



Leaching copper from shredded particles of waste printed circuit boards

Haiyu Yang^a, Jingyang Liu^b, Jiakuan Yang^{a,*}

^a School of Environmental Science and Engineering, Huazhong University of Science and Technology, Wuhan 430074, China

^b Research Center for Cleaner Production and Circular Economy, Chinese Research Academy of Environmental Sciences, Beijing 100012, China

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ABSTRACT

Leaching copper from shredded particles of waste printed circuit boards (PCBs) was carried out in sulfuric acid solution using hydrogen peroxide as an oxidant at room temperature. The influence of system variables on copper recovery by leaching was investigated, such as sulfuric concentration, amount of hydrogen peroxide addition, waste PCBs particle size, presence of cupric ion, temperature and time. The results shown that the optimum addition amount was 100 mL 15 (wt%) sulfuric acid solution and 10 mL of 30% hydrogen peroxide for leaching 10 g waste PCBs powder with a solid/liquid ratio of 1/10 for 3 h at room temperature (~23 °C). Moreover leaching temperature and initial copper ion concentration had insignificant effect on the leaching recovery of copper. The effect of different particle size of shredded waste PCBs on leaching of copper was investigated under the optimum leaching condition. The results revealed that shredding pieces of waste PCBs smaller than 1 mm was efficient and suitable for copper leaching. Then the leaching solution was concentrated to crystallize $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, and crystal liquor was reused for the next cycles.

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1. Introduction

Printed circuit boards (PCBs) are the base of the electronic industry as it is an essential part of almost all the electric and electronic equipment (EEE). Both technological innovation and market expansion accelerate the replacement of EEE, which leads to a significant increase of waste PCBs [1]. Waste PCBs has been attracting the public attention by its environmentally harmful materials and abundant valuable non-ferrous metals. If the waste PCBs are improperly disposed, hazardous materials could cause serious environmental problems and numerous valuable metals would be lost. Therefore, recovery of valuable metals from waste PCBs is one of hot topics in industrial waste reclamation [2].

It can be noted from review of the literatures that, the processes for recovering metals from waste PCBs include mechanical [3–6], pyrometallurgical [7,8] and hydrometallurgical [9–11] methods. The mechanical process is usually used as a pre-treatment process before pyrometallurgy and/or hydrometallurgy, such as shredding the boards into fine particles for improving valuable metal recovery and processing operations. Pyrometallurgical process can efficiently recover copper and precious metals, which need further refined. However, this high-temperature process easily causes serious environmental problems, especially air pollution. Pyrometallurgical methods currently are seldom used in recovery of waste PCBs for their shortcomings. Hydrometallurgy may be

more exact, more predictable and more easily controlled comparing with pyrometallurgical process [12]. So far, hydrometallurgical processes have been widely applied in recovering metal components from waste PCBs using mechanical as pre-treatment process. However, most of previous researches focused on recovering precious metals for its higher value [13]. With the development and innovation of manufacturing of PCBs, the content of precious metals is lower and lower. Thus, recovery of main metals from waste PCBs becomes more and more evident. The previously related studies were mainly focused on the recovery of main metals such as copper, tin and lead simultaneously [14–16]. Therefore the composition of leaching solution was complicated which resulted in difficulty to separate the metals. Additionally few experiments were reported on investigating the effect of the size of mechanically shredded particles on the metal recovery by leaching with hydrometallurgical process.

In this regard, a green process for leaching copper from shredded particles of waste PCBs at room temperature was investigated in the present research. The objective of the research is to recover valuable metal copper by leaching process with solutions of sulfuric acid and hydrogen peroxide. The effect of the waste PCBs particle size on the leaching behavior of copper will be discussed as well.

