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Deflocculation and Classification of Electric Arc Furnace Dust in Aqueous Solution

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Abstract: Electric arc furnace (EAF) flue dust is generated during EAF steelmaking from iron-containing scrap such as recycled automobile bodies. The relatively high level of Zn (20–30 wt%) in EAF dusts provides a potentially valuable resource, even though the zinc is present as oxide phases such as zincite (ZnO) and zinc ferrite (ZnFe_2O_4). In this work, a method to deflocculate and disperse ZnO and ZnFe_2O_4 particles in aqueous suspension of EAF dust is developed and the efficiency of Zn recovery by the classification method was evaluated. Major findings of this study indicate that citric acid, a tetradentate chelating agent, demonstrates the best liberation and dispersion efficiency for EAF dust. Elutriating at 0.01 cm/min fluid velocity we recover 10 wt% of EAF dust (mostly ZnO particles) at a grad of 70 wt% Zn from a well-liberated and dispersed suspension of EAF dust. About 30 wt% of Zn recovery may be achieved using only simple classification.

Keywords: Classification, dispersion, EAF dust, Zn recovery

INTRODUCTION

Electric arc furnace (EAF) flue dust is generated during EAF steelmaking from iron-containing scrap such as recycled automobile bodies. It is estimated that about 20 to 30 Kg of EAF dust are generated per ton of steel produced (1,2). The growth of EAF steelmaking indicates an increasing recycle of steel scrap and has obvious economic and resource conservation

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benefits. As a result of these developments there has been an increasing volume of EAF dust to be handled. Currently Taiwan produces 300000 tons of EAF dust per year (3). The EAF dust is considered as a hazardous waste in most countries because it fails the toxicity characteristic leaching procedure (TCLP) test for Pb, Cd, and Cr (4,5). However, the relatively high level of Zn in EAF dusts provides a potentially valuable resource, even though the zinc is present as oxide phases such as zincite (ZnO) and zinc ferrite (ZnFe_2O_4). Usually EAF dusts contain an average of 20–30% of Zn by weight with a proportion of 50% in the zinc ferrite form (6,7). At present most options developed to recover Zn and possibly other metals from EAF dusts were concerned with pyrometallurgical and/or hydrometallurgical routes (8–10). Zn in ZnO form can be recovered without too many difficulties for both pyrometallurgical and hydrometallurgical processes. But recovery of Zn in ZnFe_2O_4 is considerably complex and difficult due to the fact ferrite is very stable and the ferrite must be decomposed before the Zn in ferrite can be recovered (1). In other words operating conditions for Zn recovery from zincite and ferrite differ significantly. Thus it seems to be advantageous to separate zincite (ZnO) and zinc ferrite (ZnFe_2O_4) particles in EAF dust with a relatively simple process before a pyrometallurgical or hydrometallurgical Zn recovery process. It was found that the finest particles in EAF dust, whose sizes are lower than 2 or 3 μm , are frequently agglomerated to each other or around bigger particles (11). Sofilić et al. (12) shown that EAF dusts were composed of solid spherical agglomerates with Fe, Zn, Pb, and O as the principal element. After analyzing EAF dusts collected from 13 steelmaking plants in Taiwan, Li and Tsai (13) found that EAF dust exists as irregular agglomerates from 3 to 20 μm in size and are made up of much smaller round particles from 0.3 to 1 μm in size. The agglutinative substance was identified as being $\text{ZnCl}_2 \cdot 4\text{Zn}(\text{OH})_2 \cdot \text{H}_2\text{O}$ (13). Based on the above considerations, evidently a method is needed to effectively liberate ZnO and ZnFe_2O_4 particles from agglomerates and maintain a stable suspension amenable to the subsequent separation of ZnO and ZnFe_2O_4 particles for further treatments. In this work, a method to deflocculate and disperse ZnO and ZnFe_2O_4 particles in aqueous suspension of EAF dust was developed and the efficiency of ZnO and ZnFe_2O_4 particles separated by classification was evaluated.

