



Comparative study on copper leaching from waste printed circuit boards by typical ionic liquid acids



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ARTICLE INFO

Article history:

Received 14 January 2015

Accepted 25 March 2015

Available online 11 April 2015

Keywords:

WPCBs

Waste electric and electronic equipment

(WEEE)

Ionic liquids (ILs)

Copper

Leaching kinetics

ABSTRACT

Waste printed circuit boards (WPCBs) are attracting increasing concerns because the recovery of its content of valuable metallic resources is hampered by the presence of hazardous substances. In this study, we used ionic liquids (IL) to leach copper from WPCBs. [BSO₃HPy]OTf, [BSO₃HMim]OTf, [BSO₄HPy]HSO₄, [BSO₄HMim]HSO₄ and [MIm]HSO₄ were selected. Factors that affect copper leaching rate were investigated in detail and their leaching kinetics were also examined with the comparison of [Bmim]HSO₄. The results showed that all six IL acids could successfully leach copper out, with near 100% recovery. WPCB particle size and leaching time had similar influences on copper leaching performance, while IL acid concentration, hydrogen peroxide addition, solid to liquid ratio, temperature, showed different influences. Moreover, IL acid with HSO₄⁻ was more efficient than IL acid with CF₃SO₃⁻. These six IL acids indicate a similar behavior with common inorganic acids, except temperature since copper leaching rate of some IL acids decreases with its increase. The results of leaching kinetics studies showed that diffusion plays a more important role than surface reaction, whereas copper leaching by inorganic acids is usually controlled by surface reaction. This innovation provides a new option for recovering valuable materials such as copper from WPCBs.

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1. Introduction

Waste electric and electronic equipment (WEEE), also called as electronic waste or e-waste, has been attracting more and more concerns from scientists, entrepreneurs, journalists to governors all over the world. First WEEE is increasing sharply due to the technology innovation and shorter life span, e.g., the lifespan of a personal computer (PC) and a central processing unit (CPU) decreased from 4.5 to 2–3 years (1992–2005) and from 4–6 to 2 years (1997–2005), respectively (Yazici and Deveci, 2013). E-waste generation, 3–5% of the total solid waste, is 2–3 times faster than solid wastes (U.S.-EPA, 2008; Erust et al., 2013). According to the United States Environmental Protection Agency (US-EPA), it is estimated that approximately 500 million computers were discarded annually between the years 2000 and 2007. Second, e-waste is known to all because of its notorious pollution caused by artisanal recycling. Guiyu, a small town in Guangdong Province, in the south of China,

is severely contaminated because people there recycle resources from e-waste by artisanal mining (Duan et al., 2011a). The concentrations of polybrominated diphenyl ethers (PBDEs) and polybrominated dibenzo-p-dioxins, dibenzofurans (PBDD/Fs) there were about 58–691 ng/g, 30 times higher than other urban sites (Duan et al., 2011b; Nguyen Minh et al., 2013). Heavy/toxic metals (Kolias et al., 2014; Maragkos et al., 2013; Oguchi et al., 2012), such as lead, cadmium, chromium, and persistence organic pollutants, e.g., polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), polybrominated biphenyls (PBBs), polychlorinated dibenzo-p-dioxins and dibenzofurans (PCDD/Fs), were found in surface water, underground water, soil, atmosphere, sludge under the river. More seriously, these highly toxicants were all found in human's blood, hair, urine, including fetus, gravely threatening the environment and human health (Huo et al., 2007; Leung et al., 2008; Zhao et al., 2008). The last reason and also the motivation is that WEEE contain valuable metals, mainly copper and precious metals (gold, silver and platinum) in particular. For example, copper (20%) and gold content (250 g/t) of a typical personal computer printed circuit board (PCB) are much higher

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than that of a copper or gold ore, i.e., 20–40 fold and 25–250 fold, respectively (Christian, 2006).

The key to WEEE recycle is waste printed circuit boards (WPCBs), which is an essential part of almost all electric and electronic equipment, such as laptops, air conditioners, TVs, etc. Therefore, both traditional and advanced methods, such as mechanical–physical approaches, especially corona electrostatic separation which has obtained great success in China (Li and Xu, 2010), hydrometallurgy (Kim et al., 2011), pyrometallurgy (Zhan and Xu, 2008), bioleaching (Yang et al., 2014) and sub/supercritical fluid (Xiu and Zhang, 2010), are widely discussed for the recovery of metals from WPCBs. Among these processes, hydrometallurgical processes offer high metal selectivity and recovery with a flexible mode of operation and suitability for small scale applications, making it an attractive alternative for the treatment of WPCBs. In previous studies, different lixiviant systems, including sulfuric acid, hydrochloric acid, nitric acid, iodide, ammonia, cyanide, thiourea, thiosulphate and microbes, were examined (Huang et al., 2014; Li et al., 2012; Oishi et al., 2007; Hu et al., 2006; Yang et al., 2011). In most cases, an oxidant, usually hydrogen peroxide (H_2O_2) and ferric (Fe^{3+}) due to their high reduction potentials of 1.78 V and 0.77 V, was involved given that metals in WPCBs are found in native and/or alloy form (Birloaga et al., 2013). However, there has been no process solely based on hydrometallurgical methods at the industrial scale (Yazici and Devci, 2013).

