



# Selective recovery and separation of copper and iron from fine materials of electronic waste processing

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## ABSTRACT

As electronic waste (e-waste) is increasing, its by-products such as flue dust and wastewater, which are generated during its treatment process, are also increasing. In this study, the fine material generated in the processing of e-waste by both mechanical and thermal methods was investigated for the effective recovery and separation of valuable metals, including Cu and Fe, using both physical and chemical methods. The Fe in this sample was present mostly as magnetite, as determined by XRD, and was removed by magnetic separation. Three different acid solutions ( $\text{HNO}_3$ ,  $\text{HCl}$ , and  $\text{H}_2\text{SO}_4$ ) were used for selective Cu recovery. All experiments were conducted with varying length of time (1–10 h) and temperature ranges (20–60 °C). The kinetics of Cu dissolution in  $\text{HNO}_3$  was studied, based on the shrinking core model, and the activation energy was about 5.0 kcal/mol. The dissolution of Cu in  $\text{HNO}_3$  was faster than that of other metals such as Fe and Al. In the case of  $\text{H}_2\text{SO}_4$ , dissolution of Cu was not observed, while Fe was completely dissolved after about 4 h at 60 °C. Based on the results of various leaching and the kinetic study, a two-step process was performed for selective metal recovery.  $\text{H}_2\text{SO}_4$  was used to dissolve Fe as the first leaching process and  $\text{HNO}_3$  was used for Cu dissolution as the second leaching process. Through this two-step process, Fe and Cu leaching efficiencies were obtained at approximately 90% and 98%, respectively.

## 1. Introduction

As technology advances in both the electrical and electronics industries, a large amount of electronic waste (e-waste) is generated (Robinson, 2009; Widmer et al., 2005). E-waste contains components that are harmful to humans and affects environmental pollution (Kiddee et al., 2013). Therefore, some countries have established policies and legislation specific to e-waste (Nnoroma and Osibanjo, 2008). There are many options for the treatment of e-waste, including reuse, remanufacturing, recycling, incineration, and landfilling. Among them, recycling is most important in terms of environmental protection and valuable metal recovery—as long as it is both technically and economically viable.

Recently, some researchers have reported on recycling studies for e-waste based on hydrometallurgy (Le et al., 2011; Tuncuk et al., 2012; Xiao et al., 2013; Birloaga et al., 2014; Jadhav and Hocheng, 2015; Kumari et al., 2016). Compared to pyrometallurgical processes, hydrometallurgical processes have relatively low capital costs, minimal atmospheric pollution problems, and high selectivity in metal recoveries (Tuncuk et al., 2012; Jadhav and Hocheng, 2015). They seem to be suitable for small-scale applications and are generally

energetically favorable (Gupta and Mukherjee, 1990; Tuncuk et al., 2012). Hydrometallurgy can be separated into three general processes for metal recovery; precipitation, solvent extraction (SX) and ion exchange (IX), all of which follow leaching by acid or caustic solutions (Cui and Zhang, 2008).

E-waste is generated from various types of electronic equipment, and the elements and compositions of e-waste depend on the materials that make up the electronic equipment. E-waste can contain up to 61% metals and 21% plastics (Widmer et al., 2005). In particular, Fe and Cu are the most common materials found in electrical and electronic equipment and account for the largest portion of the total metal weight of e-waste (Widmer et al., 2005; Cui and Zhang, 2008). Therefore, the effective separation of Cu and Fe is important in terms of e-waste management and recovery. In addition, the separation and recovery process used for Fe can be applied to other precious metal recovery processes.

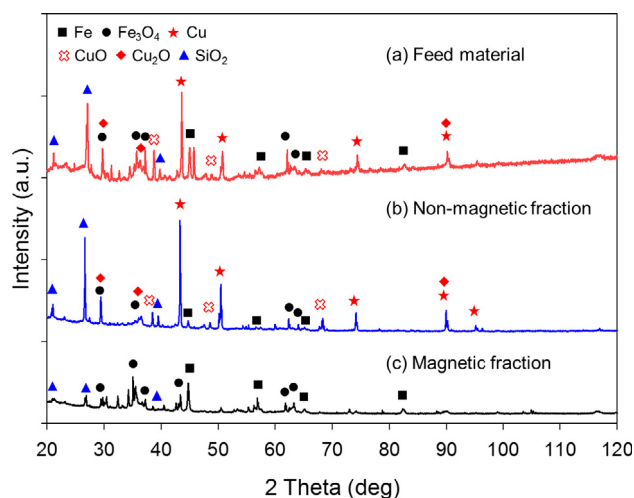
Fine materials (< 600  $\mu\text{m}$  particle size), such as flue dust, are generated in the processing of e-waste by mechanical and thermal methods. According to Balde et al. (2015) the total e-waste generated worldwide was estimated at approximately 41.8 million tonnes in 2014 and is expected to grow to 49.8 million tonnes in 2018. Therefore, flue dust,

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**Table 1**  
Composition analysis of the samples with magnetic separation.

