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Leaching behavior of copper from waste printed circuit boards with Brønsted acidic ionic liquid

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ABSTRACT

In this work, a Brønsted acidic ionic liquid, 1-butyl-3-methyl-imidazolium hydrogen sulfate ([bmim]HSO_4), was used to leach copper from waste printed circuit boards (WPCBs, mounted with electronic components) for the first time, and the leaching behavior of copper was discussed in detail. The results showed that after the pre-treatment, the metal distributions were different with the particle size: Cu, Zn and Al increased with the increasing particle size; while Ni, Sn and Pb were in the contrary. And the particle size has significant influence on copper leaching rate. Copper leaching rate was higher than 99%, almost 100%, when 1 g WPCBs powder was leached under the optimum conditions: particle size of 0.1–0.25 mm, 25 mL 80% (v/v) ionic liquid, 10 mL 30% hydrogen peroxide, solid/liquid ratio of 1/25, 70 °C and 2 h. Copper leaching by [bmim]HSO_4 can be modeled with the shrinking core model, controlled by diffusion through a solid product layer, and the kinetic apparent activation energy has been calculated to be 25.36 kJ/mol.

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1. Introduction

Printed circuit boards (PCBs) are widely used in most electric and electronic equipment (EEE), such as home appliances and P.C. etc. It is reckoned that the average rate of PCBs manufacture increased by 8.7% in the world, and the number is much higher in China (14.4%) (Cui and Forssberg, 2003; Huang et al., 2009). At the same time, the replacement of EEE is accelerated by both science and technology innovation and market expansion, which results in a huge quantity of waste PCBs (WPCBs). WPCBs is a complex material containing both hazardous substances (i.e., heavy metals and halogenated flame retardants etc.) and valuable matters (i.e., plastic, resins and metals etc.). Especially, WPCBs contain around 20 wt.% of copper as metallic constituents, and the concentration of copper in WPCBs is 10 times more than that of rich-content mineral (Li et al., 2007; Liang et al., 2010; Ilyas et al., 2007; Xiu and Zhang, 2009). Therefore, recycling of WPCBs has become an important issue in the world, not only from the perspective of environmentally harmful, but also with regard to the recovery of abundant valuable materials (Veit et al., 2006; Quan et al., 2010).

Thus far, many studies have been carried out to recover copper from WPCBs, including mechanical (Li et al., 2007, 2008; Das et al., 2009), pyrometallurgical (Grause et al., 2008; Lung et al., 2007; de Marco et al., 2008), bioleaching (Chi et al., 2011; Zhu et al., 2011; Wang et al., 2009), supercritical fluid (Xiu and Zhang, 2010; Chien et al., 2000) and hydrometallurgical (Kim et al., 2011; Xiu and Zhang, 2009; Zhu et al., 2009) processes. Mechanical process was employed to separate metals and nonmetals from WPCBs, and it cannot efficiently recover precious metals, and the metal concentrate obtained from the mechanical process need further purification. Therefore, the mechanical process is usually used as pretreatment (Cui and Zhang, 2008). As a conventional method, pyrometallurgical process was widely used to recover non-ferrous metals as well as precious metals from WPCBs in the past decades. However, the prospect of pyrometallurgical process will be limited because of the atmospheric pollution (Li et al., 2007). Recently, using microorganism to bioleach metals from WPCBs was considered as a promising alternative process, but only few data were available about the bioleaching metals from WPCBs. In addition, WEEE and WPCBs have shown bacterial toxicity in the bioleaching process due to the presence of non-metallic components thus affected the recovery rate of metals (Ilyas et al., 2010). Therefore, how to improve metals recovery in bioleaching from WPCBs need further research. Properties of supercritical fluid such as the negligible surface tension, high diffusivity, low viscosity and low solubility of inorganic salts are very unique especially in disposal of toxic compounds (Song and Wang, 2000), and the process was developed for the treatment of toxic organic matters in WPCBs recently. However, the critical temperature and pressure of supercritical fluid are very high which causes serious reactor corrosion and higher energy consumption. In the last few decades, hydrometallurgical processes have been given considerable attention

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to recover precious metals as well as base metals due to the simplicity and less energy consumer. Leaching is the first step in the extraction of metals by hydrometallurgical process (Kim et al., 2011). Some leaching processes have been developed to recover copper from WPCBs for their high leaching selectivity to date, the leaching system including nitric acid, ammoniacal sulfate and chloride solution (Yang et al., 2011). However, many of the processes have not reached commercial-scale operation due to various drawbacks, such as a large amount of waste acid liquid were produced during the processes, which must be treated carefully; furthermore, the flow of recycling metals in WPCBs was long and complicated which causes a higher recovery cost (Zhou and Qiu, 2010). Therefore, it is of great significance to develop new and green technologies for the recycling of copper with low consumption of energy and reagent, as well as free of pollutants release. The availability of room temperature ionic liquids makes it possible to explore a green leaching process for ores as it was demonstrated (Dong et al., 2009; Whitehead et al., 2007).

