steel scrap ends up in the dust and fume due to its very low solubility in molten steel and slag, and, especially, because zinc vapour pressure is higher than iron vapour pressure at steelmaking temperature. Vapour zinc is carried out from the furnace with other gaseous or particulate compounds generated during steelmaking reactions, generating compounds like ZnO and ZnFe₂O₄. The largest metallic portion of the EAFD is zinc, which varies between 7 and 40%, depending on the ratio of galvanized scrap utilized [3–5].

The disposal of this waste has become a serious problem in recent years due to the fact that the greater availability of steel scrap, which represents a big part of the charge, has increased the production of steel from electric arc furnaces. The world generation of EAFD is estimated to be around 3.7 million tonnes per year. Plants from Europe generate around 500,000–900,000 tonnes of dusts per year [6]. In Greece all five steel factories are scrap-recycling facilities, where a total amount of 15,000 tonnes of EAFD is produced annually [7]. Such significant quantities are due to the fact that for every ton of recycled steel an amount of 15–20 kg of EAFD is produced.

The cost of EAFD disposal is not negligible. For example, 200 million dollars per year are necessary to dispose EAFD in the United States [8]. Furthermore, due to its chemical and physical properties, the electric arc furnace dust was categorized as hazardous waste according to the European Waste Catalogue (EWC 2002), when dangerous substances are present above threshold concentration [9]. Being treated as hazardous waste, it is partly disposed of permanently at appropriate, regulation-prescribed waste dumps, or it can be used as secondary raw material in the production of zinc, iron, lead, etc. However, the disposal of these industrial solid wastes can cause environmental risk due to the mobility of toxic elements.

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There are two possibilities to manage EAF dusts: hydrometallurgical and pyrometallurgical processes. Pyrometallurgical processes face the problems of high-energy consumption and generation of worthless residues. Most of these installations are based on rotary kilns, plasma, and flame reactor processes [10]. They require some reducing agents and relatively high temperatures to produce raw zinc oxide of low commercial value. On the other hand, hydrometallurgical processes are still a promise for the future and they could offer an interesting alternative for zinc recycling, if iron dissolution is controlled.

As it was mentioned before, zinc in EAFD is present as $ZnFe_2O_4$ (franklinite) and ZnO (zincite). Iron is present, except of franklinite, mostly as magnetite, Fe_3O_4 . The ZnO does not cause any problems to neither alkaline nor acid leaching. However, zinc ferrite is considerably refractory against leaching. Although alkaline leaching seems to be advantageous (due to the low iron leaching) it requires relatively concentrated leaching medium [11].

On the other hand acid leaching does not need so concentrated solution as the alkaline one, but iron is partially transferred into solution in this case. The remaining zinc, after acid leaching, is in the form of zinc ferrite [12,13]. However, choosing the sulphuric acid leaching route, the highly reactive species of zinc are dissolved, whereas most of the iron remains in the residue.

According to Pearson [14], the recovery of Zn from zinc dusts and fumes can reach 78% with sulphuric acid leaching. After the purification of the leach liquors, the metal is recovered by electrowinning. Steelmaking dusts have been also leached by Duvvesteyn and Jha [15] in order to recover zinc. In a first stage the dusts are leached with the liquor coming from the second stage, in which the solids from the first stage are treated in an autoclave with fresh sulphuric acid. Zn is recovered from the solutions and Fe is rejected as haematite residue. Cruells et al. [10] have examined the sulphuric acid leaching of the electric arc furnace (EAF) flue dusts in order to obtain the reaction rates of dissolution. The results showed that it is possible to beneficiate the dusts by leaching the non-magnetic fraction with sulphuric acid at low acid concentration and room temperature, giving high recoveries of Zn up to 80%. According to Havlik et al. [12] the optimal conditions for zinc removal from used EAF dust, at the minimal iron extraction are: temperature 70–90 °C and 0.5 M H₂SO₄. They also found that the decrease of liquid/solid ratio causes a decrease on the relative amount of leached zinc. The amount of extracted iron depends on the temperature used. Langova et al. [16] examined the sulphuric acid leaching of EAFD and studied the influence of acid concentration, temperature, time, and liquid/solid ratio. Zinc extraction reaches almost 100% and iron extraction exceeds 90% in 3 M H₂SO₄ at 80 °C and liquid/solid ratio of 5 after 6 h. Furthermore, they found that a good selectivity with regard to zinc was achieved in 0.1-0.3 M H_2SO_4 at 80 °C.

The aim of the present research paper is to present results on the characterization and the leaching of EAFD by diluted sulphuric acid. Factorial design of experiments and statistical analysis of the obtained data were used to determine the main effects and interactions of the chosen factors and select the optimum conditions. The factors studied were: sulphuric acid concentration, temperature and solid to liquid ratio. The initial EAFD and the resulting leach residues were investigated by X-ray diffraction (XRD), TG-DTA and scanning electron microscopy (SEM).

