



Bioleaching of metal concentrates of waste printed circuit boards by mixed culture of acidophilic bacteria

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ABSTRACT

Metal concentrates of printed circuit boards (PCBs) are the residue valuable metals from which non-metallic components are removed. The non-metallic components show bacterial toxicity in bioleaching process and can be recycled as well. In this study, the effects of initial pH, initial Fe(II) concentration, metal concentrate dosage, particle size, and inoculation quantity on the bioleaching were investigated so as to determine the optimum conditions and evaluate the feasibility of bioleaching of metal concentrates of PCBs by mixed culture of acidophilic bacteria (MCAB). The results showed that the initial pH and Fe(II) concentration played an important role in copper extraction and precipitate formation. Under the optimized conditions of initial pH 2.00, 12 g/L initial Fe(II), 12 g/L metal concentrate dosage, 10% inoculation quantity, and 60–80 mesh particle size, 96.8% the copper leaching efficiency was achieved in 45 h, and aluminum and zinc 88.2% and 91.6% in 98 h, respectively. All findings demonstrated that metals could be efficiently leached from metal concentrates of waste PCBs by using the MCAB, and the leaching period was shorten from about 8 days to 45 h.

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1. Introduction

Metal concentrates are the residue valuable metals from which the non-metallic components have been removed. Metal concentrates are the main product of electronics recycling. In fact, it is the high content of metals in waste printed circuit boards (PCBs) that attracted many concerns. The recent reports revealed that environment and human health have been severely threatened by improper breaking of electronic wastes [1–4]. Therefore, it is a high important issue to properly treat and recycle waste PCBs.

Hydrometallurgical and pyrometallurgical approaches are commonly used to recycle metals from electronic wastes (e-wastes). Hydrometallurgical approach leaches metals mainly by acid washing, while pyrometallurgy uses a heat treatment, such as roasting and smelting. Mechanical processes, such as crushing, shape separation, jigging, density-based separation and electrostatic separation have also been used to separate metals and non-metallic materials [5,6]. The processes consume an amount of chemicals and energy as well. So, they are not considered to be economical ways to extract metals from waste PCBs. Moreover, chemical extraction

process results in environmental damages because of the release of toxic gas, i.e. dioxins and furans, and discharge of high volume acid wastewater [7–10]. Fortunately, biological process could be an environmental sound and economical alternative.

Bioleaching is considered to be one of the most promising technologies without too much capital investment, labor need, and energy consumption [6,11]. In recent years, electronic scraps and waste PCBs have also been considered to recover metals through bioleaching [2,12–18]. However, the electronic scraps and waste PCBs have shown bacterial toxicity in the bioleaching due to the presence of non-metallic components [12,17]. Moreover, non-metallic components could be recycled. Thus, if metal concentrates are used in bioleaching instead of scraps or PCBs powder, the bacterial toxicity of non-metallic components could be avoided and higher metal extraction efficiency would be achieved.

In this paper, mixed culture of acidophilic bacteria (MCAB) enriched from natural acid mine drainage was used as the inoculant to recover metals from metal concentrates of waste PCBs. The purposes of this study were to (1) evaluate the solubility of metals from metal concentrates of PCBs by MCAB enriched from natural acid mine drainage (AMD), (2) investigate effects of operating parameters including initial pH, initial ferrous concentration, metal concentrate dosage, particle size, and inoculation quantity on bioleaching process. It was prospected to determine the optimum conditions of bioleaching process and achieve higher metal leaching efficiency.

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Table 1
Metal content in metal concentrates of waste PCBs under different particle sizes.

Mesh	Fraction (mm)	Cu (%)	Al (%)	Pb (%)	Zn (%)	Rest (%)
<80	<0.178	64.150	3.900	10.693	3.004	18.253
80–60	0.178–0.250	83.800	2.320	2.205	4.115	7.560
60–40	0.250–0.420	74.350	13.150	0.675	4.425	7.400

2. Materials and methods

2.1. Metal concentrates

The waste PCBs were obtained from a local electronics shop in Guangzhou, China. For experimental use, the scraps of PCBs were firstly shredded using stainless steel blades and then crushed by a high-speed universal pulverizer (FW-400A) for 2 min. The PCBs powder under 40-mesh was collected and subjected to hydraulic sorting so as to remove the non-metallic components (mainly plastics) from PCB powder. The left part containing a high amount of metals (over 90%) was air-dried. The generated particles were sieved and stored separately (40–60 mesh, 60–80 mesh, and <80 mesh) for future use.