Room temperature ionic liquids (RTILs), also called ionic liquids (ILs), which are basically liquid at low temperature typically consisting of an organic cation with an inorganic or organic anion and have a wide liquidus temperature range (Dong et al., 2009). Numerous unique properties, such as negligible volatility and vapour pressure, thermal stability, high conductivity and wide electrochemical window, considered ionic liquids as very promising replacements for the traditional organic solvents (Whitehead et al., 2007). Because of these characteristics, ionic liquids have been increasingly investigated in the world in organic syntheses (Dubreuil and Bazureau, 2001), catalytic reactions (Berenblyum et al., 2006), electrochemical applications (Kumar and Hashmi, 2010), separation (Gu et al., 2004), biochemistry (Yang and Pan, 2005) and material engineering (Ma et al., 2012). There were several studies about leaching metals from ores. However, few experiments were reported on leaching metals from WPCBs. In this study, an ionic liquid 1-butyl-3-methyl-imidazolium hydrogen sulfate ([bmim]HSO_4) was used to leach copper from WPCBs directly and the kinetics of the leaching process were analyzed as well.

2. Materials and experimental methods

2.1. WPCBs preparation

WPCBs, mounted with electronic components, were firstly cut into small pieces of about 50 mm × 50 mm by cutting machine. Then these pieces were shredded by cutting mill (Retsch SM2000), and sieved into different fractions using standard sieves: $F_1 < 0.075$ mm, 0.075 mm $< F_2 < 0.1$ mm, 0.1 mm $< F_3 < 0.25$ mm, 0.25 mm $< F_4 < 0.5$ mm and $F_5 > 0.5$ mm. After that they were dried at 105 °C for 24 h.

2.2. Leaching experiments

All leaching experiments were carried out in a 250 mL glass conical flask placed in a constant temperature water bath oscillator, using a constant oscillating frequency of 250 rpm in the temperature range of 40–70 °C. The ionic liquid ([bmim]HSO_4, analytical reagent, Lanzhou Institute of Chemical Physics, Chinese Academy of Science) concentration (v/v) in aqueous solution were 10%, 20%, 40%, 60% and 80%, and hydrogen peroxide (30 wt.%) was used as oxidant.

Copper concentration in the leach liquor was analyzed by Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES, Thermo Scientific, iCAP 6500). The relative standard deviations of the triplicates were in the limited ranges of a certified commercial laboratory and mean values were given in the tables and figures without error bar. The copper leaching rate was expressed as the percentage of copper extracted into leaching solution from original specimen, which was calculated according to the following formula:

$$\text{Copper leaching rate} = \frac{\text{copper extracted into leaching solution}}{\text{total copper in WPCBs specimens}} \times 100\% \quad (1)$$

2.3. Characterization

The obtained WPCBs powder was digested using microwave aided HNO_3–H_2O_2-HF system (Yamasaki, 1997). Metal concentrations of the digested solution were tested by ICP-OES. The results were presented in Table 1. It can be seen from Table 1 that the metal contents of WPCBs with different particle sizes were different. As WPCBs particle size increased from <0.075 mm to >0.5 mm, the content of Cu increased from 6.75% to 20.36%, Al increased from 8.70% to 11.18% and Zn increased from 1.28% to 2.24%. However, the content of Ni decreased from 0.31% to 0.07%, Sn decreased from 1.21% to 0.10% and Pb decreased from 1.17% to 0.62% when particle size increased from <0.075 mm to >0.5 mm. Content of Ba increased from 2.98% to 3.47% when particle size increased from <0.075 mm to 0.25 mm, and then decreased to 2.00% when particle size >0.5 mm.

The phases of WPCBs specimen and leach residue were identified by X-ray diffraction (XRD, PANalytical B.V., X'Pert PRO), as given in Fig. 1.

3. Results and discussion

3.1. Effect of particle size

Numerous studies were done to recover metals from WPCBs; however, few research works were carried out on the effect of particle size on copper leaching recovery. Veit et al. (2006) reported that by mechanical techniques, metals enriched in the fraction of 0.5–1 mm, which was selected to conduct electrowinning tests because of the higher metal content in this fraction. It was reasonable that the smaller particle size, the lower metal content because there is a higher difficulty in milling metals than polymers and ceramics (more concentrated in the smaller fractions). Nevertheless, Zhu et al. (2011) performed experiments on bioleaching copper from WPCBs, and reported that the maximum leaching efficiency of copper was obtained when the particle size of metal concentrate was 60–80 mesh (about 18 mm–25 mm), and the leaching efficiencies were almost the same with or lower than that of 60–80 mesh when the particle size of metal concentrates were less than 80 mesh and 40–60 mesh (about 25 mm–42 mm). Kim et al. (2011) employed selective leaching of gold and copper from

Table 1
Contents of major metal in WPCBs specimens.

	F_1	F_2	F_3	F_4	F_5
Cu (wt.%)	6.75	6.87	10.25	19.62	20.36
Al (wt.%)	8.70	8.65	8.32	12.73	11.18
Ni (wt.%)	0.31	0.26	0.21	0.19	0.07
Sn (wt.%)	1.21	0.72	0.94	0.95	0.10
Pb (wt.%)	1.67	0.97	1.29	1.42	0.62
Zn (wt.%)	1.28	1.23	1.30	2.21	2.24
Ba (wt.%)	2.98	2.99	3.47	2.37	2.00

Notes: WPCBs powder was sieved into different fractions using standard sieves: F1 < 0.075 mm, 0.075 mm < F2 < 0.1 mm, 0.1 mm < F3 < 0.25 mm, 0.25 mm < F4 < 0.5 mm and F5 > 0.5 mm. After that they were dried at 105 °C for 24 h.

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