2. Experimental

The electric arc furnace dust was used as received from the dust collecting system of a carbon steelmaking operation in Greece. The leaching was carried out under atmospheric pressure. All batch experiments were conducted in a 500-ml five-necked, round bottomed split reactor, which was fitted with a glass stirrer, a vapour

 Table 1

 Chemical composition of EAFD used.

wt.%
1.145
0.519
24.780
18.600
3.949
1.804
2.440
3.214
0.194
6.016
25.290
2.452
0.240
0.454
3.622
6.450

condenser and a thermometer. In all the experiments, a constant stirring speed (700 rpm) was used to ensure suspension of the particles. Heating was provided by an electrical mantle and the temperature of the liquid was controlled by a Fisons temperature controller. A typical run was carried out as follows: specified amounts of preheated sulphuric acid of known concentration and EAF dust were loaded into the glass reactor. The stirring speed was maintained constant by means of a digital controlled stirrer. At the end of the runs, the content of the reactor was filtered under vacuum. The resulting leached residues were washed with water by re-pulping, dried at 110 °C and weighed.

The initial EAF dust, as well as the leached residues were analyzed chemically by atomic absorption spectrophotometer (PerkinElmer 4100) and inductively coupled plasma mass spectrometry (ICP-MS, X Series II, Thermo Scientific). The analysis of Fe (Fe²⁺ and Fe³⁺) was performed with back-titration using potassium dichromate (K₂Cr₂O₇). Mineralogical analysis was carried out by X-ray diffraction, using a Bruker D8-Focus diffractometer with nickel-filtered CuK α radiation (λ = 1.5405 Å), 40 kV and 40 mA. The particle size of the initial EAFD was determined by a Malvern Mastersizer-S laser scattering particle size distribution analyzer. An amount of 0.5 g of sample powder was put in 100 ml of ethanol and underwent dispersion treatment by an ultrasonic dispersion unit for 60s. TG/DTA analysis was conducted with a Setaram-Labsys thermal analyzer. Type S-thermocouple is used for temperature measurements in this instrument. The sample was placed in a ceramic crucible and heated from room temperature to 1200 °C at a heating rate of 5 °C/min using air as a medium under static condition. TG/DTA were done simultaneously. Finally, the morphology of the initial dust and leached residues were also examined by scanning electron microscopy using a Jeol 6380LV scanning electron microscope. Experimental conditions involved 20 kV-accelerating voltage. Chemical composition of the samples particles was carried out by an Oxford INCA energy dispersive spectrometer (EDS) connected to the SEM.

3. Results and discussions

3.1. Chemical and mineralogical characteristics of EAFD used

The chemical composition of electric arc furnace dust depends mainly on the quality of steel scrap processed and the type of steel being produced. Table 1 presents the chemical composition of the major oxides of the EAFD used. Beside iron and zinc, the dust is characterized by the relatively high content of calcium oxide, whose presence should be attributed to the lime added to the steelmaking furnace. The concentrations of heavy metals are given in Table 2.

Table 2Heavy metals content of EAFD.

0,01

0.1



1000

-10 -12

0

200

400

Particle Diameter (μm) Fig. 1. Particle size distributions of the EAFD used.

10

100

The particle size distribution of the examined electric arc furnace dust is given in Fig. 1. It presents a heterogeneous distribution of particle size and contains two major size fractions: a very finegrained portion $(0.1-1 \,\mu\text{m})$ and a coarser part $(1-100 \,\mu\text{m})$. It was found that 50% of it was below 19.3 μ m, whereas the majority (90%) of the particles were below 100 μ m.

The X-ray diffraction pattern of the as-received EAF dust is shown in Fig. 2. Three major phases were identified: zincite, franklinite and magnetite. Zinc in the examined electric arc furnace dust is in the forms of zinc oxide (zincite–ZnO) and zinc ferrite (franklinite–ZnFe₂O₄), whereas iron is mainly in the form of franklinite and magnetite (Fe₃O₄). Furthermore, wustite, CaFe₂O₅, periclase, pyrolousite, lead oxide, quartz, (Fe,Cr)₂O₃ and Ca(OH)₂ are also present as minor constituents.