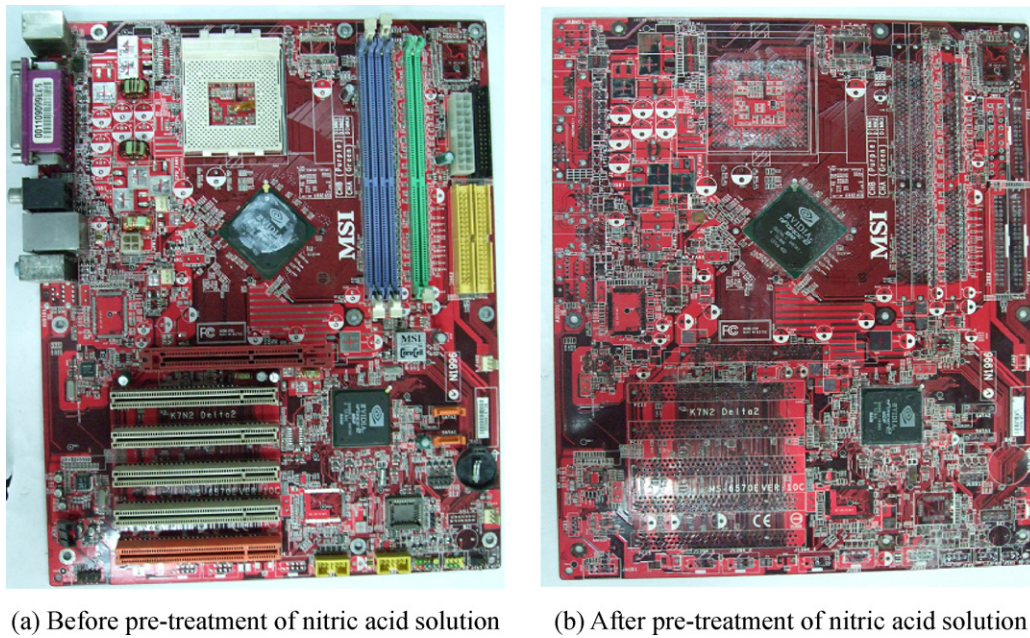
2. Materials and experimental procedures

2.1. Preparation of the PCBs

Twenty pieces of different kinds of waste computer mainboards (about 10 kg) with different sizes were immersed in dilute nitric

* Corresponding author. Tel.: +86 27 87792207; fax: +86 27 87792101.

E-mail addresses: yjiakuan@hotmail.com, jkyang@mail.hust.edu.cn (J. Yang).



(a) Before pre-treatment of nitric acid solution

(b) After pre-treatment of nitric acid solution

Fig. 1. Photos of printed circuit board obtained from waste mainboards: (a) before pre-treatment of nitric acid solution; (b) after pre-treatment of nitric acid solution.

acid solution, in which welding-jointed electronic components were removed, as presented in Fig. 1. About 4 kg of waste PCBs was selected from 10 kg waste mainboards. Waste PCBs were firstly cut into small pieces of about $3\text{ cm} \times 3\text{ cm}$ by pliers. Then the pieces were pretreated by liquid nitrogen to increase the crushability of waste PCBs and shredded by cutting mill (Retsch SM2000) with 8 mm final fineness. Then the samples obtained were mixed uniformly for later experiment.

The schematic of specimen preparation and leaching process is presented in Fig. 2. Soldering tin reacts with nitric acid to form insoluble stannic acid and soluble lead nitrate. In this way, tin is separated from lead. Then tin was recovered as the form of stannic acid. The lead ion in leaching solution was precipitated as PbSO_4 with adding sulfuric acid. Copper exists in the middle layer of the boards and it does not expose in nitric acid solution at the beginning reaction stage. Copper in the middle layer just reacts with nitric acid

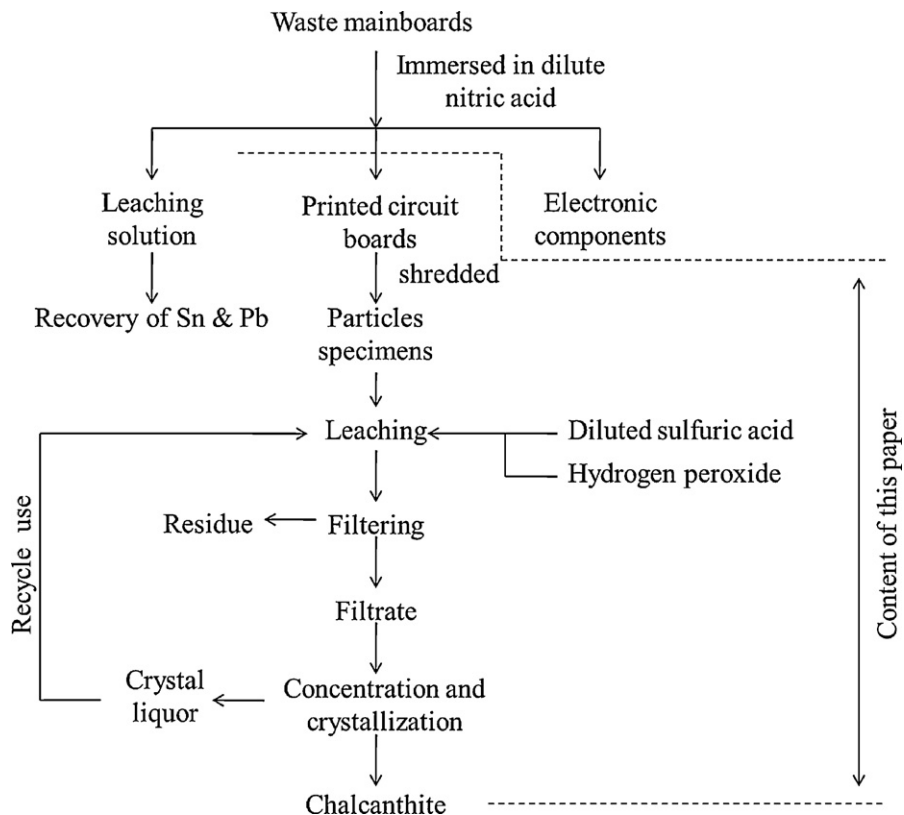


Fig. 2. Process of leaching copper from particles of PCBs.

