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Stirred-tank leaching of coarse-grained waste, printed circuit boards with *Acidithiobacillus ferrooxidans*

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Abstract: Stirred tank leaching of metals from coarse-grained waste, printed circuit boards (WPCB) used *Acidithiobacillus ferrooxidans* (*A. ferrooxidans*) at ambient temperature (20-35°C). The effect of the baffle size, WPCB concentration, and inoculation volume was tested. 95.92% of Cu, 93.53% of Al, 92.58% of Zn, 65.27% of Ni, and 95.33% of Sn in WPCBs were leached under the optimal conditions: no baffle, WPCB concentration of 5.0% (w/w), and inoculation volume of 5% (v/v). The alkaline substance and reactivity metal of WPCBs, and the oxidation of Fe^{2+}, consume H^+. Adding acid can maintain the pH value of the leaching solution, which is conducive to the growth and reproduction of the bacteria and improves the leaching efficiency of WPCBs. The second-order dynamics model can describe the acid consumption in the bioleaching process of coarse-grained WPCBs. Moreover, the Avrami equation can successfully explain the bioleaching kinetics of Cu, Al, Zn, Ni, and Sn from the coarse-grained WPCBs. The key factors controlling the bioleaching of coarse-grained WPCBs are metal reactivity and specific surface area. These results revealed that bioleaching metals from coarse-grained WPCBs using *A.ferrooxidans* is feasible, and has important significance to guiding its industrialization.

Keywords: stirred tank leaching, coarse-grained WPCBs, *A. ferrooxidans*, H^+ consumption, Avrami equation

1. Introduction

Waste printed circuit boards (WPCBs) are increasing drastically with the rapid innovation of electronic devices (Li et al., 2020). Based on a United Nations report, electronic waste reached 50 M tons in 2018, and the amount will reach 56.2 M tons in 2022, increasing at an efficiency from 3% to 4% per year (Qiu et al., 2020). Almost 2.3 M tons of electronic waste are produced each year in China alone (Bigum et al., 2012). WPCBs contain many valuable metals, such as Cu, Pb, Sn, Au, Ag, as well as some nonmetals. These metals are in much higher concentrations than those typically found in their corresponding ores. Electronic waste has been considered a “secondary ore” in “urban mining” (Lúcia et al., 2019). Therefore, the proper disposal and recycling of WPCBs has become an urgent global issue not only for recovering valuable materials, but also for protecting the environment.

Crushing circuit boards is the key to separate metal and nonmetal components. Generally, coarse crushing and fine crushing compose a two-step crushing process; coarse crushing is accomplished with a shear-type rotary crusher and fine crushing is accomplished with an impact-type rotary grinding machine (Yang et al., 2021). In mineral processing, comminution is the most capital-intensive unit operation and typically accounts for 30% to 50% of the total power consumption (Emmanuel et al., 2021). Fine crushing has higher energy consumption than coarse crushing. With the reduction in particle size, comminution energy consumption increases. On the other hand, in the dry crushing process, the temperature in the cavity of the crusher may reach 300-350°C, which causes the resin component in the

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circuit boards to pyrolyze and to produce harmful gases (Zhao et al., 2006). If crushing is done under wet conditions, it produces wastewater with plenty of suspended particulate matter, which is an environmental pollutant. Secondary pollution created by crushing is harmful to the environment and causes many health risks.

WPCBs are generally treated by hydrometallurgy, pyrometallurgy, and physical methods, as well as various combinations (Gu et al., 2019). These techniques can recycle and recover valuable components, but also have some obvious shortcomings in some cases (Awasthi et al., 2017). Pyrometallurgical processes are energy intensive, environmentally unfriendly, and expensive (Wang et al., 2017). The hydrometallurgical process for recovering metals from WPCBs is complicated and time-consuming, and produced liquid and solid waste (Li et al., 2018). Due to the similar properties of the metals, it is difficult for physical methods to completely separate metals (Zhu et al., 2020). Although some metals are toxic for micro-organisms, bioleaching processes are still regarded as promising due to their environmental friendliness and low cost (Yuan et al., 2018). In general, the baffle length ending of the mechanical agitation leaching tank is just above the maximum liquid level and its width equals 1/12 of its diameter. However, few studies have reported the specifications for the baffle of the agitated-leaching tank for coarse particles. The WPCB concentration, bacterial inoculation volume and leaching-solution pH value have shown a very significant influence on the bioleaching of metals. In the bioleaching process of minerals, the slurry concentration directly affects the oxidation activity of the bacteria, the leaching effect, and the formation of secondary minerals (Sasaki et al., 2011). The amount of inoculum affects the stagnation period of bacterial growth and reproduction. WPCBs are alkaline; however, *A. ferrooxidans* and other bioleaching microorganisms are suitable for acidic environments, and dilute sulfuric acid has been added to maintain a stable-solution pH environment, which has also been conducive to bacterial growth and has achieved higher metal extraction (Yang et al., 2014). To the best of our knowledge, few studies have reported the bioleaching of coarse-grained WPCBs by mesophiles in stirred tank reactors (STRs). Information concerning the baffle size will affect the hydrodynamic behavior of coarse-grained WPCBs and will provide a theoretical direction for the design of industrial-stirred tank reactors. However, the relationship between baffle size and coarse-grained WPCB bioleaching remains poorly understood.

The effect of baffle size, WPCB concentration, and inoculation volume on bioleaching coarse-grained WPCBs by mesophiles was investigated and described for the first time. We revealed H^+ consumption kinetics and metal-bioleaching kinetics in the leaching process, which is beneficial for using a bioleaching method for the extraction of metals from coarse-grained WPCBs and will further facilitate the implementation of continuous-stirred, tank-reactor (CSTR) bioleaching at an operational scale.