Metals content in metal concentrates was determined by Atomic absorption spectrophotometer (AA6000, Techcomp, China). The final results were given in Table 1. The major metal was found to be copper with a considerable amount of aluminum, lead and zinc as well.

2.2. Mixed culture of acidophilic bacteria

AMD collected from a local pyrite mine in Guangdong, China was used to enrich acidophilic bacteria. The mineral salts solution used for bacteria enrichment was prepared according to the Silverman and Lundgren 9K medium [19]. The solution contained (g/L): $(\text{NH}_4)_2\text{SO}_4$ 3.0, KCl 0.1, K_2HPO_4 0.5, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ 0.5, $\text{Ca}(\text{NO}_3)_2$ 0.01, and 45 of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$. The pH of medium was adjusted to 2.0 by sulfuric acid.

The MCAB was prepared as previously described [17]. 200 mL of Silverman and Lundgren 9K medium was prepared in 500 mL Erlenmeyer flasks and 40 mL AMD sample was added as the source of bacteria. The flasks were incubated in a shaking incubator at 30 °C and 120 rpm for 5–8 days when reddish brown precipitate was observed in solutions. Then, the bacterial consortium was harvested by filtrating with 0.45 μm Whatman paper and followed by centrifugation at 10,000 rpm for 20 min. It was finally suspended in distilled water and stored at 4 °C for future inoculation. The average microbial concentration was enumerated 2.2×10^7 cells/mL by using hemocytometer counting method (Olympus CX31, Japan).

2.3. Bioleaching experiments

Bioleaching experiments were conducted in 500 mL Erlenmeyer flasks containing 200 mL Silverman and Lundgren 9K medium and 20 mL as-prepared MCAB. The pH was adjusted with sulfuric acid. The metal concentrate under 80-mesh was used for the factor experiments.

In order to investigate the influence of initial pH and Fe(II) concentration, dosage of metal concentrate, particle size, and inoculation quantity on bioleaching process, a series of flasks were arranged with the initial pH at 1.25, 1.50, 1.75, 2.00 and 2.50, initial concentration of Fe^{2+} at 3 g/L, 6 g/L, 9 g/L, 12 g/L, and 15 g/L, various amounts of metal concentrate powder at 4 g/L, 8 g/L, 12 g/L and 16 g/L, particle size with <80 mesh, 60–80 mesh, and 40–60 mesh, and inoculation quantities of 5%, 10%, 20% and 30%. Cell-free and

iron-free incubations were performed under the same conditions as the control experiments. Meanwhile, standing experiments with aeration manually were conducted to explore the ability of direct bioleaching. The extraction of copper, aluminum, and zinc was then investigated under the optimum conditions obtained from above factor experiments.

All flasks were weighed at the beginning of experiments and incubated in a shaking incubator at 30 °C and 160 rpm. Each flask experiment was carried out in duplicates.

2.4. Analytical determination

The leaching solutions were periodically analyzed for pH, Fe^{2+} and Cu^{2+} concentration. Any decrease in weight due to evaporation and sampling was complemented with distilled water. During the leaching experiments, 1 mL of liquid was taken from each flask after the pH was measured by an S-3C pH meter (Shanghai Rex Instrument Factory, China). The samples used for metal determination were firstly passed through 0.22 μm nitrocellulose membrane filters to remove cells and precipitation, and then diluted to appropriate concentration range with distilled water.

The concentration of copper in each sample was determined by an Atomic Absorption Spectrophotometer (AA6000, Techcomp), and ferric and ferrous ions were measured by o-phenanthroline method at the absorption wavelength of 510 nm (UV759, SPS) according to the GB 7475-87 and Water and Wastewater Monitoring Analysis Method (Fourth Edition), respectively. Reddish brown precipitates were filtered, dried and finally analyzed by X-ray diffraction. SEM-EDAX (energy dispersive X-ray analysis) mapping was applied to judge the copper leaching by comparing the metal concentrate powder residue after removing precipitates with the original sample.