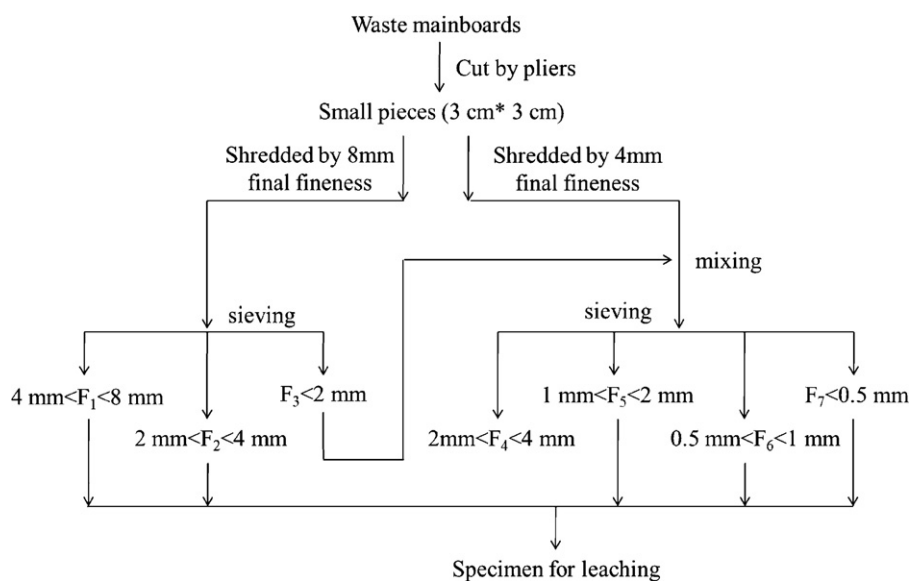


Fig. 3. Preparation of the shredded specimen of different sizes for leaching experiment.

Table 1

Contents of metal in waste PCBs specimens.

Element	Cu	Sn	Pb	Fe	Ni	Au	Ag	Cd	Cr	Mn	Pd	Pt	Others
Content (wt%)	25.30	0.3301	0.1357	0.1533	0.0222	0.0105	0.0007	BDI	0.0024	0.0036	BDI	BDI	74.04

Notes: BDI means below detectable limit.

solution when the soldering-tin completely reacts with nitric acid solution. Therefore, copper almost did not leach out by controlling the concentration of nitric acid and the immersed time. The concentration of the nitric acid used for preparation of waste PCBs is about 2 mol/L and the PCBs were immersed for about 3 h at room temperature. The waste mainboards mainly contained metal copper after the pretreatment. The current research just focuses on the characterization of copper leaching in dilute sulfuric acid solution using hydrogen peroxide as oxidant.

2.2. Material characteristics of waste PCBs specimen

Aqua regia and perchloric acid was used to fully leach the metals from the specimen. The metal concentrations of the digestion solution were tested by Atomic Absorption Spectrophotometer (Hitachi, Z2000). The digestion results are showed in Table 1.

X-ray diffraction pattern was investigated on grounded waste PCBs powders by X-ray diffractometer (XRD) (PANalytical B.V. company, X' Pert PRO), and the result indicates that the copper in the PCBs exists as metal copper.

2.3. Leaching procedures of waste PCBs specimen

Specimen sampled from particles shredded by 8 mm final fineness by coning and quartering method were shredded gradually to pass the 1 mm final fineness for leaching experiment.

Analytical grade commercial concentrated sulfuric acid, hydrogen peroxide (30%) and chalcantite were used as received in experiments. Leaching experiments were carried out in a 200 mL

conical flask under magnetically stirring at room temperature ($\sim 23^\circ\text{C}$). Initial concentration of copper ion was adjusted by adding chalcantite.