2. Materials and methods

2.1. Waste printed circuit boards

The WPCBs (Fig.1) were obtained from Qingdao New World Solid Waste Comprehensive Treatment Co., Ltd., in Qingdao, China. The scrap was cut into small pieces (<15 mm) before transport to the laboratory. Cu (11.4%), Al (4.7%), Zn (1.0%), Ni (0.2%), Pb (1.6%), Sn (2.1%), and Fe (1.2%) were present.

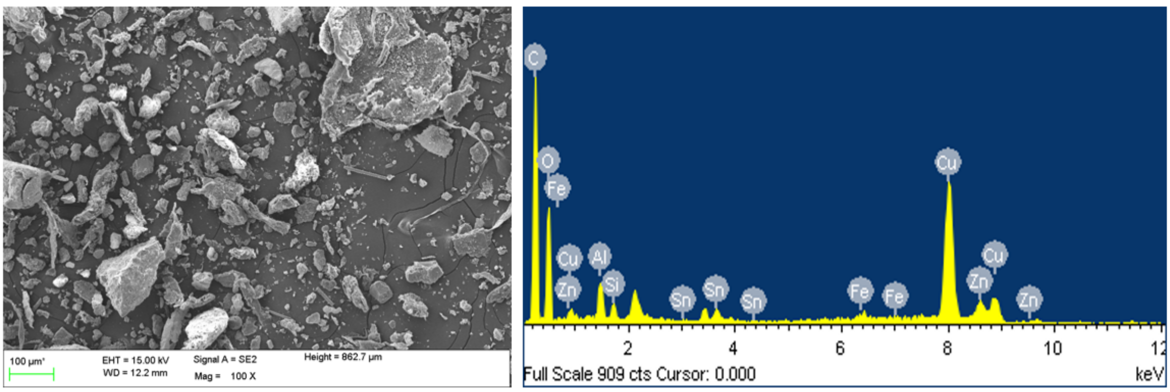

Fig. 1. SEM image and EDX analysis of WPCBs

2.2. Microorganisms and adapted cultivation

Microbes used in this study were obtained from acid mine drainage from a copper mine in the Sichuan province and were identified by 16S rDNA gene sequence (Fig.2) as *A. ferrooxidans*. *A. ferrooxidans* was initially cultured in freshly prepared and optimized 4.5K medium (Table 1) at 170 rpm and 30℃ (Fu et al., 2013). The pH of the medium was adjusted to 2.0 with H_2SO_4. The adaptation of *A. ferrooxidans* was carried out by serial sub-culturing in the late logarithmic phase at approximately 2×10^8 cells/mL, while gradually increasing the concentration of WPCBs. Finally, the cells were harvested using centrifugation, and the inoculum was prepared as discussed by Fu et al. (2012).

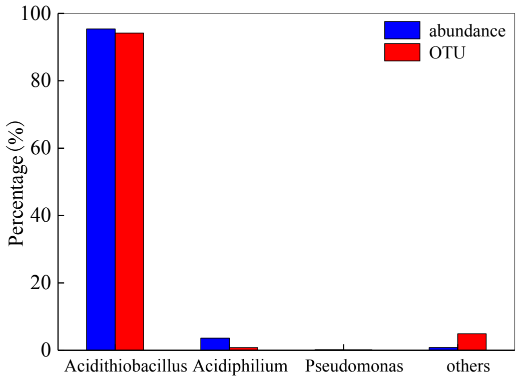

Fig. 2. Abundance and operational taxonomic units (OTUs) of the microorganisms

Table 1 Chemical composition of two kinds of cultures

Composition	4.5K medium	Iron-free 4.5K medium
$(NH_4)_2SO_4$ (g/L)	2.0	2.0
K_2HPO_4 (g/L)	0.25	0.25
$MgSO_4\cdot7H_2O$ (g/L)	0.25	0.25
KCl (g/L)	0.1	0.1
$Ca(NO_3)_2$ (g/L)	0.01	0.01
$FeSO_4\cdot7H_2O$ (g/L)	22.0	0

2.3. Bioleaching of WPCBs in a stirred tank reactor

All bioleaching experiments were carried out in a stirred tank (Fig. 3) of 165 mm height and 160 mm internal diameter. Baffles were 17mm wide and either 50 or 70mm tall. Conditions were ambient indoor temperature, 1000 rpm, and 80 d. Each reactor contained 2000 mL of iron-free 4.5K medium (Table 1). Distilled water was used to compensate for water loss due to evaporation before sampling, and the sample was supplemented with iron-free 4.5K medium to ensure a constant volume. The initial pH was maintained at 2.0 using H_2SO_4 (1.0 mol/L). All other conditions were set according to the design matrix (Table 2). After bioleaching, the residue was rinsed with distilled water, dried and analysed.