EXPERIMENTAL

Materials

The EAF dust sample used in this study was obtained from dust containers located at the bottom of baghouses in a steelmaking plant at southern

Taiwan. The dust sample was pre-washed with distilled water to remove dissolvable salts. Five analytical grad deflocculants, citric acid ($\text{C}_6\text{H}_8\text{O}_7$), sodium citrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$), oxalic acid ($\text{C}_2\text{H}_2\text{O}_4$), hydrochloric acid (HCl), and ethylenediaminetetraacetate ($\text{Na}_4\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_6$; EDTA- Na_4) obtained from Merck were used in this study.

Dust Characterization

The mixture of 0.1 g of EAF dust and 15 mL of acid (20% 29 N HF and 80% 12 N HCl) was placed in a stainless steel autoclave lined with Teflon with a capacity of 50 mL. The autoclave was heated at 250J for 24 h to fully digest EAF dust. After cooling, the digest was transferred to a 100-ml volumetric flask and brought to volume. The Fe and Zn contents of EAF dusts were determined by an atomic absorption (AA) spectrometric analyzer. The Zn contents in ZnO and ZnFe_2O_4 form were distinguished by a simple HCl leaching test. 2.0 g of EAF dust was added into a glass beaker containing 50 ml of HCl solution (0.1–0.6 N), and then leached by magnetic stirring at 600 rpm for 0.5 h. The filtered liquid was analyzed for Zn and Fe by an AA spectrometric analyzer and the leaching curves for Zn and Fe were compared to identify the onset of ZnFe_2O_4 dissolution. The crystalline phase was determined by means of an X-ray diffractometer (XRD, Siemens D5000) using $\text{CuK}_{\alpha 1}$ radiation with a scanning speed of $2.4^\circ \text{min}^{-1}$. The morphology and surface composition of EAF dust were recorded using a Hitachi S-4100 SEM with an EDAX detector. The hydrodynamic particle size distribution of EAF dust was determined by the Andreasen pipette method. In this method the concentration of solid changes occurring within a settling suspension of EAF dust are followed by drawing off definite volumes by means of a pipette and from these the hydrodynamic size distribution of EAF dust may be found.

Deflocculation Test

The deflocculation efficiency of EAF dust after the addition of deflocculant was determined as follows. 0.5 g of EAF dusts were mixed with 50 ml aqueous solutions containing specific amounts of deflocculant. The suspensions were mixed using a magnetic stirrer at 600 rpm for 12 h and left to settle for 25 min. Top 20-ml supernatant was removed and filtrated through a $0.22 \mu\text{m}$ filter under vacuum. The filtered liquid was analyzed for Zn and Fe by an AA spectrometric analyzer. The remaining solid on filter was dried and the mass was recorded. The relative deflocculation

efficiency was determined as follows:

$$\% \text{ Deflocculation} = \frac{(\text{Mass of dusts in 20-ml supernatant})}{\text{Total mass}} \times 100.$$

Classification Test

The effect of deflocculation on subsequent classification of ZnO and ZnFe₂O₄ particles in aqueous suspensions of EAF dust was evaluated by a sedimentation test adapted from the Andreasen pipette method. In this method the mass and Zn, Fe contents of solids collected by drawing off definite volumes from a settling suspension of EAF dust at specified times were monitored. Each sample of solid thus represents a portion of EAF dust that can be elutriated by an upward flowing water with a rising velocity corresponding to a certain time of drawing. The recovery and grad of the elutriated EAF dust corresponding to a certain rising velocity of water were then evaluated.

RESULTS AND DISCUSSION

Characterization of EAF Dust

The pre-washed EAF dust used in this study contained 32.9% Fe, 29.4% Zn, and 3.5% Pb as determined by AA analyzer. The X-ray diffraction