2.4. Shredding and size separation of waste PCBs specimen

Samples were sieved into different fraction by sieve shaker (Retsch, AS200) using a series standard sieve with a pore size of 8 mm, 4 mm, 2 mm, 1 mm, and 0.5 mm. Specimen sampled from particles shredded by 8 mm final fineness by coning and quartering method was separated into three different fractions by sieve shaker (amplitude = 1.0 mm/g, interval = 30 s, interval on, time = 5 min), i.e. fraction $4\text{ mm} < F_1 < 8\text{ mm}$, $2\text{ mm} < F_2 < 4\text{ mm}$ and $F_3 < 2\text{ mm}$. The sample shredded by 4 mm final fineness and fraction F_3 were mixed and sieved into four different fractions: $2\text{ mm} < F_4 < 4\text{ mm}$, $1\text{ mm} < F_5 < 2\text{ mm}$, $0.5\text{ mm} < F_6 < 1\text{ mm}$ and $F_7 < 0.5\text{ mm}$. The shredding process is showed in Fig. 3. The sample F_1 , F_2 , F_5 , F_6 and F_7 were utilized to study effects of particle sizes on copper recovery. Copper content in sections F_1 , F_2 , F_5 , F_6 and F_7 is showed in Table 2. The results show that individual shredded fractions with different particle sizes have different copper content. The particles of leaching residues of sections F_1 , F_2 and F_5 were too large to be digested completely. Therefore all those residues were separately collected and then calcined in muffle furnace at 600°C for 2 h. The residues calcined were then digested to determine the copper content in residues. Leaching residues of sections F_6 and F_7 with fine particle sizes were digested directly. Specimens of shredded waste PCBs are not homogenous, especially for sampling specimen of large particles. Therefore, material balance method was used to deduce

Table 2

Copper content in individual fractions.

Individual fraction (mm)	4–8 (F_1)	2–4 (F_2)	1–2 (F_5)	0.5–1 (F_6)	<0.5 (F_7)
Copper content (wt%)	22.75	21.94	27.93	27.19	12.17

the total amount of copper in shredded waste PCBs specimens, expressed as copper extracted into leaching solution + copper in residues. The amount of recovered copper was expressed as the amount of copper extracted into leaching solution from original specimen, deduced as copper in final leaching solution – copper in initial leaching solution. Therefore, copper recovery could be expressed as the percentage of copper extracted into leaching solution from original specimen, which was calculated as the following formula:

$$\text{Copper recovery} = \frac{\text{Copper extracted into leaching solution}}{\text{Total copper in shredded specimens}} \times 100\%$$

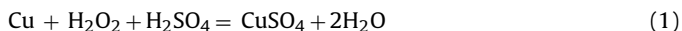
3. Results and discussion

3.1. Effect of different leaching conditions on copper leaching

Oishi et al. [17] and Alam et al. [18] carried out numerous experiments on recovery of copper from waste PCBs. The leaching system they used was ammoniacal sulfate or chloride solutions for its high leaching selectivity of copper. For there was a high impurity content (Sn 4.9%, Fe 3.4%, Pb 3.2%, etc.) in original materials. The leaching solution was purified with solvent extraction by LIX 26. The purified solution was then electrowon to obtain high purity metal copper.

As Table 1 shows, the samples used for current experiment contains about 25.3 (wt%) of copper and other heavy metals are pretty low (below 0.35%). The solvent extraction is not necessary to purify the leaching solution since other impurities of metals are much low, i.e. Sn, Fe, and Pb, etc. Therefore, sulfate acid leaching system could be suitable for waste PCBs samples used in this study to obtain cupric sulfate directly. Oxidants commonly used in sulfuric acid system for leaching copper from waste PCBs are air or hydrogen peroxide. Air is a promising oxidant for the consideration of economy. Hydrogen peroxide is not regenerable and its price is higher, however, the condition of experiment is easily controlled, and the leaching reaction is more effective comparing to other oxidant, i.e. air. Therefore, this work only focuses on hydrogen peroxide as oxidant, and air will be comparatively investigated in our further research.

In the leaching system of sulfuric acid and hydrogen peroxide, the overall reaction for copper dissolution can be expressed by reaction follows Eq. (1):



10 g of waste PCBs sample contains about 2.53 g metal copper. The copper theoretically needs 3.98 g concentrated sulfuric acid and 3.1 mL hydrogen peroxide (30 wt%) to fully dissolve into aqueous phase according to Eq. (1). However, as the processes are designed, the raffinate from crystallization of cupric sulfate would be reused for copper leaching. In this regard, the influence of copper ion concentration on copper leaching kinetics was investigated.

Copper recovery from waste PCBs specimen was carried out at room temperature (~23 °C). There are many factors influencing the leaching rate or copper recovery such as sulfuric acid concentration, amount of hydrogen peroxide added, copper ion concentration, solid–liquid ratio, leaching temperature and time.