The results of the differential thermal analysis (DTA) and thermal analysis (TG) of the initial EAFD sample are given in Fig. 3. The TG diagram shows three steps for the weight loss. The first one occurred in the range of 30-550 °C (weight loss was about

0.6% of the total weight), corresponding to the evaporation of physically adsorbed water ($30-140 \,^{\circ}$ C) and to the loss of chemically adsorbed water ($140-550 \,^{\circ}$ C). The second one, in the range of 550–800 $\,^{\circ}$ C (weight loss: 3.05%), was attributed mainly to the release of CO₂ during decomposition of carbonates. Above this temperature, weight losses (about 7%) are also observed possibly due to the volatilization of Pb and Zn. The endothermic peak at 380 $\,^{\circ}$ C, on the DTA curve, is associated with the dehydration and the loss of chemically adsorbed water. Finally, the second small endothermic peak at 510 $\,^{\circ}$ C was attributed to the decomposition of Ca(OH)₂.

600

Temperature (°C)

Fig. 3. TG/DTA curves of the initial EAFD sample.

800

1000

1200

Scanning electron microscopy, with energy dispersive spectrometer, was performed to gain further knowledge of the mineralogical species contained in the EAF dusts and their morphology. The sample was examined in secondary electron (SE) and backscattered electron (BSE) modes. The EAFD particles appeared as spherical fine-grained particles, or as elongated grains. An agglomerated morphology was predominant with fine particles



Fig. 2. Mineralogical phases of EAFD used.



Fig. 4. Spheres of franklinite and finer particles of zincite.

Table 3

EAFD leaching by diluted sulphuric acid – minimum and maximum levels of variables.

Factor Variable	s Low level (–)	High level (·	+) Units measured
A Acid no B Temper	mality 2 ature 40	3 60 20	N °C ∝



Fig. 5. Detection of magnetite spheres and large hexagonal plate-like crystals of $Ca(OH)_2$.

Table 4

% Recovery of metals in the leach liquor after leaching by diluted sulphuric acid.

Treatment code	Variables studied		Zinc (%)	Iron (%)	
	Α	В	С		
1	_	_	-	72.34 ± 0.98	32.54 ± 1.04
a	+	_	_	77.20 ± 0.98	36.82 ± 1.04
b	_	+	_	76.28 ± 0.98	34.60 ± 1.04
ab	+	+	_	80.60 ± 0.98	44.55 ± 1.04
с	_	_	+	63.84 ± 0.98	24.22 ± 1.04
ac	+	_	+	72.86 ± 0.98	28.80 ± 1.04
bc	_	+	+	66.44 ± 0.98	30.55 ± 1.04
abc	+	+	+	76.50 ± 0.98	39.65 ± 1.04

Table 5

Yates' analysis and analysis of variance-response: % Zn leaching by diluted sulphuric acid.

Treatment code	Response Zn leaching (%)	Yates' analysis						Analysis of variance		
		1	2	3	Divisor	Effects	Indentification	$t = eff/S_e$	t _{0.01}	Significance at $\alpha = 0.01$
(1)	72.34 ± 0.98	149.54	306.42	586.06	8	73.26 ± 0.35	Average	$\Phi = 8$		
a	77.20 ± 0.98	156.88	279.64	28.26	4	7.07 ± 0.70	A	10.16	3.36	S
b	76.28 ± 0.98	136.70	9.18	13.58	4	3.40 ± 0.70	В	4.88	3.36	S
ab	80.60 ± 0.98	142.94	19.08	0.50	4	0.13 ± 0.70	AB	0.18	3.36	NS
с	63.84 ± 0.98	4.86	7.34	-26.78	4	-6.70 ± 0.70	С	-9.63	3.36	S
ac	72.86 ± 0.98	4.32	6.24	9.90	4	2.48 ± 0.70	AC	3.56	3.36	S
bc	66.44 ± 0.98	9.02	-0.54	-1.10	4	-0.27 ± 0.70	BC	-0.40	3.36	NS
abc	76.50 ± 0.98	10.06	1.04	1.58	4	0.40 ± 0.70	ABC	0.57	3.36	NS



Fig. 6. X-ray diffraction of the EAFD leached residue, after drying at 110 °C.

forming aggregates or covering larger particles. This shape is in agreement with the main generation mechanism, i.e., ejection of the slag and metal particles by bubble-burst. Spherical particles with wrinkled surface and elongated non-defined forms were also observed. EDS studies revealed the presence of Zn and Fe, whereas smaller amounts of Ca, Pb, Mg and Cr were also found. Zinc is mainly associated with the finer dust particles, probably as zincite. The presence of franklinite was confirmed and observed mainly as spheres. As it is shown in Figs. 4 and 5, the observed aggregates of fine particles comprise essentially zinc and oxygen (zincite) and are spherical in shape. Iron and zinc are present, in a higher amount, in the larger particles. Probably it is the region where the franklinite phase (ZnFe₂O₄) is present. Iron and oxygen (magnetite) corresponded to smaller spherical granules, homogeneously distributed in the EAFD. Calcium appeared in high amount when iron is not present. A high density of hexagonal plates of Ca(OH)₂ was observed. The nature of the hexagonal crystals was determined by EDS. Their presence should be attributed to the fluxes addition in the steelmaking process.