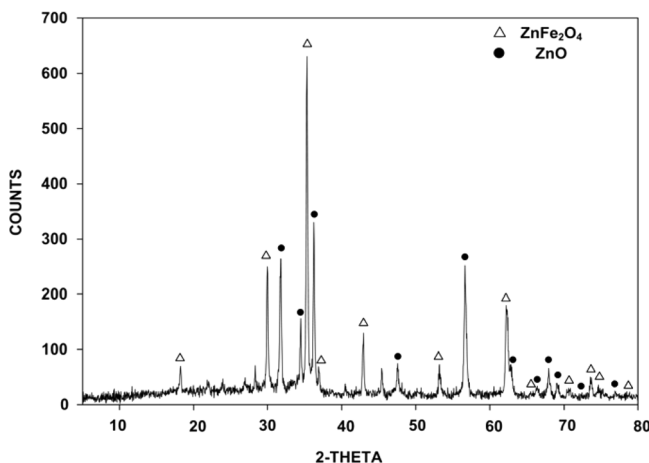
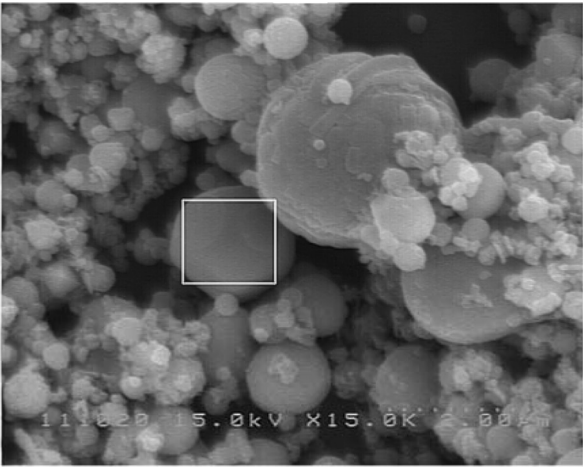
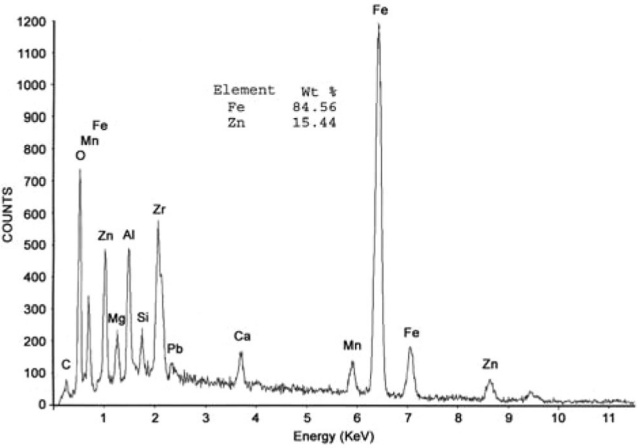


Figure 1. The X-ray diffraction pattern of the pre-washed EAF dust.

pattern of the pre-washed EAF dust is shown in Fig. 1. It shows that zincite (ZnO) and zinc ferrite (ZnFe_2O_4) are the two dominant phases. Selected SEM images of EAF dust are given in Figs. 2a and 3a. The dust comprises small and large particles of various shapes but with dominant large spherical particles. Many small particles appear to be stuck or fused to large spherical particles. The results of X-ray energy dispersive spectro-

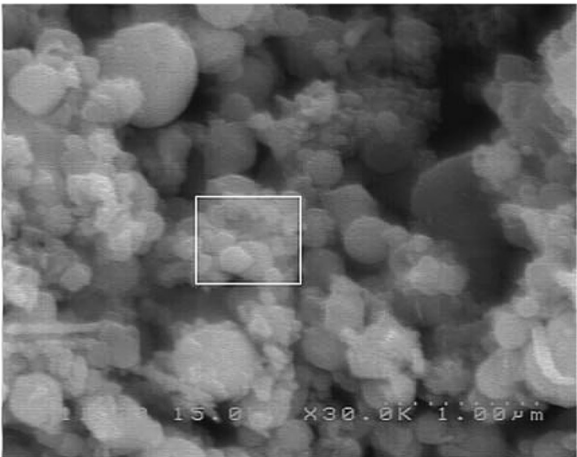


(a)

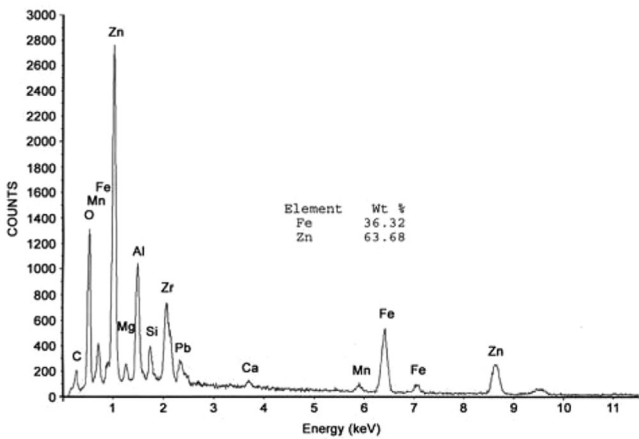


(b)

Figure 2. (a) SEM images of EAF dust; (b) EDS results on selected particles (enclosed by a square in SEM images).