Fig. 4a shows the copper recovery by leaching at different concentration of sulfuric acid. The recovery increases with the increase of the concentration of sulfuric acid. It appears that recovery reaches the maximum when the concentration at 15% of sulfuric acid and then keeps constant. Fig. 4b demonstrates the copper recovery by leaching with variation of hydrogen peroxide addition. It can be seen from Fig. 4b that copper recovery increases with the increase of hydrogen peroxide concentration until addition of hydrogen peroxide reaches to or over 10 mL. Oxygen produced from the decomposition of hydrogen peroxide reacts with metal

copper to form cupric oxide, which can further react with sulfuric acid to form cupric sulfate. Thus sulfuric acid and hydrogen peroxide actively together act as the reactants. In this way, the copper recovery increases with the increase of their concentrations. The solubility of oxygen in water is only 0.031:1 of oxygen to water in volume at 20 °C. Solubility of oxygen in sulfuric acid is slightly decreased (about 2%) in diluted sulfuric acid corresponding value with for pure water at 25 °C according the Ref. [19]. It is expected that the copper recovery does not increase anymore when the amount of hydrogen peroxide added is over 10 mL under the condition investigated.

Zhu et al. [20] carried out the test of copper leaching from the metal powders of waste PCBs in sulfuric acid solution using Cu^{2+} ion and air as oxidants in the presence of chloride ion as a catalyst. Fig. 4c presents the effect of copper (II) concentration on copper recovery. And there is no significant impact on copper recovery when the concentration of copper ion is below 13 g/L. It decreases when the initial concentration is over 13 g/L and the copper recovery decreases to less than 80% when the initial copper concentration reaches 25 g/L. These results may indicate that initial copper ion does not play a role of oxidant because copper ion added at the beginning does not accelerate the copper recovery. Copper ion (I) is unstable in the water solution and can disproportionate to cupric ion and element copper quickly unless chlorine ion is present as ligand to form stable complex CuCl_2^- . It can be concluded that the presence of copper ion does not accelerate the copper recovery as shown in Fig. 4c and that a high initial concentration of copper ion (≥ 13 g/L) does decrease the copper recovery at the condition studied. The decrease in copper recovery may be restricted by the solubility of the CuSO_4 in sulfuric acid.

Fig. 4d presents the effect of solid–liquid ratio on copper recovery. The recovery increases with the decrease in solid–liquid ratio. The decrease in solid–liquid ratio means a smaller volume of leaching solution, which increases the reaction temperature and the efficiency of mass transfer to accelerate the copper recovery.

Fig. 4e demonstrates the relationship between copper recovery and reaction temperature. The result shown in Fig. 4e indicates that temperature has no significant impact on the copper recovery when it is in a range from 20 °C to 50 °C. With the increase of the reaction temperature, the reaction and the decomposition of hydrogen peroxide will be speeded. However, the content of dissolved oxygen in aqueous phase decreases, which results in that there is no significant impact on copper recovery.

The results of copper recovery by leaching versus time are presented in Fig. 4f. It can be found from Fig. 4f that the copper recovery has no significant increase when the leaching time is extended more than 3 h. Based on this phenomenon, the copper recovery at 2 h was adopted to examine the influence of system variables on it.

It can be summarized from the experiment results above that the optimum condition of leaching copper from 10 g waste PCBs powders is 100 mL of 15 (wt%) sulfuric acid, 10 mL hydrogen peroxide, and solid–liquid ratio of 1/10 for 3 h at room temperature (~23 °C) with an initial concentration of copper ion range from 0 g/L to 13 g/L.

3.2. Effect of particle sizes on copper recovery

Some research works were done to recover metal from waste PCBs. Nevertheless, few studies were carried out on the effect of shredded particle size on copper leaching behavior. Yang et al. [21] performed experiments on bioleaching copper from waste PCBs and the particle size of the specimen was less than 0.5 mm. Xiu and Zhang [14] recovered copper from waste PCBs by supercritical water oxidation combining with electrokinetic process and the materials used in his research was the fraction less than 0.1 mm.

