Hydrometallurgical Extraction of Zinc from EAF Dust in Nitric Acid Solution

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Abstract

Zinc oxide (ZnO) and zinc ferrite (ZnFe₂O₄) are the main components in the electric arc furnace dust (EAFD). Due to the presence of significant amount of zinc, iron and lead, EAFD is classified as hazardous wastes. Turkey is the eighth largest crude steel producer in the world with an annual 37.3 million tons of steel production. Approximately 80% of steel production in Turkey is secondary steel production. In the secondary steel production, 14-20 kg EAFD containing 25-45% Zn is occurred depending on the scrap composition used per 1 ton steel production. This work aims to investigate the extraction of zinc from EAFD in nitric acid (HNO₃) solutions. The effects of stirring speed, temperature and HNO₃ concentration on the zinc extraction were determined. The amount of zinc passed to the solution was determined by using ICP-OES. The extraction of zinc increased with increasing temperature and HNO₃ concentration. The maximum extraction, 98%, was achieved by using 4 M HNO₃ and 80 °C.

1. Introduction

One of the most important issues with which steel producers are faced is the question of environmental protection. It refers e.g. to the necessity to utilize dusts resulting from the process of steel production from scrap in electric furnaces, containing mainly of Zn, Fe, Pb and a considerable amount of harmful elements, such as Cd, As, Cr and F [1].

The world generation of EAFD is estimated to be around 3.7 million tons per year. Plants from Europe generate around 500,000-900,000 tons of dusts per year. All five steel factories are scrap-recycling facilities, where a total amount of 15,000 tons of EAFD is produced annually [2].

The composition of EAFD depends on the following aspects: on the scrap processed, on the type of steel to be produced, on operating conditions and on the degree recirculation of the dust to the process. The contents of the main elements in EAFD may vary between: 30% of Zn, 0.3–6% of Pb, 0.01–0.2% of Cd, 20–35% of Fe, 0.2–0.7% of Cr, 1–10% of Ca, etc. Zinc is present mostly in two basic compounds, namely as oxide ZnO and ferrite ZnFe₂O₄, and possibly as a complex ferrite, e.g. (ZnMnFe)₂O₄. ZnO in principle is an easily workable form for both the pyro and the hydrometallurgical method, but the ferrite form is considerably complex and difficult [1].

Turkey was Europe’s second and the world’s 8th biggest crude steel producer in 2016 with 33.2 tons of production. While 30% of the world’s steel production (1.7 billion tons) was made in Electric Arc Furnace (EAF) as secondary production, 80% of steel production was made in EFA in Turkey in 2016. 14-20 kg EAFD that contains 25-45% Zn is generated depending on the composition of scrap during the production of 1 ton steel [3].

Different processes (hydrometallurgical + electrometallurgical and pyrometallurgical) have been applied for the processing of EAFD. Because zinc oxide is reduced in gas phase, pyrometallurgical processes (ZTT Ferroline process, Waelz process, Inmetco direct reduction process and etc.) suffers from difficulties in the control of zinc loses and high energy consumptions. Thus industrial use of pyrometallurgical methods are limited. Zinc in ferrite phase should be separated to obtain high extraction rates in hydrometallurgical processes. These working conditions can be achieved only at high temperatures and using high acid concentrations which in turn result in the dissolution of iron together with zinc in leach liquor. On the other hand, when alkaline leach solutions are used, lead is passed to the leach liquor together with zinc. There are difficulties for the obtaining of final product in both cases [4-7].

2. Experimental Procedure

2.1. Materials and Methods

EAFD with particle size fraction of -150 μm obtained from Çolakoğlu Metalurji Company was used in leaching experiments. XRF (Panalytical Axios-Minerals) and XRD (Rigaku D/Max-2200) analyses were used for the determination of elemental and phase composition of the EAFD.
The dissolution experiments were carried out in water-heated, jacketed borosilicate glass reactor (HWS DN 100, Germany) having volume of 1 L. A thermostat with water circulation (Julabo MV4, Germany) was used to heat the reactor and to achieve isothermal conditions. The solution in the reactor was stirred by a mechanical stirrer (IKA RW 20 DZM, Germany). The solution temperature in the reactor was measured with a PT100 temperature sensor. A glass pipe including G-3 porous alumina disk at the end was used as a sampler.

Dissolution experiments were carried out using 5 g of sample, 0.5, 1, 2 and 4 M HNO₃, 40, 60 and 80 °C and 300 rpm stirring speed for the determination of the effect of HNO₃ concentration, temperature and stirring speed on the dissolution behavior of EAFD in HNO₃ solutions. When isothermal conditions obtained sample weighed in a ceramic crucible was added to the reactor. Samples were taken from the reactor at certain time intervals and analyzed in ICP-OES instrument. Fresh acid solution that was equal to the sample volume was added to the reactor after sampling to maintain constant reactor volume and acid concentration.

