



Complex electronic waste treatment – An effective process to selectively recover copper with solutions containing different ammonium salts



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ABSTRACT

Recovery of valuable metals from electronic waste has been highlighted by the EU directives. The difficulties for recycling are induced by the high complexity of such waste. In this research, copper could be selectively recovered using an ammonia-based process, from industrially processed information and communication technology (ICT) waste with high complexity. A detailed understanding on the role of ammonium salt was focused during both stages of leaching copper into a solution and the subsequent step for copper recovery from the solution. By comparing the reactivity of the leaching solution with different ammonium salts, their physiochemical behaviour as well as the leaching efficiency could be identified. The copper recovery rate could reach 95% with ammonium carbonate as the leaching salt. In the stage of copper recovery from the solution, electrodeposition was introduced without an additional solvent extraction step and the electrochemical behaviour of the solution was figured out. With a careful control of the electrodeposition conditions, the current efficiency could be improved to be 80–90% depending on the ammonia salts and high purity copper (99.9 wt.%). This research provides basis for improving the recyclability and efficiency of copper recovery from such electronic waste and the whole process design for copper recycling.

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1. Introduction

Electronic waste or waste electrical and electronic equipment (WEEE) contains significant amount of valuable metals including copper, zinc, gold, silver, etc (Bigum et al., 2012; Cui and Zhang, 2008; Kiddee et al., 2013). It has been considered as one of the most important resources during urban mining. Processing of WEEE or recovering valuable metals from the waste is however energy intensive because of its complex nature. A typical industrial process is based on pyro-metallurgy including high temperature burning of organics, smelting of metal and oxides, refining and further electrochemical treatment after chemical leaching (Bigum et al., 2012). Comparing with high-temperature processes, a low temperature hydrometallurgical process is always preferable especially for WEEE with low calorific values (Akcil et al., 2015b; Havlik et al., 2011; Jha et al., 2013). However, it has been mostly focused

on mono-streams or relatively ‘clean’ WEEE, for instance, printed circuit boards (PCB), batteries and mobile phones after disassembly/dismantling (Koyama et al., 2006). Processing of a more complex type of electronic waste, usually mixtures of all kinds of end-of-life products or waste from different streams is still not well considered.

In previous research, the possibility to selectively extract metals from a highly complex industrial information and communication technology (ICT) waste by using a hydrometallurgical method has been approved to be feasible (Sun et al., 2015c; Xiao et al., 2013). Specifically, copper in the ICT waste could be selectively extracted by using an ammonia-ammonium carbonate solution. In the downstream to recover copper from the leached solution, direct copper electrowinning from the ammonia-based leaching solutions is usually introduced. It was found that the current efficiency is quite low comparing with traditional copper electrowinning from acidic solutions (Rudnik et al., 2014). With this concern, research was carried out trying to obtain Cu^+ in the ammonia-based solution in order to develop an energy saving copper electrowinning process by electrowinning Cu^+ instead of Cu^{2+} into metallic copper (Oishi et al., 2006). Cu^{2+} in the solution is in principle to react with metal-

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lic copper through the $\text{Cu}^{2+}/\text{Cu}^+$ redox reaction to form Cu^+ . However, it is difficult in practice to keep a high concentration of Cu^+ in the solution since Cu^+ is easily oxidised into Cu^{2+} even at low oxygen partial pressure. On the other hand, it is rather sensitive to the impurities contents in the solution and solvent extraction is usually required prior to copper electrowinning (Oishi et al., 2008). As a follow-up of our previous research (Sun et al., 2015b, c; Xiao et al., 2013), this research focuses on the role of different ammonium salts during copper recovery from the complex ICT waste. In both stages i.e. selective extraction of copper from the waste and copper electrowinning from the solution, the effects of ammonia salt are being identified, for the leaching kinetics and electrochemical features, respectively. During copper electrodeposition, instead of trying to maintain a high concentration of Cu^+ , ammonia salt is found to have significant effects on the current efficiency as well as the energy consumption. With this research, it is providing more established understanding on the nature of selective copper recovery from complex electronic waste.

2. Experimental

2.1. Materials

The ICT waste is provided by Van Gansewinkel Groep (VGG). The material has been pretreated and concentrated physically and shredded into size of smaller than or equal to 8 mm. This size is defined to be a limit since further shredding will significantly increase the processing cost. The material is inherent with high inhomogeneity from observation and contains a large fraction of sand, stones, glass, ceramics and plastic particles. Metallic materials are either trapped in non-metallic components or present as metal wires. The general compositions are given elsewhere (Xiao et al., 2013). Copper content in the ICT waste is 40.8 wt.%. Ammonia (25% in water, Alfa Aesar), ammonium carbonate, ammonium sulphate and ammonium chloride (99.0%, Alfa Aesar) are used in the experiments.