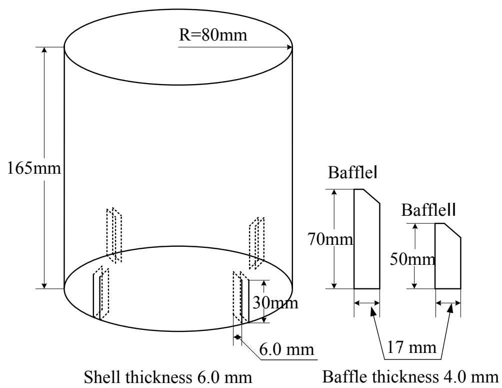

Fig. 3. Self-made stirred tank reactor

Table 2. Experimental design matrix of the test regime

Test No.	Variables	Tested Conditions	Fixed conditions
1	Baffle size	70 mm×17mm 50 mm×17mm no baffle	WPCB concentration 10%, microbial volume 5.0%, initial pH 2, 20 to 35°C, agitation speed 1000 rpm, 80 d
2	WPCB concentration	5%, 10%, 15%, 20%	Baffle size 30 mm×6mm, microbial volume 5.0%, initial pH 2, 20 to 35°C, agitation speed 1000 rpm, 80 d
3	microbial inoculation	5%, 10%, 20%, 30%	Baffle size 30 mm×6mm, WPCB concentration 5%, initial pH 2, 20 to 35°C, agitation speed 1000 rpm, 80 d

2.4. H^+ consumption for bioleaching WPCBs

The bioleaching experiments were carried out in stirred reactor without baffle (Fig. 3) at an indoor temperature, an agitation speed of 1000 rpm and a leaching period of 80 d. A total of 1900 mL of the medium and a predetermined amount of WPCBs (by weight) were mixed and added to the reactor and inoculated with a 5% inoculum (v/v). The initial pH of the solution was adjusted to 2.0 (±0.05) with a 1.0 mol/L sulfuric acid solution. High pH values have negative effects on bacterial activity (Yu et al., 2017). When the pH of the solution was greater than 3.0, it was adjusted to 2.0 (±0.05) by adding sulfuric acid solution every day to calculate the actual H^+ consumption.

2.5. Analytical methods

After digestion of the WPCBs or the residues in aqua regia, and the leachate, the concentrations of Cu^{2+}, Al^{3+}, Zn^{2+}, Ni^{2+} and Sn^{2+} were determined by inductively coupled plasma atomic emission spectrometry (ICP-OES, ThermoFisher iCAP6500, USA). Additionally, the pH value and redox potential (ORP) of the solution were measured using a digital pH meter (Mettler Toledo SevenCompact pH/ion S220, Germany). Indoor temperature was recorded using a ordinary thermometer. The number of bacteria was counted using a Helber bacteria counting chamber.

3. Results and discussion

3.1. Effect of baffle size on bioleaching

Baffle eliminates vortex, and increase turbulence and mixing. In general, the baffle extends above the maximum slurry level, and its width equals 1/12 ~ 1/10 of the diameter; 4 ~ 8 baffles are installed evenly on the inner wall of the tank. The leaching extent of Cu, Al, Zn, Ni, and Sn is shown in Fig. 4.

As shown in Fig. 4, the leaching efficiency of the metals increased gradually with increasing time, but declined with increasing baffle size. After 80 d, the large baffle (70×17 mm), medium baffle (50×17 mm) and no baffle yielded 1.7%, 28.55% and 91.93% Cu leaching. Al behaved similarly, and so did Zn, Ni and Sn. Baffle has negative effect on the metal leaching efficiency, the larger size of baffle, the lower leaching extraction of metal. The metal leached in the stirred tank without baffle was the best, the leaching efficiencies Cu, Al, Zn, Ni and Sn were 91.93%, 91.43%, 85.97%, 58.33%, and 92.03%, respectively. The particle size of the WPCBs used in the experiment was coarse, less than 15.0 mm. Coarse particle size and high density caused WPCBs to settle the bottom of the reactor, and baffles prevented the tangential movement of particles. The precipitated WPCBs could not fully contact the microorganisms, resulting in a low metal leaching efficiency. Accordingly, the following experiments were carried out in the stirred tank without baffle.

3.2. Effect of WPCB concentration on bioleaching

Fig. 5 shows that Cu, Al, Zn, Ni, and Sn leaching efficiency increased over time. It decreased with increasing WPCB concentration. After 80 d, when WPCB concentration was 5%, 10%, 15% and 20%, 95.92%, 91.93%, 56.48% and 35.62% of Cu leached. A similar trend was noted for the other metals. When

WPCBs concentration was 5%, the leaching efficiencies Cu, Al, Zn, Ni and Sn were 95.92%, 93.53%, 92.58%, 65.27%, and 95.33%, respectively. Nickel existed in nickel-containing alloy, and the effective