3. Results and discussion

3.1. Effect of initial Fe^{2+} concentration on bioleaching of metal concentrate of PCBs

Bio-dissolution of copper under different initial Fe(II) concentration was shown in Fig. 1. In this experiment, ferrous-free medium was added into the flasks as control, and standing experiment with aeration manually was also conducted to explore the ability of direct bioleaching. It was obvious that the leaching efficiency increased gradually with the increase of initial Fe(II) concentration (0 g/L, 6 g/L, 9 g/L and 12 g/L), but declined when the concentration raised up to 15 g/L. The results revealed that 7.4%, 43.9%, 48.6%, 92.9% and 75.1% copper were mobilized from metal concentrates to solution after 3 days when fed with above described initial Fe(II) concentration, respectively. It indicated that ferrous iron played an important role in the leaching process.

The reddish brown precipitate observed from the bottom of flasks was determined as jarosite through X-ray diffraction analysis (Fig. 2). It was found that longer incubation time more jarosite precipitate was produced, and the lixiviating efficiency of copper slightly slowed down (Fig. 1). This might be due to the fact that jarosite precipitate resulted in the decrease of Fe^{3+} concentration

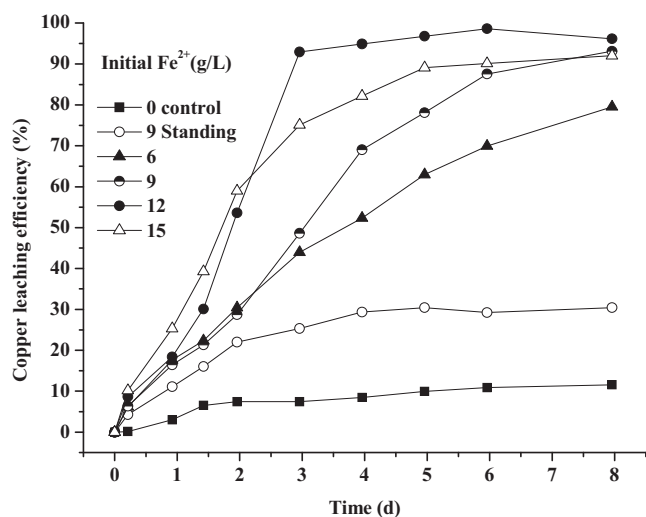


Fig. 1. Copper leaching efficiency under different initial ferric concentrations with respect to time (other conditions: 2.00 initial pH, 8 g/L metal concentrate, 10% inoculation quantity, <80 mesh metal concentrate, 30 °C, 160 rpm).

in the solution, and even formed a layer covered on the surface of metal concentrate of PCBs which caused the phenomenon of passivation. The precipitates covered on the surface of metal concentrate of PCBs adversely affected microbial leaching. It partly explained why higher Fe(II) (15 g/L) concentration showed a lower efficiency than 12 g/L Fe(II) in dissolution. Another possible reason may result from the co-precipitation of Fe(III) with the metals in the residues [13]. Further tests were conducted by measuring the concentration of copper in precipitate and metal concentrate powder residues in the case of Fe(II) (15 g/L) and Fe(II) (12 g/L). The results showed that only a few amount of copper was included in precipitate in both cases. But the metallic copper remained in metal concentrate residues under Fe(II) (15 g/L) conditions was more than that under Fe(II) (12 g/L) conditions. So the main reason why higher Fe(II) (15 g/L) concentration showed a lower efficiency than 12 g/L Fe(II) in dissolution could be concluded that jarosite precipitate caused the phenomenon of passivation. Choi found a considerable amount of copper remained in the precipitates, and the content was even higher than that in solution under same condition [13].