Sample ( $< 600 \mu\text{m}$ )	Mass (Wt. %)	Assay (wt.%)						Distribution (%) – (calculated)					
		Cu	Zn	Fe	Mn	Al	Ca	Cu	Zn	Fe	Mn	Al	Ca
Feed material	100	6.2	0.9	27.1	0.7	3.6	1.7	101.8	99.4	98.6	98.6	98.3	97.1
Non-mag fraction	41	14.1	0.6	6.3	0.1	5.9	2.3	93.2	27.3	9.5	5.9	67.2	55.5
Mag fraction	59	0.9	1.1	40.9	1.1	1.9	1.2	8.6	72.1	89.0	92.7	31.1	41.6



**Fig. 1.** XRD patterns of samples after magnetic separation.

which is the by-product of the e-waste treatment process, will also be increasing. Some researchers have investigated the recovery of valuable metals from Cu smelter slag by a leaching process (Banza et al., 2002; Vítková et al., 2011). Yang et al. (2010) reported the selective extraction of base metals, such as Co, Zn, and Cu from Cu smelter slag at atmospheric pressure. However, the recovery and separation valuable metals such as Cu, Fe, Al, Au and Ag from fine materials (including flue dust) in the processing of e-waste have not been well studied. Therefore, more research on their recovery process is necessary.

In this study, the selective recovery and separation of Cu and Fe from fine materials of e-waste were examined. Magnetic separation was used as a physical method to remove ferrous materials. Cu was selectively recovered by using a leaching method as a chemical process. An acid leaching process was carried out with a variation of time and temperature in  $\text{HNO}_3$ ,  $\text{HCl}$ , and  $\text{H}_2\text{SO}_4$  solutions. The Cu dissolution kinetics was measured, based on the shrinking core model, to obtain the activation energy. For the selective recovery of Fe and Cu,  $\text{H}_2\text{SO}_4$  was used to dissolve Fe as the first leaching process and  $\text{HNO}_3$  was used for Cu dissolution as the second leaching process.

## 2. Experimental

An e-waste recycling company provided the fine materials for this work. In order to optimize the effective recovery process, size separation (RX-29, W.S. Tyler) was performed as a pre-treatment process and the max. size of the sieve was  $600 \mu\text{m}$ . Next, magnetic separation was carried out to remove ferrous materials. The LB-1 magnetic barrier separator (S.G. Frantz Co.) was used, which consists of an electromagnet with two long pole pieces. An additional piece of low-field

control (LFC-3) equipment was used for the separation of ferromagnetic materials from other magnetic and non-magnetic materials. The forward slope of the chute regulates the travel speed of the sample stream as a vertical slope under which the sample moves. The side slope of the chute is the horizontal slope in the left and right directions, and sample moves into magnetic and non-magnetic fractions under the influence of gravity, depending on the side slope. The LFC-3 can generate and control low magnetic fields at currents from 0 to 100 mA. The forward slope ( $20^\circ$ ) and the side slope ( $15^\circ$ ) of equipment were fixed and the applied current was 25 mA. In addition, the crystalline phase of the flue dust before and after magnetic separation was confirmed by XRD (Empyrean, PANalytical) equipped with a Cr tube. HighScorePlus (PANalytical) was used as analysis software of the XRD patterns.

Samples were fused with a lithium borate mixture (40 wt% lithium tetraborate, 60 wt% lithium metaborate) at  $1100^\circ\text{C}$  for 1 h. After the borate fusion process, the melt was dissolved in  $\text{HNO}_3$  for ICP-OES (Optima 8000, Perkin Elmer) analysis. This fusion process has several advantages, in that minerals are readily dissolved, no pressure vessel is needed, and a clear aqueous solution is obtained for a variety of analytical procedures (Verbeek et al., 1982).

Three acidic solutions,  $\text{HNO}_3$  (68–70%, GR ACS),  $\text{H}_2\text{SO}_4$  (95–98%, J. T. Baker) and  $\text{HCl}$  (36.5–38.0%, GR ACS) were used as leaching reagents. During leaching, solids/liquid content was fixed at 20 g/L and the bath temperature was changed from 20 to  $60^\circ\text{C}$ . A magnetic stir bar was also used to mix the solution during leaching, and the stirring speed was fixed at 200 rpm. In the case of a two-stage process, the solid/liquid ratio was increased to 100g/L to verify the effect of selective leaching. After leaching, the solid residue was separated from the leachate by vacuum filtration, and the leachate was analyzed by the ICP-OES process.

## 3. Results and discussion

### 3.1. Magnetic separation of the as-received sample

Table 1 shows the result of ICP-OES analysis of the sample with magnetic separation. The major elements of the feed material were Cu and Fe, and the compositions were about 6 wt% and 27 wt%, respectively. As shown in Fig. 1, the Fe in the feed material was confirmed to be magnetite ( $\text{Fe}_3\text{O}_4$ ) in a metallic Fe phase.  $\text{Fe}_3\text{O}_4$  is ferromagnetic and can be magnetized to become a permanent magnet (Wasilewski and Kletetschka, 1999). Therefore, it is easily separated by magnetic separation. In the non-magnetic fraction, the composition of Cu increased from 6.2 to 14.1 wt%, while the composition of Fe decreased from 27.1 to 6.3 wt%. However, approximately 6 wt% Fe still remained in the non-magnetic fraction after the magnetic separation. This finding is probably because some Fe particles and non-magnetic particles have aggregated and transferred to non-magnetic fractions. Therefore, the leaching process as a chemical method was used to selectively recover Cu and Fe in the non-magnetic fraction.

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