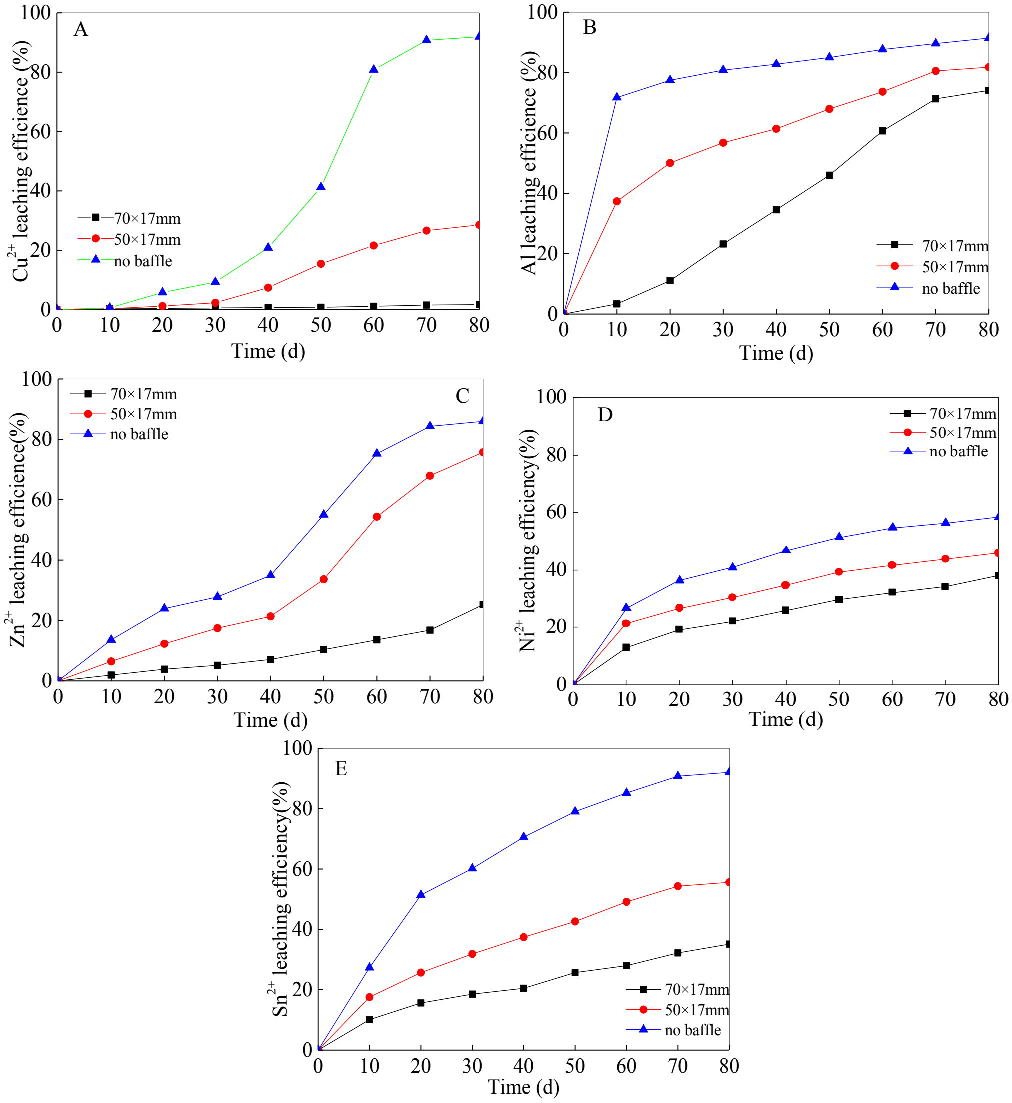

Fig. 4. Effect of baffle size on the leaching efficiency of Cu, Al, Zn, Ni, and Sn from WPCBs. (the size of the baffle was 70×7 mm, 50×7 mm and None)

contact with *A. ferrooxidans* was low; therefore, the leaching efficiency of nickel was lower than other metals (Erust et al., 2020). Therefore, 5% WPCBs was selected as the optimum concentration.

During the microbial agitation leaching process of WPCBs, the adverse effect of WPCBs concentration on bioleaching process is mainly reflected in: (1) The reaction of some metals with H_2SO_4 consumes H^+ (Fig. 6(A)), which increases the pH value of the leaching solution and produces a large amount of hydrogen at the same time, which may form a reducing atmosphere environment (Fig. 6(B)). The higher WPCBs concentration, the higher the acid consumption and hydrogen production, which is not conducive to the growth and reproduction of bacteria. (2) The higher WPCBs concentration, the higher the collision and the shear effect between particles in the flow field, which will increase the damage of bacteria; (3) WPCBs contain a large number of coarse-grained copper sheets, tiny particles, zinc particles, etc. Metal particles have poor suspension and are deposited in large quantities, forming a reducing environment and reducing oxidation reduction potential (ORP) (Fig. 6(B)), which is not conducive to the growth and reproduction of bacteria. (4) the organic particles such as plastic in the WPCBs might be caused by inhibitory effects on bacterial growth.

3.3. Effect of inoculation of bacteria on bioleaching

As shown in Fig. 7, Cu, Al, Zn, Ni, and Sn leaching efficiency decreased with increasing inoculum volume. After 80 d, when inoculum volume was 5%, the leaching efficiencies Cu, Al, Zn, Ni and Sn