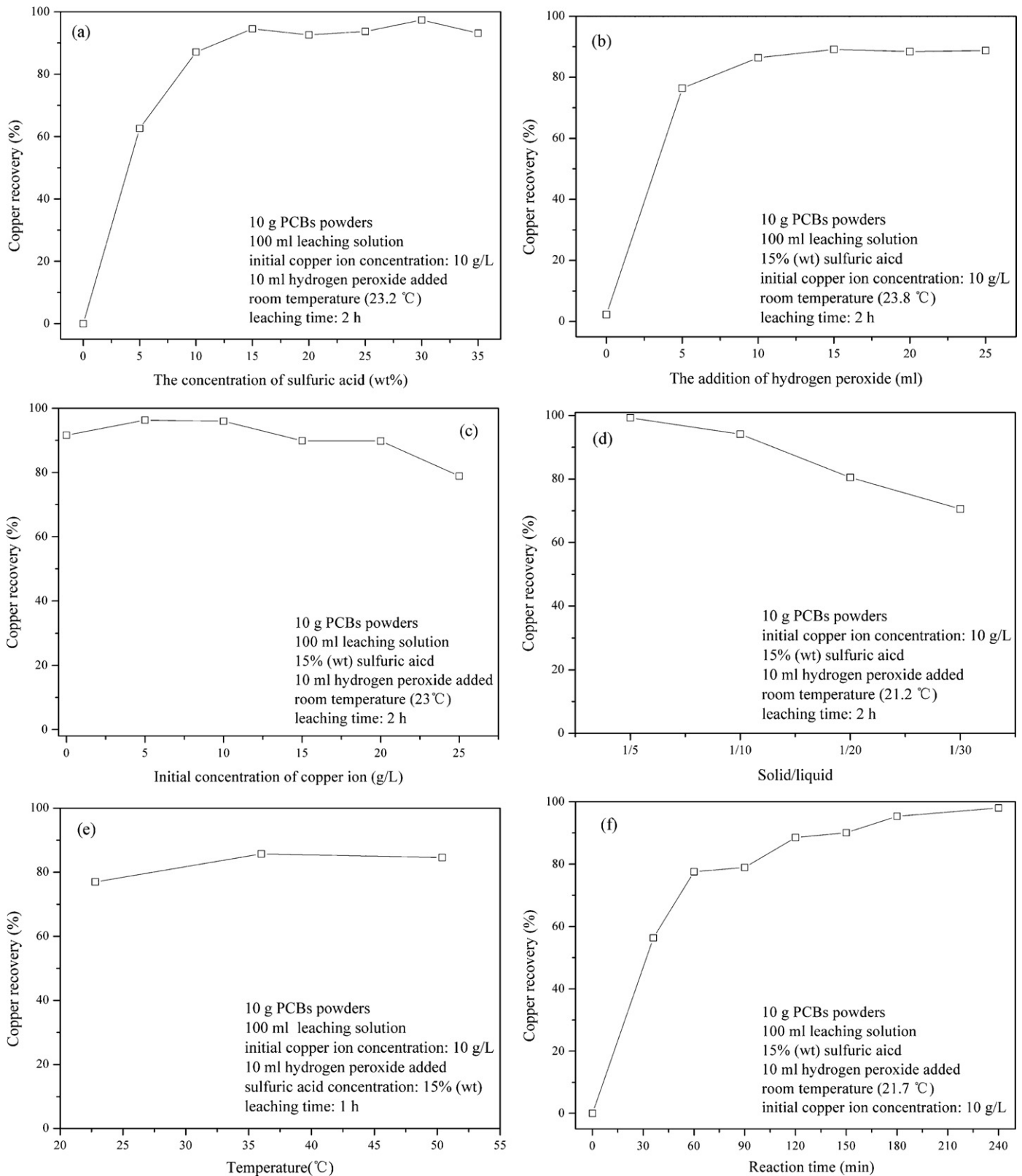


Fig. 4. Effects of different leaching conditions on copper recovery: (a) effect of dilute sulfuric acid concentration on copper recovery; (b) effect of the amount hydrogen peroxide addition on copper recovery; (c) effect of concentration of initial copper ion on copper recovery; (d) effect of solid/liquid ratio on copper recovery; (e) effect of temperature on copper recovery; (f) effect of reaction time on copper recovery.

The size of the specimen Mecucci and Scott [15] used to recover copper, tin and lead by nitric acid was about 2.5 mm². The samples Oishi et al. [17] and Alam et al. [18] used for copper leaching were the fraction of 0.5–3 mm. Shredding waste PCBs was a high-energy

consumption process and it was extremely hard to shred all the waste PCBs samples smaller than 0.5 mm in practice.

In the present research, the influence of shredded particle size on copper leaching behavior was examined. Fig. 5 presents the

Table 3
Weight percentage of different section shredded by different final fineness (%).

Final fineness of the cutting mill (mm)	<0.15 (mm)	<0.25 (mm)	<0.5 (mm)	<1.0 (mm)	<2.0 (mm)	<4.0 (mm)	<8.0 (mm)
8				10.82	22.44	81.36	100
4			12.88	23.08	60.31	100	
1		28.93	43.93	86.22	99.99		
0.5	33.02	47.46	90.37	100			

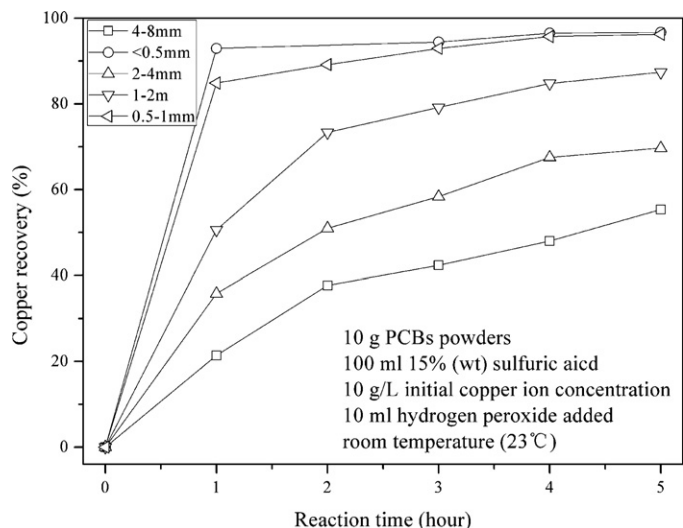


Fig. 5. Effect of sample particle size on copper recovery.