Recently, Zhu et al. found a novel method to recycle resources from WPCBs, using organic solvent dimethyl sulfoxide (DMSO) or ionic liquid 1-ethyl-3-methylimidazolium tetrafluoroborate ($[EMIM^+][BF_4^-]$) to dissolve bromine epoxy resin (Zhu et al., 2012a,b, 2013). Meanwhile, Zeng et al. also reported the use of water soluble ionic liquid 1-Butyl-3-methylimidazolium tetrafluoroborate ($[BMIM][BF_4]$) to melt solders, disassembling electric and electronic components from WPCBs (Zeng et al., 2013). Ionic liquids (ILs), also called as room temperature ionic liquids (RTILs), are basically liquid at low temperature. Typically, it consists of an organic cation with an inorganic or organic anion. ILs have a wide liquid temperature range and also possess numerous unique properties, such as negligible volatility, vapor pressure, thermal stability, high conductivity and wide electrochemical window. (Berenblyum et al., 2006). Currently, ILs were successfully applied in leaching metals from ores (Whitehead et al., 2007). However, the literature on leaching metals from WPCBs by ILs is limited.

In a previous study, Huang et al. found that 1-butyl-3-methylimidazolium hydrogen sulfate ($[bmim]HSO_4$) could successfully leach copper out from WPCBs (Huang et al., 2014). At optimized conditions, copper leaching rate was up to 99%. This leaching process can be modeled by the shrinking core model, showing that it is controlled by diffusion. In order to obtain a detailed and fundamental understanding about leaching copper from WPCBs by ILs, five typical ionic acids were selected and the results were compared with published data using $[Bmim]HSO_4$. Factors that affect copper leaching efficiency, such as WPCBs particle size, IL acid concentration, liquid to solid ratio, leaching time and temperature were studied in detail. In addition, their leaching kinetics were also investigated.

2. Materials and methods

2.1. Sample preparation

First, WPCBs, without disassembling electronic components, were cut into small pieces, around $50\text{ mm} \times 50\text{ mm}$. Then it was further shredded using Retsch SM-2000 Cutting Mill (Retsch, Germany), and sieved into five fractions: $F1 < 0.075\text{ mm}$, $0.075\text{ mm} < F2 < 0.1\text{ mm}$, $0.1\text{ mm} < F3 < 0.25\text{ mm}$, $0.25\text{ mm} < F4 < 0.5\text{ mm}$ and $F5 > 0.5\text{ mm}$, as described elsewhere (Huang et al.,

2014). After that, the obtained powders were digested by microwave aided HNO_3 – H_2O_2 –HF system (Yamasaki, 1997). Cu concentration of the digested solutions was analyzed by Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES, Thermo Scientific, iCAP 6500) and the results are shown in Table 1.

2.2. Leaching

All the leaching experiments were carried out in a batch of 250 mL glass conical flasks placed in a constant temperature water bath oscillator, using a constant oscillating frequency of 250 rpm at 40–70 °C. After each experiment, the leachate was filtered through a glass fiber filter of 0.45 μm pore size and preserved using 2 mL nitric acid before being analyzed by ICP-OES. Five ionic liquid acids, $[BSO_4HPy]HSO_4$ (N-sulfobutylpyridinium hydrosulfate), $[BSO_3HMIm]HSO_4$ (1-sulfobutyl-3-methylimidazolium hydrosulfate), $[BSO_3HMIm]OTf$ (N-sulfobutylpyridinium trifluoromethanesulfonate), $[MIm]HSO_4$ (Methylimidazolium hydrosulfate), $[BSO_3HPy]OTf$ (1-sulfobutyl-3-methylimidazolium trifluoromethanesulfonate), were provided by Lanzhou Institute of Chemical Physics, Chinese Academy of Science, and their structures as well as the structure of $[bmim]HSO_4$ are given in Table 2. Hydrogen peroxide (H_2O_2 , 30 wt.%) was used as oxidant. Chemicals used in the experiments were all analytical reagents unless otherwise mentioned. Factors that affect the leaching efficiency for the five IL acids were investigated separately, which were WPCBs particle size, IL acid concentration (V/V, in liquid solution), IL acid adding amount, H_2O_2 adding amount, temperature and time, and the detailed parameters associated with each experiment are given in Table 3. When concerning about the reaction time, 2 mL suspension was sampled at certain fixed intervals and filtered by a 0.45 μm glass fiber filter prior to determination. Data of $[bmim]HSO_4$ leaching system were cited from a previous study (Huang et al., 2014).

The copper leaching rate was expressed as the percentage of copper extracted into leaching solution from original specimen. For quality control, the relative standard deviations of the triplicates were in the limited ranges of a certified commercial laboratory and mean values are given in the Tables and Figures without error bar.

3. Results and discussion

3.1. Particle size

As it can be seen in Table 1, Cu content varies as WPCBs particle sizes, e.g., Cu concentration increases from 6.75% to 20.36% when WPCBs particle size increases from $<0.075\text{ mm}$ to $>0.5\text{ mm}$. It is reasonable that the smaller the particle size, the lower the Cu content because, compared to polymers and ceramics, it is more difficult to smash metals to fine powders. This result consists with the study of Veit et al. (2006), which reported that copper enriched in the fraction of 0.5–1 mms. Since copper contained in the fraction of particle size $>0.5\text{ mm}$ is the highest, this fraction was selected for the study of other sections.

Particle size plays a crucial role in metal recovery, and most of the recycling processes have a certain effective size range. Zhu et al. (2011) showed that the maximum leaching efficiency of

Table 1
Copper contents in WPCBs specimens.

Particle size, mm	F1 ($<0.075\text{ mm}$)	F2 (0.075–0.1 mm)	F3 (0.1–0.25 mm)	F4 (0.25–0.5 mm)	F5 ($>0.5\text{ mm}$)
Cu wt. %	6.75	6.87	10.25	19.62	20.36

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