2.2. Experimental set-up and procedures

The ICT waste received from VGG was directly used as the feedstock for metal recovery. In each lab-scale experiment, a sample size of around 50 g was taken which would be a relatively representative sample size of the bulk composition according to our previous investigations (Xiao et al., 2013). The leaching experiments were carried out in a glass reactor and the detailed set-ups have been given in our previous research (Sun et al., 2015c). A round shape of the reactor could help to ensure a better mixing condition. Air was purged into the bottom of the reactor and the gas bubbles could be further broken up by the agitator. The gas flow rate was controlled by a flow meter. During leaching, system temperature was slightly fluctuating because of the exothermic reactions. A heating bath/thermo-stat with water was used to control the temperature and an additional thermometer was used to monitor the temperature fluctuation during leaching. The temperature was found to change within ± 2.5 °C of the required temperature. The pH values were detected by a pH meter in the whole leaching process (pH meter was calibrated at 18 °C). Prior to the leaching, the ammonium salt ($(\text{NH}_4)_2\text{CO}_3$, $(\text{NH}_4)_2\text{SO}_4$ and NH_4Cl) was firstly dissolved into the ammonia solution and demineralised water was added to reach a liquid volume of the required liquid to solid ratio (5 ml/1 g in current research). The starting ammonia concentration is set to be 7.8 wt.%. The solution was kept in the reactor for a period to reach the required temperature (18–45 °C) before the ICT waste was added. Liquid samples were taken at certain time intervals to track the leaching behaviour of different metals in the ICT

waste. The sample size was 1–2 ml which is considered to have insignificant effect on the liquid to solid ratio. Since the waste exhibits high heterogeneity, the residue after leaching was also analysed to diminish any discrepancy in composition. The extracted fraction of different metals is therefore calculated from

$$X_M = W_t / (W_f + W_r) \quad (1)$$

where W_t and W_f are the metal contents in the sampled solution at time t and the final solution, respectively; W_r is in the metal content in the final residue after leaching.

2.3. Copper electrodeposition

From the leached solution, copper is further recovered by electrowinning/electrodeposition process (galvanostatic). In previous research, cylindrical electrodes had been proved significant to improve the current efficiency (Sun et al., 2015b). The copper electrodeposition was conducted in a rectangular cell. A copper cylinder (height 10 mm \times diameter 10 mm), a height 10 mm \times diameter 40 mm graphite cylinder and the Ag/AgCl electrode (3 M KCl) were used as the cathode, anode and reference electrodes, respectively. The reference electrode was connecting to the solution via a salt bridge. The current efficiency was determined as the ratio of the experimental weight difference of the cathode before and after the electrodeposition to the theoretical weight change by assuming that metallic copper is deposited from the bivalent state. At a later stage, cylindrical electrodes were used with the aim to improve the current efficiency. Copper obtained at the cathode was analysed to determine the purity.

2.4. Characterisation

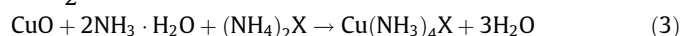
A sequential wavelength dispersive X-ray Fluorescence Spectrometry (XRF, PANalytical Axios) was used for compositional analyses of the bulk composition and the composition of the residue after leaching. The liquid solution was analysed by inductively coupled plasma – optical emission spectrometry (ICP-OES, PerkinElmer Optima 3000DV) and the residue was analysed by XRF. Although XRF has relatively high detection limit, the sample was prepared according to the procedures in Sun et al. (2015a) was pressed into pellets for analyses in order to minimise the inhomogeneity of the sample and ensure the reliability of the analyses. The morphologies of the ICT waste were both characterised with digital camera and scanning electron microscope (SEM, JEOL JSM 6500F) with energy-dispersive spectroscopy (EDS). The sample was polished and carbon coated when necessary. Liquid samples were diluted and prepared for analyses with ICP-OES.

3. Results and discussion

3.1. Copper leaching from ICT waste using different ammonium salts

3.1.1. Leaching efficiency

During copper extraction from the ICT waste using an ammonia-based solution, the role of ammonium salt is providing anions (e.g. CO_3^{2-}) to the ammine complex ($\text{Cu}(\text{NH}_3)_4^{2+}$) and proton to compensate with hydroxide anion generated during ammine complex formation. The reactions between copper and the leaching solution include two steps as (Meng and Han, 1996; Sun et al., 2015c)



where X represents an anion from the ammonium salt (carbonate or sulphate for instance).

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