It was believed that the bio-oxidation of copper was achieved mainly through indirect mechanism, involving a redox intermedi-

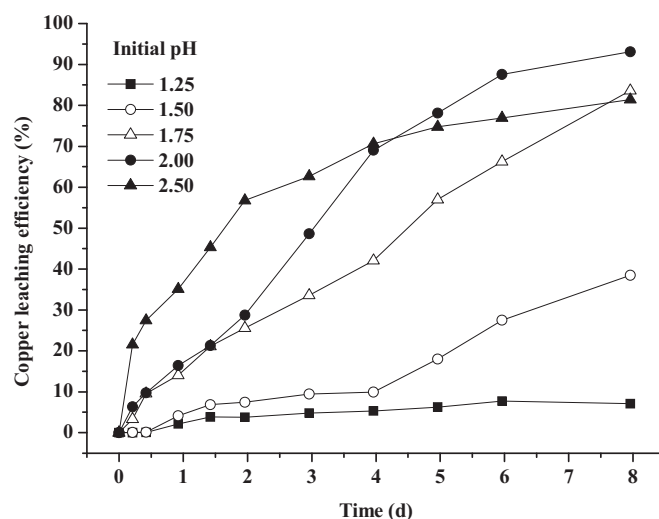
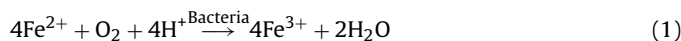


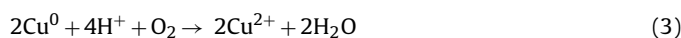
Fig. 3. Copper leaching efficiency under different initial pHs with respect to time (other conditions: 9 g/L initial Fe²⁺, 8 g/L metal concentrate, 10% inoculation quantity, <80 mesh metal concentrate, 30 °C, 160 rpm).

ate such as ferrous/ferric couple (Xiang et al., 2010), which oxidized the metals according to the following reactions:



Ferric iron in aqueous solution exhibited a standard reduction potential (E_h) as high as +0.77 V and could readily solubilize most metals in electronic scraps including copper. As predicted in Eq. (2), the recovery of copper was easily accomplished via a cementation reaction, and the regenerated ferrous ions supported the growth of bacteria as energy source and were recyclable (Eq. (1)).

It was also reasonable to presume that part of zero-valence copper was directly leached out based on the following overall reaction Eq. (3):



The mechanism was confirmed by control test with no ferrous sulphate adding into the medium. A slight increase in copper dissolution was observed since the second day, when the solution contained barely any ferric or ferrous iron to support the bioleaching of copper (Eqs. (1) and (2)). The standing experiments with aeration manually showed a inhibition of copper extraction after 4 days when lots of filamentous bacteria were found covering the surface of metal concentrate particles of waste PCBs, indicating that the indirect mechanism was the predominant one in the dissolution of copper. So, it was reasonable to conclude that microbial leaching of copper from waste PCBs was mainly accomplished indirectly through oxidation by ferric ions generated from ferrous ion oxidation bacteria. Only a small part of metallic copper was leached chemically or biologically through direct oxidation.

3.2. Effect of pH on bioleaching of metal concentrate of PCBs

Solution pH is a significant parameter in the leaching process, and usually controls bacterial growth [20]. Fig. 3 showed the effect of initial pH of medium on the dissolution of copper by bacterial leaching. The leaching efficiency of copper appeared significantly different among treatments. At initial pH 1.25, copper extraction in the solution increased slowly and was even lower than that of ferrous-free control. While at initial pH 1.50, a 4-day lag phase was observed and later a fast dissolution of copper was obtained. The extraction of copper at pH 2.00 increased steadily and reached a

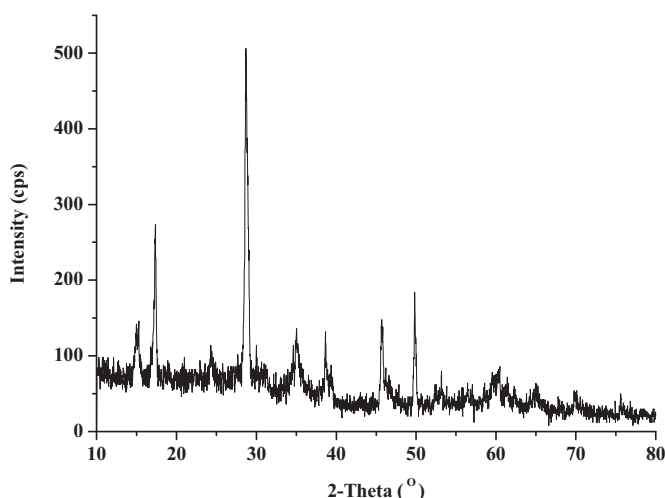


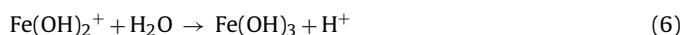
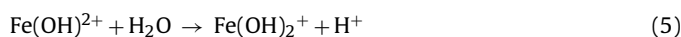
Fig. 2. XRD spectra of the precipitate.