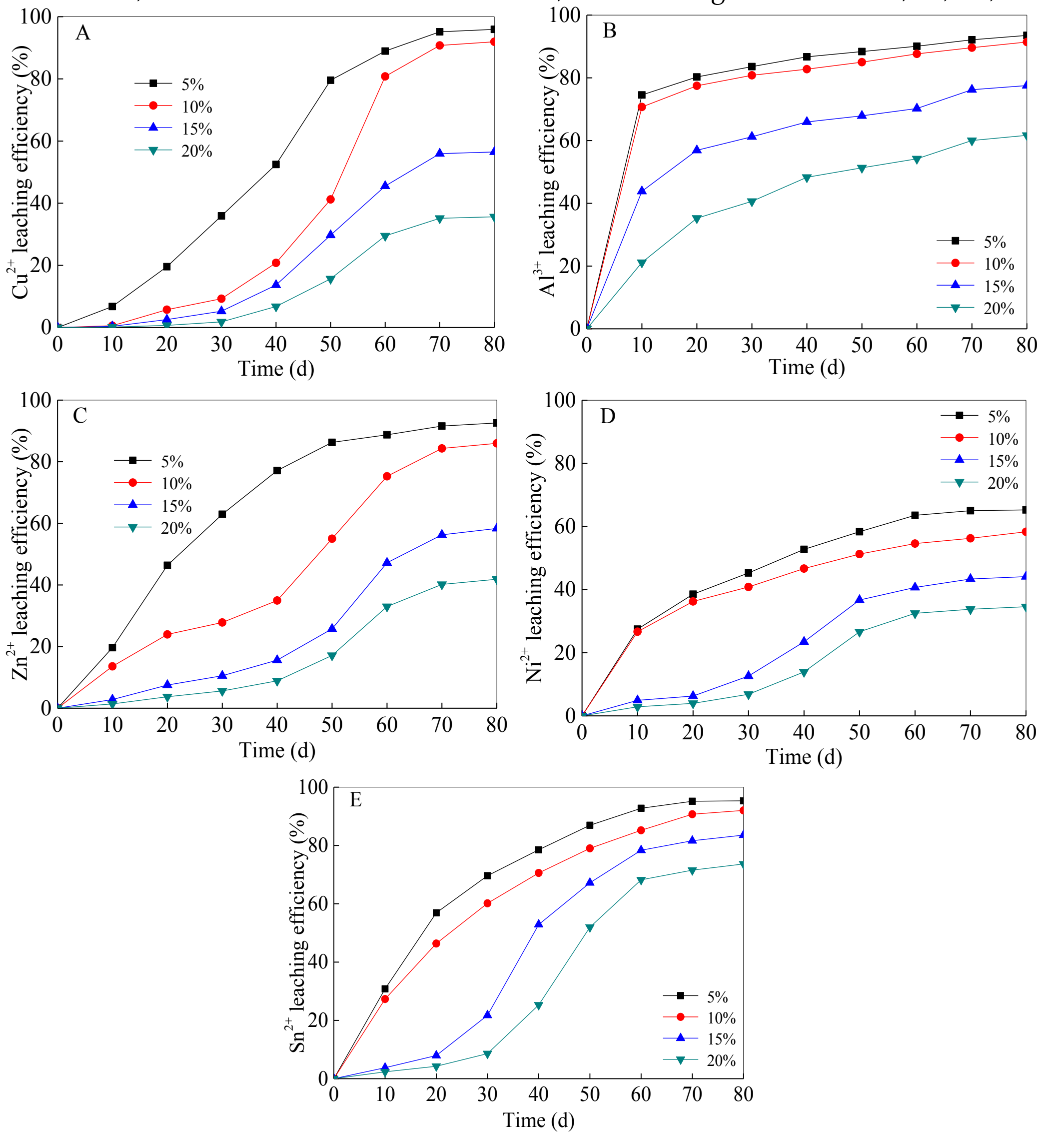

Fig. 5. Effect of WPCB concentration on Cu, Al, Zn, Ni, and Sn leaching efficiency (WPCB concentration of 5%, 10%, 15% and 20% (w/w))

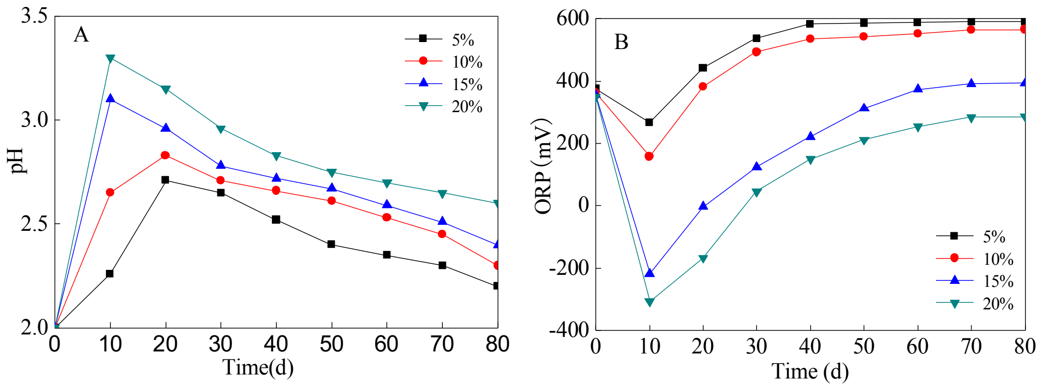

Fig. 6. pH (A) and ORP (B) profile of WPCBs concentration experiments. (WPCB concentration of 5%, 10%, 15% and 20% (w/w))

were 95.92%, 93.53%, 92.58%, 65.27%, and 95.33%, respectively. The inoculum volume influenced the leaching process of WPCBs. In the early stage (approximately 0～50 days), the higher the inoculation number of bacteria was, the higher the metal-leaching rate of the WPCBs. On the one hand, the number of bacteria on WPCB particles per unit area increased, the contact between bacteria and the active site on the surface of the WPCB particles increased, and metal erosion was accelerated. On the other hand, ferric ion has a strong ability to oxidize and etch metals, and a large amount of ferric ion in the bacterial culture medium can accelerate the dissolution rate of metals (Liang et al., 2009). In the middle-late period (50-80 d), with the growth and reproduction of bacteria and the dissolution of metals from the WPCBs, the bacterial number and the ferric-ion concentration in the system with less initial inoculation increased, while the metal-leaching efficiency in the system with more inoculation decreased due to the precipitation of jarosite and the lack of nutrients. Considering the industrial application cost, the inoculation number of bacteria was determined to be 5%.