3.2. Statistical study of the EAFD leaching process

Factorial design and analysis of experiments were used in order to determine the main effects and interactions of the leaching factors [17]. The factors studied and their levels, for EAFD leaching by diluted sulphuric acid, are shown in Table 3. Stirring speed and reaction time were kept fixed at 700 rpm and 1.5 h, respectively.

The main response under investigation was the percentage of zinc recovery in the leach liquor. The leaching percentages of iron are also given in Table 4. As can be seen in Table 4, zinc leaching varied from 63.84% to 80.60%. Under the present experimental conditions iron dissolution varied from 24.22% to 44.55%. To study the



Fig. 7. Calcium sulphate needle-like crystals and idiomorphic anglesite crystals.

main effects and interactions of the factors on the zinc leaching, a Yates' analysis and an analysis of variance were carried out on the percentage of Zn recovery from EAFD by diluted sulphuric acid leaching. The results are shown in Table 5.

The main effects A (acid normality) and B (temperature) and the two-factor interaction AC were found to be statistically significant at α = 0.01 and positives. On the other hand, the main effect of solid to liquid ratio (C) was also found to be statistically significant at



Fig. 8. Spheres of franklinite and magnetite.

 α = 0.01, but negative. In the range of variables studied, the acid normality (A) had the largest effects. The existence of statistically significant interactions indicated that a model, based on the main effects only, would not be suitable and that the response surface was curved in the particular region of the present design. Based on the above, the suggested "best" fitting model for zinc recovery, in the ranges of variables studied and under the present conditions of experimentation, is the following:

$$Y = 73.26 + 3.53X_1 + 1.70X_2 - 3.35X_3 + 1.24X_1X_3$$
(1)

where Y is the predicted value of zinc recovery from EAFD and X_1 , X_2 and X_3 are coded variables, related to the natural variables by the following equations:

- $X_1 = (acid normality 2.5)/0.5$
- $X_2 = (\text{temperature} 60)/10$
- $X_3 = (\text{solid to liquid ratio} 15)/5$

Based on the statistical analysis of the results, the optimum conditions determined for zinc leaching, from EAFD by diluted sulphuric acid, were: acid normality = 3 N, temperature = $60 \circ \text{C}$ and solid to liquid ratio = 10%.

3.3. Characterization of the leached residues

The X-ray diffraction data of the residue (after drying at 110 °C), after leaching by diluted sulphuric acid, at the optimum leaching conditions is presented in Fig. 6. Its main mineralogical phases were bassanite (CaSO₄ 0.5H₂O) anhydrite (CaSO₄) and anglesite (PbSO₄). The low solubility of calcium and lead sulphates led to the production of the above secondary precipitates. The main peaks were sharper and with higher intensity, a fact that was attributed to the grain size and the degree of crystallinity of the two main products. Although zincite (ZnO) was totally leached within 1.5 h of leaching, with 3 N H₂SO₄ at 60 °C, the dissolution of franklinite (ZnFe₂O₄) and magnetite (Fe₃O₄) were lower. Periclase (MgO) and quartz (SiO₂) were not leached under the present experimentation conditions.

The results of the mineralogical analysis by X-ray diffraction were also confirmed by electron microscopic observations. As shown in Fig. 7, bassanite and anhydrite consisted of needle-like crystals of $5-10 \,\mu$ m in length and $1-2 \,\mu$ m in diameter. As the leaching reaction proceeded, zincite was totally dissolved, while the amount of the iron phases leaching (magnetite and franklinite) was lower (Fig. 8). The elements that hold or increased their composition in the leach residue were sulphur, calcium and lead because of the formation of CaSO₄, CaSO₄0.5H₂O and PbSO₄ phases.

4. Conclusions

The results obtained in the present work showed that zinc can be leached by diluted sulphuric acid from the solid waste called electric arc furnace dust, which is generated in the collection of particulate material during steelmaking process via electric arc furnace. Its characterization showed that it contained mainly zinc oxide (zincite–ZnO) and zinc ferrite (franklinite–ZnFe₂O₄).

A statistically designed experiment was performed to investigate the recovery of zinc from EAFD with diluted sulphuric acid leaching. Zinc extraction reached 80% at the following the optimum conditions: acid normality = 3 N, temperature = $60 \,^{\circ}$ C and solid to liquid ratio = 10%. Under these experimental conditions, iron leaching reached 45%.

The leaching results, combined with the SEM/EDS and X-ray diffraction analyses, indicated that zinc present in an EAF dust as zincite is readily dissolved, while the zinc ferrites dissolution is lower. The main mineralogical phases of the leached residue were bassanite (CaSO₄ 0.5H₂O), anhydrite (CaSO₄) and anglesite (PbSO₄).

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