higher efficiency (90%) after 6 days. The leaching experiments at pH 2.50 showed faster leaching efficiencies in the first 3 days, but ended with lower final leaching efficiency than that of pH 2.00 and 1.75. The results indicated that initial pH 2.00 was most efficient for bioleaching copper from metal concentrate of PCBs.

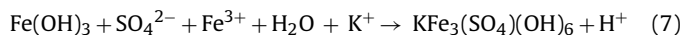
Copper extraction in the solution was slower at pH 1.25 and 1.50, mainly due to the inhibition effect of low pH on the microorganism metabolism. While at high pH 2.50, precipitate was the predominant factor resulting in lower final leaching efficiencies, because the variation of pH was closely related to the amount of precipitate being produced. In general, the variation of pH could be separated into two stages.

In the first stage, the bio-oxidation of Fe(II) would consume protons in the solution (Eq. (1)). Thus the Fe(II) oxidation was accompanied by an increase in pH.

As the quantity of Fe(III) accumulated, the hydrolysis of ferric ion occurred in the second stage (Eqs. (4)–(6)).



Compared with Eq. (1), hydrolysis of ferric iron produced protons, which decreased and tended to stabilize the pH. The above reactions suggested that the extent of the ferric iron hydrolysis was depended on pH. It was reported that ferric iron had an extremely low solubility at pH >2.50 [20]. A competing reaction for the hydrolysis was the formation of a number of jarosite complexes and all of them were known to precipitate. The main reaction Eq. (7) was as follows:



In the experiments, it was found that more jarosite precipitate was produced when pH decreased. At initial pH 2.50, there was a fast increase of pH and reached 3.48 at day 3. The fast increase of pH promoted the sedimentation of ferric iron, which better explained why the copper leaching efficiency at initial pH 2.50 was higher than others in the first 3 days but lower in the later.

3.3. Effect of metal concentrate dosage on bioleaching of PCBs

Experiments at different dosages of metal concentrate of PCBs were conducted with bacterial consortium (Fig. 4). In this study maximum leaching for copper was obtained at 4 g/L. The leaching efficiencies at 8 g/L and 12 g/L dosages were almost same since the first day. The efficiency was lowest at 16 g/L dosage. After 8 days, copper leaching efficiencies were 97.5%, 93.1%, 94.2% and 73.3% at 4 g/L, 8 g/L, 12 g/L and 16 g/L metal concentrate dosages, respectively. The data directly demonstrated that the extractions of copper decreased with the increase of metal concentrate dosage. Considering the utilization of ferric iron and disposal ability, 12 g/L dosage was more suitable for bio-dissolution of copper.

It was believed that high PCB dosage resulted in lower copper extraction mainly due to limitation in air distribution and oxygen mass transfer [13,21], thus inhibited both the efficiency and degree of the oxidation of Fe(II) by bacterial consortium. Reversely, it affected the further dissolution of copper into the solution. The toxicity of metal ions might be another important factor when powder dosage was beyond the limitation of the bacteria in solution.