(a)



(b)

Figure 3. (a) SEM images of EAF dust; (b) EDS results on selected particles (enclosed by a square in SEM images).

metry (EDS) on selected particles (enclosed by a square in SEM images) are shown in Figs. 2b and 3b. Figure 2b shows that the major element of large spherical particles is Fe (84.56%). In contrast, Fig. 3b indicates that the major element of small irregular particles is Zn (63.68%). Combining information obtained from Figs. 1, 2, and 3, it is reasonable to identify the EAF dust as agglomerates of small irregular ZnO particles stuck or fused to large ZnFe₂O₄ spherical particles. The fine nature of ZnO grains relative to the coarser ZnFe₂O₄ particles is consistent with the test results

discussed latter. Figure 4 demonstrates the hydrodynamic particle size distribution of raw pre-washed EAF dust (labeled as dust without citric acid in Fig. 4) determined by the Andreasen pipette method. The fact that most of the particles in raw pre-washed EAF dust are bigger than $10\text{ }\mu\text{m}$ indicates the raw pre-washed EAF dust is highly agglomerated and an effective deflocculation method is needed to liberate ZnO and ZnFe_2O_4 particles in aqueous suspension. The results of the HCl leaching tests (Fig. 5) show that the leaching curves for both Zn and Fe demonstrate a break-point at a HCl concentration of 0.3 N which indicates the onset of ZnFe_2O_4 dissolution. Thus the amount of Zn leached at 0.3 N HCl solution is a reasonable estimation of the amount of Zn in ZnO form. It is estimated that about 50% of Zn in this EAF dust is in ZnO form. This means that a complete separation of ZnO and ZnFe_2O_4 particles will result in a 50% recovery of Zn as ZnO from EAF dust.

Deflocculation Test

The deflocculation efficiency and concentration of Zn ions dissolved for EAF dust suspensions as a function of deflocculant dosage for various deflocculants are shown in Fig. 6. The dosage of deflocculant applied is

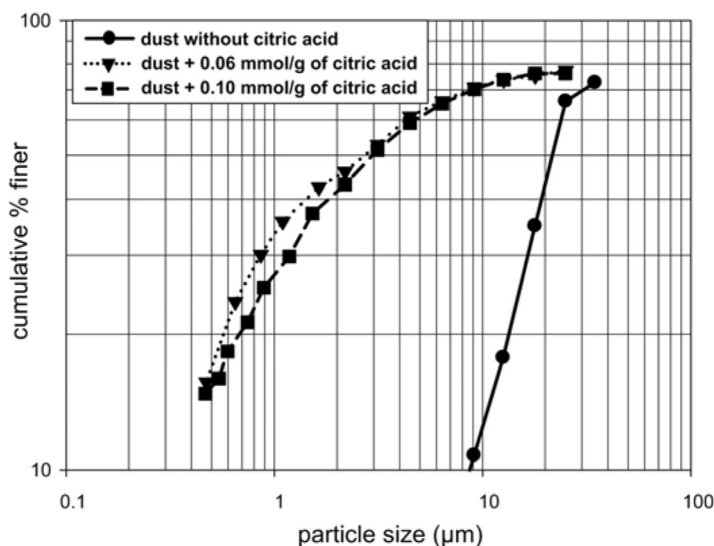


Figure 4. The hydrodynamic particle size distribution of EAF dust determined by the Andreasen pipette method.