copper recovery by leaching versus reaction time with different sample particle sizes. The copper recovery increases with the decrease in particle size. The accumulated copper recoveries for samples F₁, F₂ and F₅ increase with the increase of leaching time, and copper recovery reaches 55.35%, 69.69% and 87.38% after 5 h, respectively. There is no difference on leaching recovery between sample F₆ and F₇. The leaching recovery for both samples reaches over 96% after 5 h.

The temperature was recorded by thermometer when leached samples with different particle sizes. The curve of temperature versus time is shown as Fig. 6. The results presented in Fig. 6 indicate that the leaching is an exothermic reaction. Wu et al. [22] carried out experiment of thermal decomposition of hydrogen per-

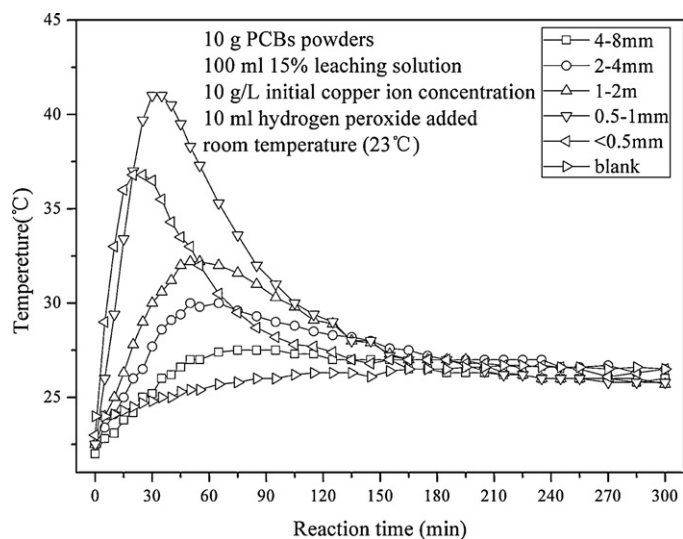


Fig. 6. Temperature changes versus reaction time using specimen with different particle size for copper leaching.

oxide in the presence of sulfuric acid. The T_0 of hydrogen peroxide (20%) in 1 N sulfuric acid is about 100 °C corresponding value with pure water about 67 °C. Although the max temperature occurs at 42 °C in our experiment as shown in Fig. 6, it is possible that the max temperature exceed 67 °C at a situation of more reactant. Hydrogen peroxide is more stable in diluted sulfuric acid, which makes the use of hydrogen peroxide more effective. The temperature change is evident during the leaching experiments. The maximum temperatures are observed at 140 min, 65 min, 52.5 min, 32.5 min, and 22.5 min for the samples of F₁, F₂, F₅, F₆ and F₇, respectively. Copper exists in the middle layer of the PCBs. The small particle size is, the more copper exposes in leaching solution, and the less mass transfer resistance in the middle layer for a certain amount of PCBs samples, the quicker the reaction occurs, and the more energy releases. The results are consistent with those shown in Fig. 5.

3.3. A reasonable shredding processes

A reasonable shredding process should be considered since it can extract copper efficiently when the particle size of waste PCBs is smaller than 1 mm.

422.8 sample of about 3 cm × 3 cm pieces of waste PCBs was shredded with cutting mill by 8 mm final fineness. The crushed sample was separated into different fractions by sieve shaker and the weight of each fraction was measured. Then all the sections were collected and shredded by 4 mm final fineness. Crushed sample was separated and the weight of each section was measured. The sample was shredded by 1 mm and 0.5 mm final fineness at the same way step by step. The accumulated percentage (wt.%) of different section shredded by different final fineness is presented in Table 3.

It is showed in Table 3 that there is a certain amount of particles which size is smaller than 1 mm even when shred with 8 mm final fineness. The section smaller than 1 mm in particle size is collected and this fraction does not need to freeze and shred anymore. Therefore, a reasonable shredding process is proposed as Fig. 7. About 450 g pieces of waste PCBs were shredded follow the process as shown in Fig. 7 and 84.67 (wt%) of PCBs pieces were shredded smaller than 1 mm finally.