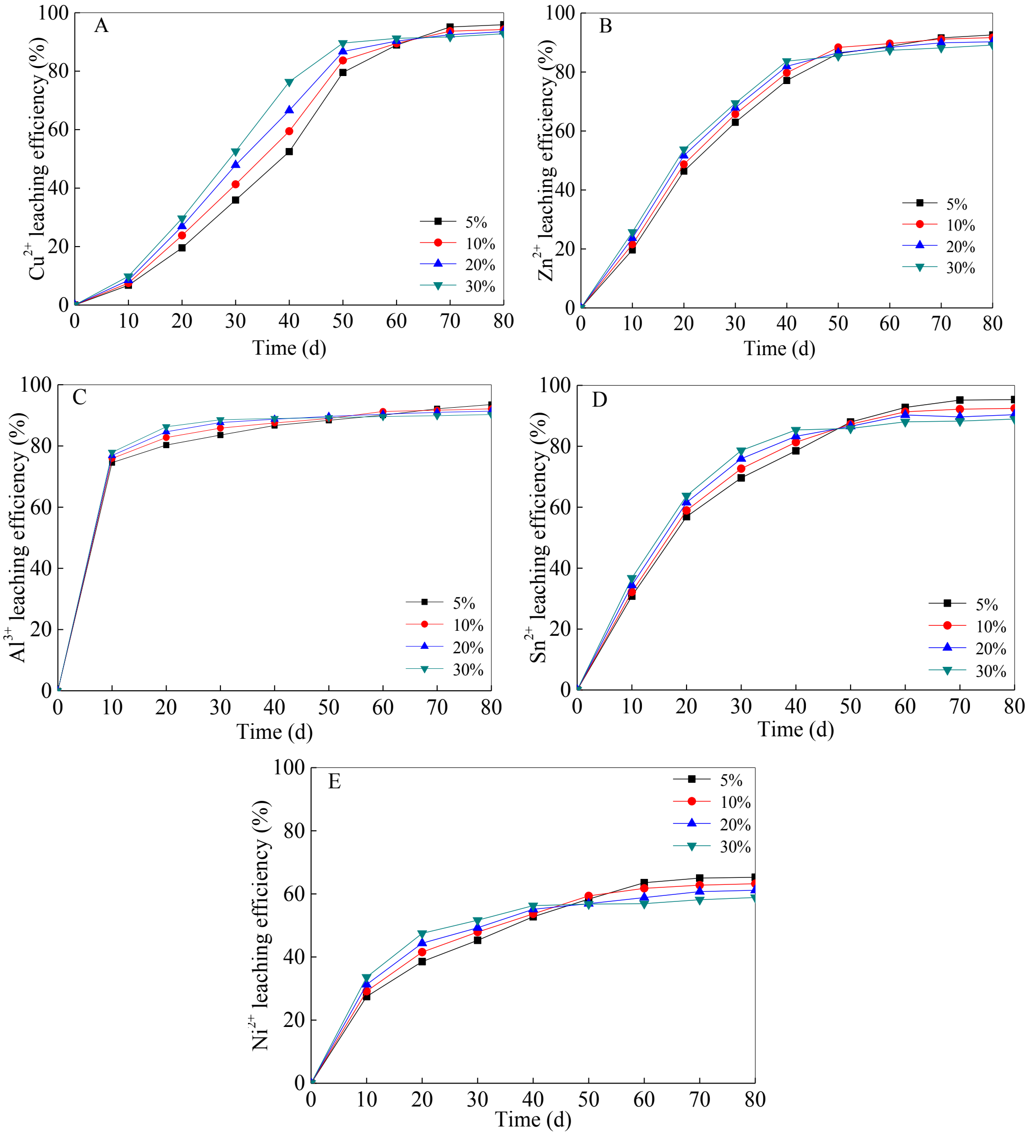

Fig. 7. Effect of inoculation on Cu, Al, Zn, Ni, and Sn leaching efficiency (inoculum volume were 5%, 10%, 15% and 30% (v/v))

3.4. H^+ consumption for bioleaching WPCBs

When the WPCBs concentration was 5%, 10%, 15% and 20%, the H^+ consumption during the bioleaching process is shown in Fig. 8.

Fig. 8(A) shows that H^+ consumption gradually increased over time, from 0 days to 45 days, and an appropriate amount of dilute sulfuric acid should be added regularly to maintain the pH value of the leaching system below 3.0. Research (Yang et al., 2019) has shown that the path of H^+ consumption by bioleaching WPCBs includes direct and indirect consumption. Direct consumption mainly refers to the interaction of an acid with substances in the WPCBs (such as alkaline substances and reactive metals such as Al), and indirect consumption consists of natural and bacterial oxidation of Fe^{2+}. The acid consumption reactions were as follows:

$$Fe^0 + 2H^+ \rightarrow Fe^{2+} + 3H_2\uparrow \tag{1}$$

$$4Fe^{2+} + 4H^+ + O_2 \rightarrow 4Fe^{3+} + 2H_2O \tag{2}$$

$$Ni^0 + 2H^+ \rightarrow Ni^{2+} + H_2\uparrow \tag{3}$$

$$Sn^0 + 2H^+ \rightarrow Sn^{2+} + H_2\uparrow \tag{4}$$

$$2Al^0 + 6H^+ \rightarrow 2Al^{3+} + 3H_2\uparrow \tag{5}$$

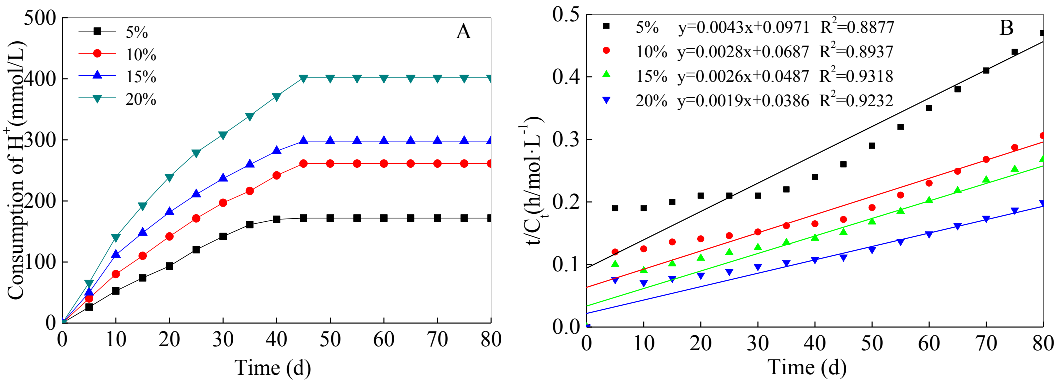

Fig. 8. H^+ consumption (A) and Second-order model (B) of leaching WPCBs. (WPCB concentration were 5%, 10%, 15% and 20%)

From the 45th day, dilute sulfuric acid was basically not added, and the acid produced by the reaction system basically maintained the solution pH value within 3.0. The acid production reactions were as follows:

$$Fe^{3+} + 3H_2O \rightarrow Fe(OH)_3 + 3H^+ \tag{6}$$

$$K^+ + 3Fe^{3+} + 2SO_4^{2-} + 6H_2O \rightarrow KFe_3(SO_4)_2(OH)_6 + 6H^+ \tag{7}$$

$$NH_4^+ + 3Fe^{3+} + 2SO_4^{2-} + 6H_2O \rightarrow (NH_4)Fe_3(SO_4)_2(OH)_6 + 6H^+ \tag{8}$$

According to the acid-consumption experimental results, when the WPCB concentrations were 5%, 10%, 15% and 20%, the acid consumption per unit mass of WPCBs was 3.44 mol/kg, 2.61 mol/kg, 2.01 mol/kg and 1.99 mol/kg, respectively. The average acid consumption was 2.51 mol/kg.

The study of H^+ consumption kinetics is conducive to more scientific prediction of the sulfuric-acid consumption and guides the industrialization of WPCB bioleaching, and was analyzed by the second-order kinetics (Eq. 9) using H^+ consumption data from Fig. 8(A) (Haghshenas et al., 2009). Fitting results are plotted in Fig. 8B.

$$\frac{t}{C_t} = \frac{1}{k_2 {C_e}^2} + \frac{t}{C_e} \tag{9}$$

In Eq. 9, t represents the leaching time; C_t represents the amount of H^+ consumption (mol/L); k_2 represents the reaction efficiency constant of the second-order kinetics; and C_e represents the total H^+ consumption in the entire process of bioleaching. Fig.8(B) shows that the second-order kinetic model may describe the dynamic consumption of H^+ during the bioleaching of coarse-grained WPCBs. It also shows the possibility of calculating the amount of acid consumption in the bioleaching process by using this model.

3.5. Kinetics of metal bioleaching

The actual production process of hydrometallurgy is often determined not by thermodynamic conditions but by the reaction speed; that is, by kinetic conditions. Kinetic analysis is of great significance to reveal the bioleaching mechanism in the metal recovery from WPCBs. The test data have usually analyzed by the shrinking core models to determine the kinetic parameters and the rate-controlling step for the leaching of metals. The shrinking core model is considered to be suitable to describe the bioleaching of metal from WPCBs by *A. ferrooxidans* (Li et al., 2015), and the model considers that the leaching process is controlled either by the diffusion of reactant through the solution boundary, through a solid product layer, or by the surface chemical reaction rate (Wei et al., 2010). The bioleaching rate may be regulated by the diffusion-controlled model (Eq. 10) due to the production of jarosite precipitates (Mishra et al., 2008). It has been reported that the semiempirical Avrami equation (Eq. 11) has been successfully used to explain the multimetal-leaching kinetic data for some cases of solid-liquid reactions (Demirkırana and Künkül, 2007).

Diffusion-controlled model: $$1-2/3\alpha-(1-\alpha)^{2/3} = kt \tag{10}$$

Avrami equation model: $$\ln[-\ln(1-\alpha)] = \ln k + n \cdot \ln t \tag{11}$$

where α represents the extent of leaching, t represents the leaching time; k represents the kinetics constant; and n represents parameters for linear fitting.

The experimental results showed that the optimal conditions for bioleaching of coarse-grained WPCBs were as follows: no baffle, initial pH value was 2.0, WPCB concentration was 5.0%, temperature was controlled to the indoor temperature, and bacterial inoculation volume was 5%. The metal extraction data under the optimal conditions are shown in Fig.9. The kinetics fitted by the diffusion-controlled model (Eq. 10) and the Avrami equation (Eq. 11) are shown in Fig. 10.