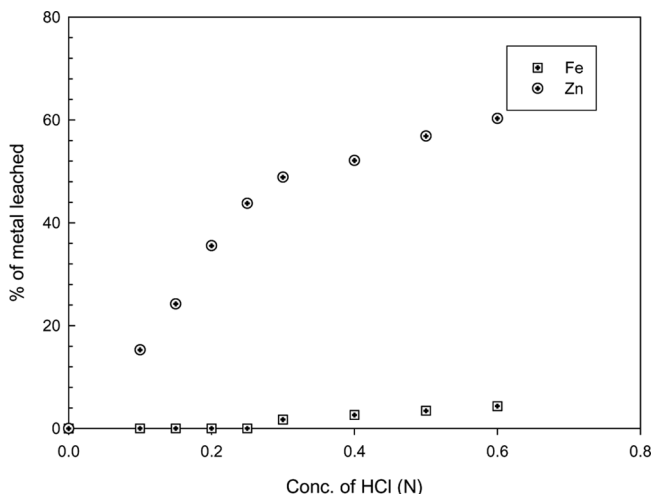


Figure 5. Percent of Zn and Fe ions leached as a function of HCl concentration.

expressed in mmol of deflocculant per gram of dust (mmol/g of dust). In here we are seeking for a deflocculant to effectively dissolve the agglutinative substance binding ZnO and ZnFe_2O_4 particles together and consequently disperse EAF dust suspensions. It is clear that citric acid and Na-citrate, both a tetradentate chelating agent, demonstrate the best deflocculation efficiency among deflocculants tested (Figs. 6a and 6b). A dosage of 0.06 mmol/g of citric acid and 0.16 mmol/g of Na-citrate resulted in a deflocculation efficiency of 24% and 33% respectively for EAF dust suspension. In addition the amount of Zn ions released was 5 ppm and 1.1 ppm respectively at those deflocculant dosages. It is noted that in the absence of deflocculant, EAF dust suspension is fully agglomerated (0% deflocculation efficiency). Test results also indicated that Fe ions released for all deflocculation tests were negligible in contrast to Zn ions. This implies that Zn containing species such as $\text{ZnCl}_2 \cdot 4\text{Zn}(\text{OH})_2 \cdot \text{H}_2\text{O}$ are responsible for the agglomeration of EAF dust. It is seen from Fig. 6a that the deflocculation efficiency increases dramatically with the addition of citric acid, with constant deflocculation efficiency being maintained at citric acid dosages of 0.06 mmol/g or higher even though the amount of Zn ion released is continuous to increase. Thus, it is reasonable to speculate that dust particles will be liberated once the agglutinative substance (such as $\text{ZnCl}_2 \cdot 4\text{Zn}(\text{OH})_2 \cdot \text{H}_2\text{O}$) binding ZnO and ZnFe_2O_4 particles together have been dissolved at 0.06 mmol/g of citric acid. Further increase of citric acid dosage resulted only in more Zn ions released probably due to the decrease of solution