3.4. Effect of particle size on bioleaching of metal concentrate of PCBs

According to the above discussions about the bioleaching mechanism, microbial leaching of copper was mainly accomplished

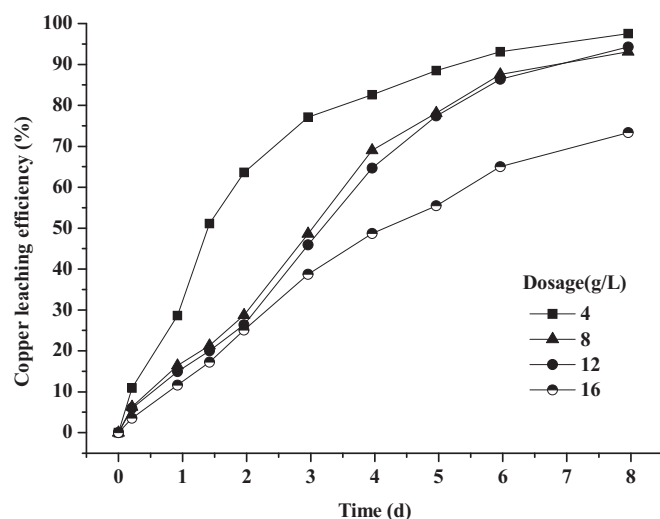


Fig. 4. Copper leaching efficiency under different metal concentrate dosages with respect to time (other conditions: 2.00 initial pH, 9 g/L initial Fe^{2+} , 10% inoculation quantity, <80 mesh metal concentrate, 30 °C, 160 rpm).

indirectly through oxidation by ferric ions. So, it was reasonable that the smaller the particles, the higher copper leaching efficiency because the contact between Fe(III) and copper was closer. But it appeared different in experiments of effect of particle size of metal concentrate of PCBs (Fig. 5). Maximum leaching efficiency for copper was obtained at 60–80 mesh metal concentrate particle size. The leaching efficiencies at meshes <80 and 40–60 were almost same and lower than that of 60–80 mesh. After 4 days, copper leaching efficiency of 60–80 mesh metal concentrate was 98.5%, however, 40–60 mesh and <80 mesh were 75.2% and 69.0%, respectively. After 8 days, they reached 98.7% and 93.1%, respectively. Thus, 60–80 mesh metal concentrate of PCBs was used in the optimum conditions experiment.

In the experiments of particle size effects in bioleaching of pyrite by acidophilic thermophile *Sulfolobus metallicus*, the particle size distribution of the mineral in the set range did not influence the morphology and growth kinetics of the cell [22]. It is considered that the enhancement of bioleaching efficiency can mainly be attributed to the effect of the particle size on the surface area of the mineral. By decreasing the particle size, the surface area per

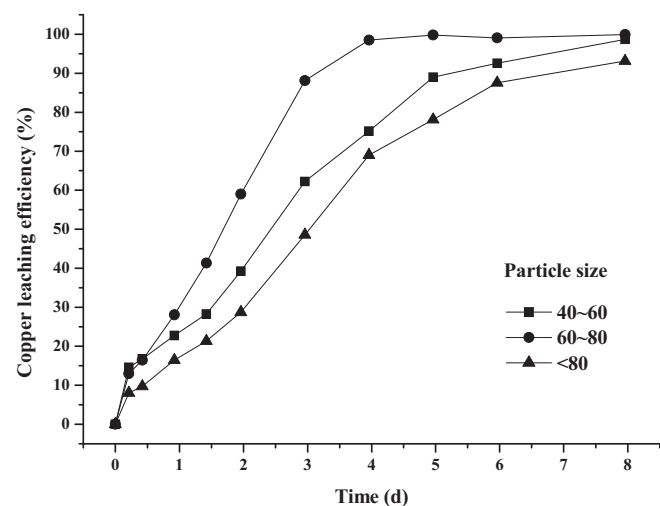


Fig. 5. Copper leaching efficiency under different particle sizes of metal concentrate with respect to time (other conditions: 2.00 initial pH, 9 g/L initial Fe^{2+} , 8 g/L metal concentrate, <80 mesh metal concentrate, 10% inoculation quantity, 30 °C, 160 rpm).