3.4. Experiment of mother liquor cycles

Table 4 shows the condition of crystal liquor reused for copper leaching. Concentrated sulfuric acid was added into the solution to compensate its consumption when the crystal liquor was reused for copper leaching. Table 4 also presents the leaching recovery versus mother liquor cycles. Leaching recovery of copper reaches 96% for the 5 cycles. Reusing of the raffinate produced from the crystallization appears to be no effect on copper recovery by leaching. It should be point out that addition of concentrated sulfuric acid for the cycle 1 was not enough which may limit the leaching recovery of copper.

The product from crystallization of leaching pregnant was examined with XRD technique. The XRD pattern of the product is showed in Fig. 8, which is a pure chalcantite pattern according to the standard card (72-2355).

Table 4
Leaching condition of crystal liquor reuse.

Cycle	0	1	2	3	4	5
Concentration of H ₂ SO ₄ (mol/L)	1.63	–	0.37	0.80	1.01	1.19
Concentrated H ₂ SO ₄ added (mL)	0	2	6	6	6	1
Initial concentration of copper ion (g/L)	0	4.26	11.13	9.80	7.72	12.94
Copper recovery (%)	96.02	91.13	97.04	96.69	96.78	97.52

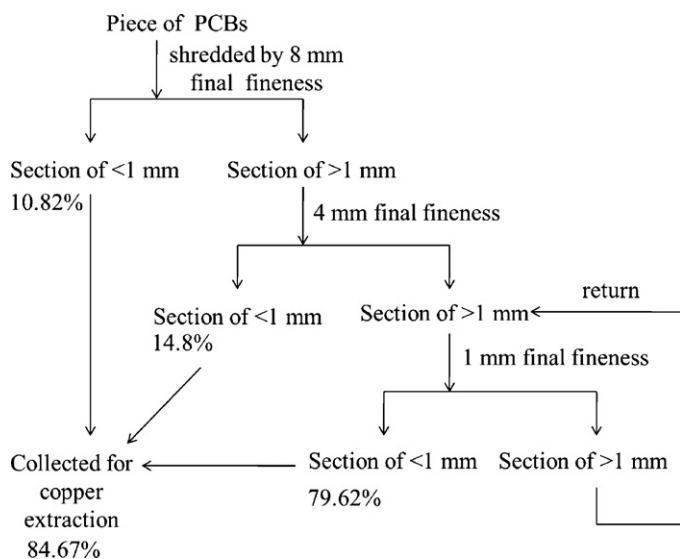
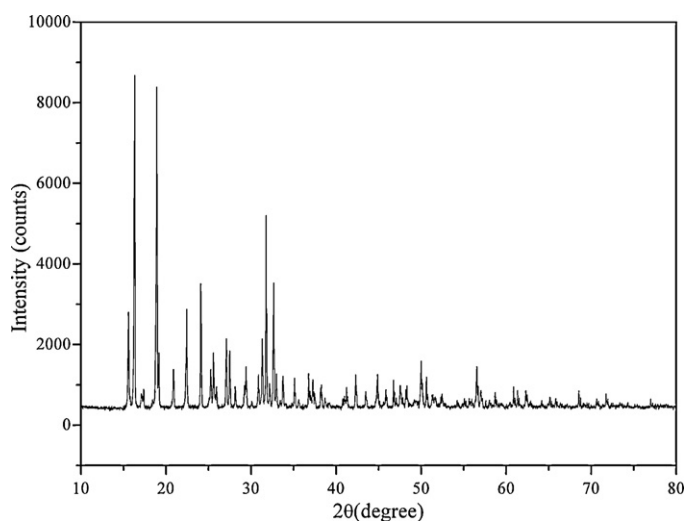


Fig. 7. A shredding process proposed.

Fig. 8. The XRD pattern of CuSO₄·5H₂O products.

4. Conclusions

- (1) The copper in powder of waste PCBs was recovered by leaching process in sulfuric acid using hydrogen peroxide as an oxidant. The optimum leaching condition for 10 g of waste PCBs at room temperature (~23 °C) is proposed as following: 100 mL of 15 (wt%) sulfuric acid, 10 mL hydrogen peroxide addition, solid–liquid ratio of 1/10, initial concentration of copper ion 10 g/L and a leaching time of 3 h.
- (2) Copper was extracted efficiently by the leaching process when the pieces of waste PCBs were shredded smaller than 1 mm. When particle sizes of waste PCBs decrease smaller than 0.5 mm it did not increase the copper recovery by leaching. How-

ever, grinding of the PCBs smaller than 0.5 mm significantly increased the energy consumption. Therefore, a suitable shredding pretreatment process was proposed.

- (3) The leaching solution was concentrated to produce CuSO₄·5H₂O. The mother liquor from crystallization was recycled to leach copper from waste PCBs powders. The leaching recovery reached 96% for 5 cycles.

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