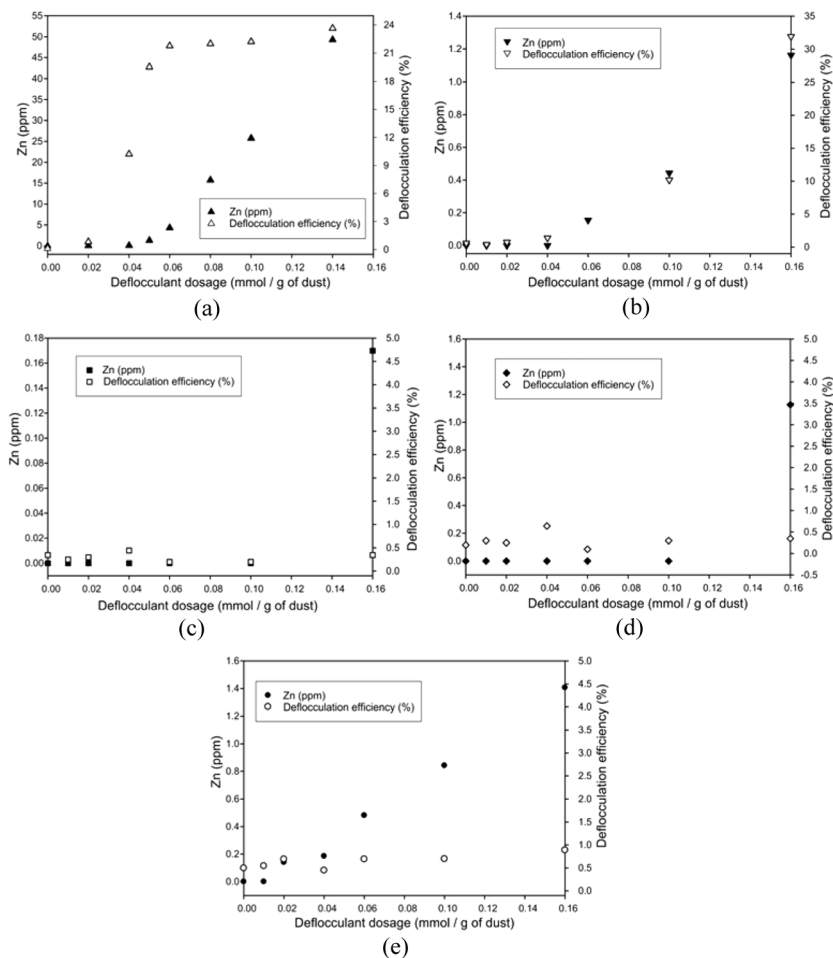


Figure 6. Deflocculation efficiency and concentration of Zn leached as a function of deflocculant dosage for (a) citric acid; (b) Na-citrate; (c) oxalic acid; (d) HCl; (e) EDTA.

pH (Fig. 7). For the deflocculation of dust suspensions by Na-citrate (Fig. 6b), the deflocculation efficiency was found to increase with increasing Na-citrate dosage. However, the amount of Zn ion released is quite low (0–1.2 ppm) compared to deflocculation using citric acid. Because of the relatively high pH conditions resulting from using Na-citrate (Fig. 7), we speculate that Na-citrate, a weak base, releases Zn ions mainly by forming Zn-citrate complexes in solution. The effect of citric

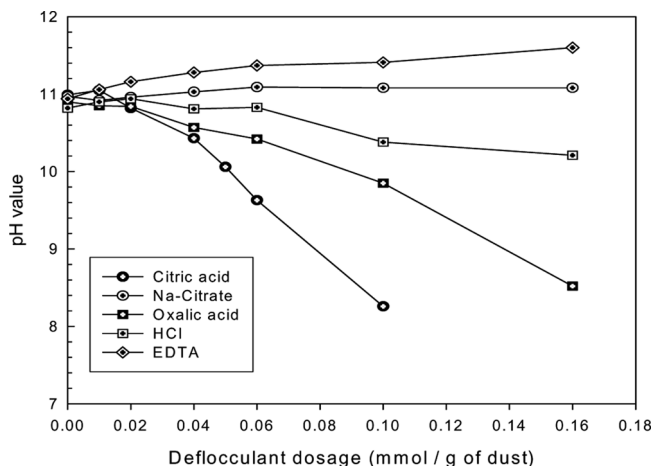


Figure 7. The change of pH values as a function of deflocculant dosage.

acid on the liberation and dispersion of EAF dust was further supported by the hydrodynamic particle size distribution of EAF dust after citric acid addition (Fig. 4). The average particle size (d_{50}) of EAF dust decreased from 20 μm to 3 μm with a 0.06 mmol/g of citric acid addition. However, double the citric acid dosage did not further decrease the particle size of dust and this result was in line with the result from deflocculation test (Fig. 6a). It is also interesting to note that for deflocculants such as oxalic acid (Fig. 6c) and HCl (Fig. 6d), a dosage of 0.16 mmol/g also resulted in a considerable amount of Zn ions releasing (1.0 ppm) probably due to pH lowering (Fig. 7) but without any deflocculating effect. Evidently a good deflocculant must be a good Zn-complexing agent which also serves to stabilize the dispersed ZnO and ZnFe₂O₄ particles. Finally, a traditionally excellent complexing agent, EDTA, did not show any deflocculation effect even some Zn ions were released (Fig. 6e). Based on the above test results, citric acid owing to its low dosage consumption and high deflocculation efficiency was chosen as a deflocculant to conduct classification tests.