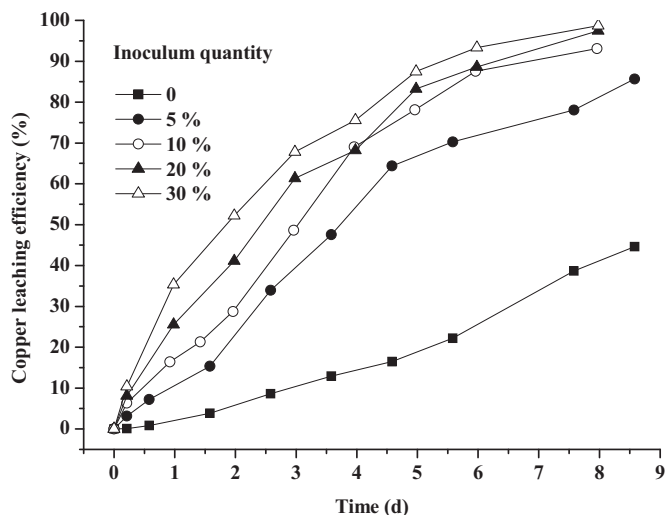


Fig. 6. Copper leaching efficiency under different MCAB inoculums with respect to time (other conditions: 2.00 initial pH, 9 g/L initial Fe^{2+} , 8 g/L metal concentrate, <80 mesh metal concentrate, 30 °C, 160 rpm).

unit mass of the mineral is increased. However, the reduction of particle size below a critical level could increase the extent of the particle–particle collision and impose severe attrition on the cells [22]. That is one reason why different particle size metal concentrates showed different bioleaching efficiencies. More important, different leaching efficiencies of different particle sizes may contribute to the toxicity of metal ions. It was reported by Sampson that copper was found to cause the largest inhibition followed by nickel and cobalt [23]. In this case, copper was not the main inhibition factor. It could be seen from Table 1 that the content of copper in 60–80 mesh was the highest (83.8%), same with its leaching efficiency. Therefore, the toxicity of the rest metals in metal concentrate might be the main inhibition factor.

3.5. Effect of inoculation quantity on bioleaching of metal concentrate of PCBs

Fig. 6 showed the influence of inoculation quantity on leaching copper from metal concentrate of waste PCBs. While the flasks were inoculated with 10%, 20%, and 30% MCAB, 93.1%, 97.5%, and 98.7% copper was mobilized into the solution after 8 days, respectively.

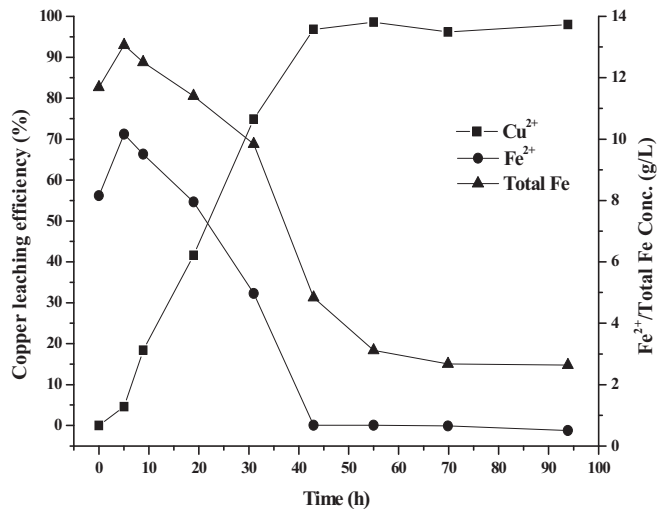


Fig. 7. Copper leaching efficiency and concentration of Fe^{2+} and total iron under the optimum conditions with respect to time (conditions: 2.00 initial pH, 12 g/L initial Fe^{2+} , 12 g/L metal concentrate, 10% inoculation quantity, 60–80 mesh metal concentrate, 30 °C, 160 rpm).

However, after 8.5 days leaching, the copper mobilization efficiency was just 85.7% with 5% inoculation quantity. Without MCAB in the medium (0% inoculation quantity), the copper leaching efficiency was observed to be just 44.6% after 8.5 days.

It could be observed that the greater the MCAB inoculation quantity the faster copper was leached (Fig. 5). It implied that the large amounts of bacteria accelerated copper leaching. It resulted from more Fe^{3+} ions could be formed by greater addition of MCAB. However, without MCAB inoculation, ferrous ion could only be oxidized to ferric ion by oxygen from air, but this process was very slow and took long time.