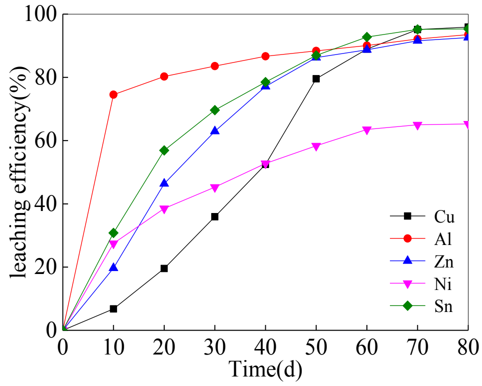

Fig. 9. Cu, Al, Zn, Ni, and Sn leaching efficiency under the optimal conditions

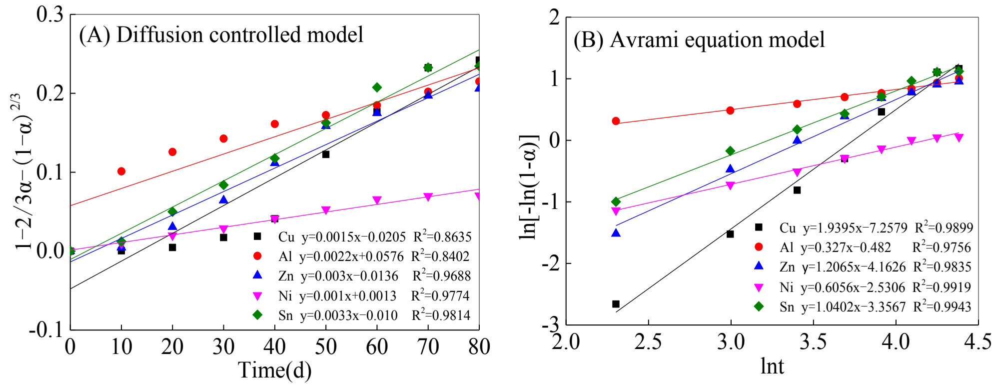

Fig. 10. Bioleaching kinetics of Cu, Al, Zn, Ni, and Sn from WPCBs

As shown in Fig. 10, the semiempirical Avrami equation model of Cu, Al, Zn, Ni, and Sn from WPCBs approached linearity more closely than the diffusion-controlled model, which indicated that the Avrami equation could successfully explain the bioleaching kinetics of Cu, Al, Zn, Ni, and Sn from coarse-grained WPCBs. The Avrami equation assumes that dissolution is surface controlled with random initiation of dissolution sites (Cornell and Giovanoli, 1993). The n in Eq. 11 may indicate the nature of the controlling step. The value of n is calculated to be 0.327 for Al and 0.605 for Ni, and both of them are less than one, which indicates that the initial leaching rate is infinite and that the rate continually decreases with time. At the beginning of leaching, the bacteria are at a lag phase, this indicates that Al and Ni may be leached more easily than Cu, Zn and Sn by acid leaching. The n value of Sn was 1.0402 and close to 1.0, which indicates that the initial leaching rate of Sn is finite. The n value of Cu, and Zn were 1.9395, and,1.2065, respectively, and both were greater than one, the initial bioleaching rate of Cu and Zn approached zero. Therefore, the key factors controlling the bioleaching of coarse-grained WPCBs are metal reactivity and specific surface area.

Fig. 11 shows that a WPCBs residue was covered with a layer of crystallized precipitate, and suggests that this was potassium jarosite (Eq. 7) or ammonium jarosite (Eq. 8). This was further evidence that the metal bioleaching rate of coarse-grained WPCBs was not regulated by the diffusion-controlled model.

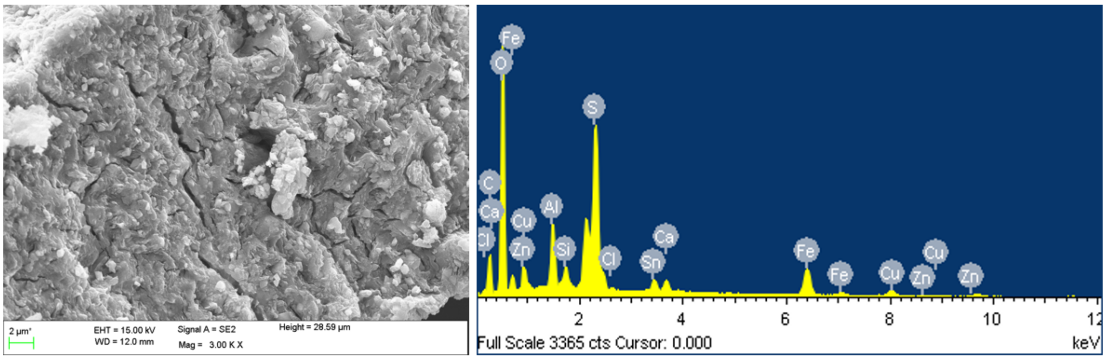

Fig. 11. SEM image and EDX analysis of WPCBs residues

4. Conclusions

In this study, we successfully demonstrated that biological agitation leaching of coarse-grained WPCBs by *A. ferrooxidans* at room temperature (20～35°C) was feasible. The experimental results showed that the optimal conditions for bioleaching of coarse-grained WPCBs were as follows: no baffle, initial pH value was 2.0, WPCB concentration was 5.0%, temperature was controlled to the indoor temperature, and bacterial inoculation volume was 5%. The alkaline substance and reactivity metal of the WPCBs and the oxidation of Fe^{2+} consumed H^+. Therefore, adding acid can maintain the pH value of the leaching solution, which is conducive to the growth and reproduction of the bacteria and improves the leaching efficiency of the WPCBs. The second-order dynamics model can describe the acid consumption in the bioleaching process of coarse-grained WPCBs. Moreover, the Avrami equation could successfully explain the bioleaching kinetics of Cu, Al, Zn, Ni, and Sn from coarse-grained WPCBs. The key factors controlling the bioleaching of coarse-grained WPCBs are metal reactivity and specific surface area. These results revealed that bioleaching metals from coarse-grained WPCBs using *A.ferrooxidans* has potential for commercial exploitation.

Acknowledgments

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