Classification Test

The effect of deflocculation on subsequent classification of ZnO and ZnFe₂O₄ particles in aqueous suspensions of EAF dust was evaluated by a sedimentation test adapted from the Andreasen pipette method using citric acid at a dosage of 0.16 mmol/g. Table 1 shows the mass

Table 1. The mass and Zn, Fe content of solids collected from a settling suspension of EAF dusts at specified times

Sampling time (min)	Velocity (cm/min)	Dust collected (g/10 ml)	Fe content (wt%)	Zn content (wt%)
2	9.30228	0.0912	26.1%	31.2%
25	0.77865	0.0816	25.1%	33.1%
90	0.21265	0.0549	18.8%	37.0%
240	0.07808	0.0300	18.2%	41.7%
502	0.03651	0.0227	14.7%	42.0%
1088	0.01646	0.0167	13.8%	54.2%
1430	0.01223	0.0139	13.8%	65.9%
2700	0.00632	0.0083	5.8%	72.8%
3990	0.00418	0.0051	4.1%	74.0%

and Zn, Fe content of solids collected from a settling suspension of EAF dusts at specified times each corresponding to a certain settling velocity. Thus each sample of solid represents a portion of EAF dust that will be elutriated by an upward flowing fluid with a rising velocity equal to the settling velocity. The parameters used to evaluate the separation are percent Zn recovery and Zn grad of the collected dust. Figure 8 plots dust

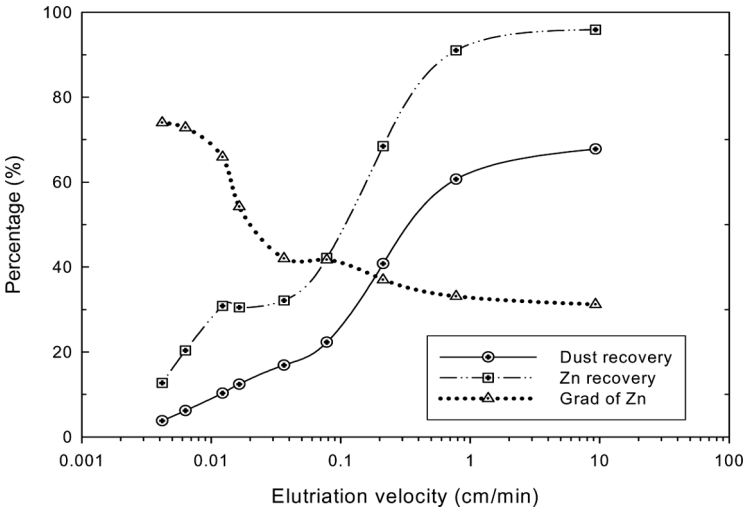


Figure 8. Dust recovery, Zn recovery and Zn-grad of overflow products as a function of elutriation velocity during classification.

recovery, Zn recovery, and Zn grad of overflow products as a function of elutriation velocity during classification. It is noted that the dust recovery curve was relatively parallel to the Zn recovery curve presumably due to that only 49.3% of Zn in ZnO form was recoverable by classification. This means that a complete separation of ZnO and ZnFe₂O₄ particles will result in a 49.3% recovery of Zn in dust. However elutriating at 0.01 (cm/min) fluid velocity we recover about 10% dust particles with a grad of 70% Zn which corresponding to a 30% Zn recovery from well liberated and dispersed suspension of EAF dust. This high-grad zincite concentrate may be reused directly and the 90% underflow product (mostly ZnFe₂O₄) may require further treatment to separate Zn and Fe.

CONCLUSIONS

Major findings of this study are summarized as follows:

1. The pre-washed EAF dust used in this study contains 32.9% Fe, 29.4% Zn with about 50% of Zn in the ZnFe₂O₄ form.
2. The pre-washed EAF dust contains ZnO and ZnFe₂O₄ as the two dominant phases. Dust is identified as agglomerates of small irregular ZnO particles stuck or fused to large ZnFe₂O₄ spherical particles with an average size of 20 μm.
3. Citric acid, a tetradentate chelating agent, demonstrates the best deflocculation efficiency. The dust suspension is dispersed once the agglutinative substance (such as ZnCl₂ · 4Zn(OH)₂ · H₂O) binding ZnO and ZnFe₂O₄ particles together having been dissolved at a 0.06 mmol/g dosage of citric acid.
4. Elutriating at 0.01 (cm/min) fluid velocity we recover about 10% dust particles with a grad of 70% Zn which corresponds to a 30% Zn recovery from a well-liberated and dispersed suspension of EAF dust.

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