3.6. Two-step bioleaching of metal concentrate under optimum conditions

As previously described [17], the main role of the MCAB in bioleaching is to generate ferric ions. In this experiment, a two-step process was adopted instead of one step. In the first step, the medium was inoculated with 10% MCAB and pre-cultured without metal concentrate of waste PCBs. When 8 g/L Fe^{2+} left in the medium, metal concentrate of waste PCBs was added into the solu-

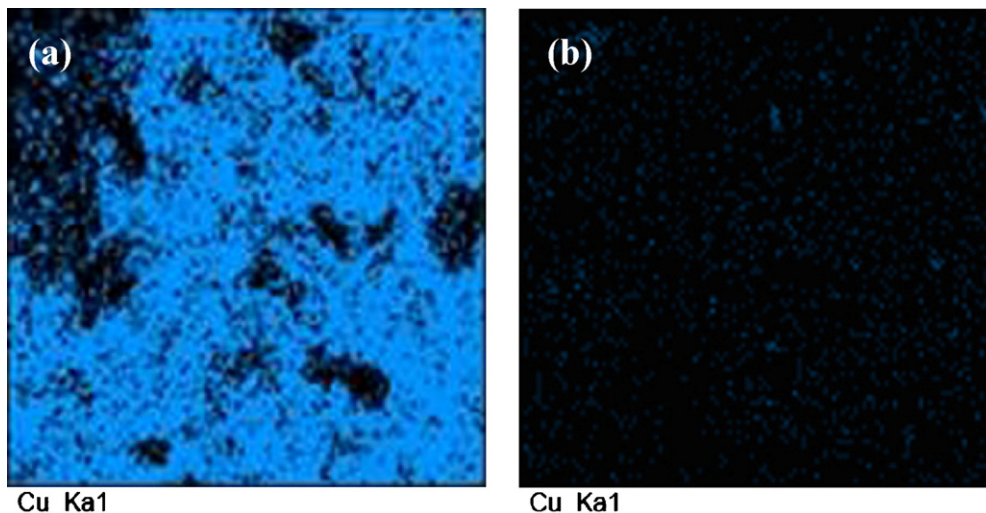


Fig. 8. X-ray energy dispersive spectrum element distribution of copper (a) raw sample, and (b) leaching residue.

tion under the optimum conditions (pH 2.00, 12 g/L initial Fe^{2+} , 12 g/L metal concentrate dosage, 10% inoculation quantity, and 60–80 mesh metal concentrate of PCBs) obtained from the above experiments as the second step. Compared with the data shown in the Figs. 1, 3–6, a two-step process could sharply shorten the leaching period, and it only took 45 h to reach the 96.8% copper leaching efficiency (Fig. 7). Aluminum and zinc both needed 98 h to get 88.2% and 91.6% leaching efficiency, respectively.

To prove the solubility of copper, EDX mapping of the leached residues was carried out and compared with the original sample (Fig. 8). The distributions of Cu have been indicated in the diagram. It was clearly that only a little distribution of copper appeared on the surface of the leached residue. It proved that most of copper has been leached into the solution during the bioleaching process.

When PCBs powder was used for the bioleaching experiments, the period was 5 days by using a two-step process under the optimum conditions [17]. The results of this experiment indicated that removal of non-metallic components indeed enhanced the bioleaching efficiency, and the period was shortened to 45 h. It also verified that the presence of the non-metallic components would like to be a limit factor in bioleaching process.

4. Conclusions

The MCAB enriched from AMD showed a good performance in bioleaching copper from metal concentrate of waste PCBs. The results indicated that microbial leaching was significantly more effective than chemical leaching. The extraction of copper was mainly accomplished indirectly through oxidation by ferric ions. Solution pH and Fe^{2+} played an important role in copper extraction and precipitate formation. The initial pH around 2.00 and Fe^{2+} concentration 12 g/L were suggested. By using a two-step bioleaching process and adding metal concentrate of waste PCBs when 8 g/L of Fe^{2+} remained in the solution, the highest extraction efficiency of copper was achieved 96.8% in 45 h under the conditions of initial pH 2.00, 12 g/L initial Fe^{2+} , 12 g/L metal concentrate dosage, 10% inoculation quantity, and 60–80 mesh metal concentrate of PCBs. Meanwhile, 88.2% aluminum and 91.6% zinc could also be mobilized in 98 h.

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