### 3. Results and Discussion

XRD diagram of EAFD is shown in Figure 1. Intense peaks of ZnFe₂O₄ (PDF 22-1012), ZnO (PDF 36-1451), Fe₂O₃ (PDF 39-1346), ZnO (PDF 21-1486) and weak peaks of PbO₂ (PDF 37-0517) and PbO (PDF 05-0561) are seen on Figure 1. XRF quantitative analysis results of EAFD was shown in Table 1.

#### 3.1. Dissolution Behavior of EAFD in Nitric Acid Solutions

Extraction % - time diagrams were plotted for Zn and Fe using ICP-OES analyses of leach solutions taken from the reactor at certain time intervals and XRF analysis of EAFD.

#### The Effect of Stirring Speed on the Dissolution of EAFD

Dissolution experiments were carried out at 80 °C using 4 M HNO₃ for the determination of the effect of stirring speed on the dissolution rate of EAFD in HNO₃ solutions. Zinc extraction (%) – time diagrams are shown in Figure 2.

![Figure 2. Zn extraction (%) - time diagrams for different stirring speeds (80 °C, 4 M HNO₃)](image)

As it is seen from Figure 2, stirring speed has no significant effect on the dissolution rate of Zn in EAFD. Therefore, it was determined that 300 rpm was enough to eliminate the resistance of the liquid film layer around the solid EAFD particles. Thus, 300 rpm stirring speed was used in all of the dissolution experiments.

#### The Effect of Temperature on the Dissolution of EAFD

Dissolution experiments were carried out at 40, 60 and 80 °C temperature and using 0.5, 1, 2 and 4 M HNO₃ solutions and 300 rpm stirring speed for the determination of the effect of temperature on the dissolution rate of EAFD in HNO₃ solutions. Diagrams of zinc extraction (%) – time and iron extraction (%) – time were shown in Figure 3 and 4, respectively. It is seen from Figure 4 that zinc extraction (%) increased with increasing temperature.

The highest zinc extraction, 97 %, was obtained from the experiment carried out at 80 °C using 4 M HNO₃ solutions and 300 rpm stirring speed. It is seen from the zinc extraction (%) – time diagrams that dissolution rate of EAFD is very high at the beginning of the dissolution experiments where 75-80% Zn extraction was obtained and then maximum extraction values are reached during 1 h of dissolution reaction.
It is seen from Figure 5 that dissolution of Fe in EAFD is more dependent on the temperature than that of Zn and Fe concentration is increased with increasing temperature. Very high Fe extraction, about 85%, was obtained from the experiments carried out using higher HNO₃ concentration. It is obvious that high Fe extraction together with Zn results in difficulties in the purification process of Zn in leach solutions. Thus appropriate leaching conditions where high Zn and low Fe extraction rates were obtained (0.5 M HNO₃ and 60/80 °C) should be chosen for the leaching of EAFD in HNO₃ solutions.

Figure 3. Zn extraction (%) vs t diagrams for different temperatures at constant HNO₃ concentrations; (a) 0.5, (b) 1, (c) 2 and (d) 4 M (stirring speed: 300 rpm)
**The Effect of HNO₃ Concentration on the Dissolution of EAFD**

Diagrams of zinc extraction (%) – time and iron extraction – time were shown for the determination of the effect of temperature on the dissolution rate of EAFD in HNO₃ solutions in Figure 3 and 4, respectively. Since same experimental data is used for the plotting of the diagrams to determine the effect of HNO₃ concentration on the dissolution rate of EAFD in HNO₃ solutions, two of Zn extraction (%) – time (Figure 5) and Fe extraction – time diagrams (Figure 6) were shown to avoid repetition. While dissolution of Zn is not effected by the increase of HNO₃ concentration significantly, dissolution rate of Fe increased rapidly (Figure 6).

**4. Conclusion**

97 % Zn extraction is obtained by the dissolution of EAFD in HNO₃ solutions at the experiments carried out at higher temperature (80 °C). Although temperature is effective on dissolution rate of both Zn and Fe, dissolution of Fe is more temperature dependent that that of Zn.

While HNO₃ concentration has slight effect on the dissolution rate of Zn, dissolution rate of Fe increased significantly with increasing HNO₃ concentration.

Low HNO₃ concentration (0.5 M) and high experimental temperatures (60-80 °C) should be chosen for the dissolution of EAFD in HNO₃ solutions to prevent high Fe extraction (%) which results in difficulties in subsequent processes and obtain Zn rich leach solutions.

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