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The ultrasonically assisted metals recovery treatment of printed circuit board waste sludge by leaching separation☆

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ABSTRACT

This paper provides a practical technique that realized industrial scale copper and iron separation from printed circuit board (PCB) waste sludge by ultrasonically assisted acid leaching in a low cost, low energy consumption and zero discharge of wastes manner. The separation efficiencies of copper and iron from acid leaching with assistance of ultrasound were compared with the one without assistance of ultrasound and the effects of the leaching procedure, pH value, and ultrasonic strength have been investigated in the paper. With the appropriate leaching procedure, a final pH of 3.0, an ultrasonic generator power of 160 W (in 1 l tank), leaching time of 60 min, leaching efficiencies of copper and iron had reached 97.83% and 1.23%, respectively. Therefore the separation of copper and iron in PCB waste sludge was virtually achieved. The lab results had been successfully applied to the industrial scaled applications in a heavy metal recovery plant in city of Huizhou, China for more than two years. It has great potentials to be used in even the broad metal recovery practices.

1. Introduction

Printed circuit board (PCB) waste sludge generated by the PCB spent rinsewater treatment process is classified as hazardous waste. The sludge mainly contains a settled mixture of copper and iron hydroxides, some basic salts and other solids as well. While toxicity of heavy metal to public health and nature environment is well known [1,2], heavy metals can also be reused as valuable resources. Metal recovery from heavy metal bearing PCB waste sludge will not only provide benefits of eliminating the pollution to environment, but also reducing consumption on scarce valuable metal resources. As the rapid development of China's PCB manufacturing capacities, the quantities of PCB waste have been increased. It is becoming more and more important in China for the metal recovery treatment of PCB sludge. The key of a valuable metal recovery process relies on a reliable separation technique that can separate different metal components from a complicated metal bearing mixture in a low cost and without producing further negative impacts to environment (second pollution). In recent years, several approaches, such as emulsion liquid membranes (ELM) [3,4], co-precipitation, adsorption, solvent extraction, cementation, electrowinning (EW) and solid-phase extraction (SPE), etc., have been investigated by various researchers for separating the metals from field test samples [5–11]. However, these separation techniques have many limits if applied to industrial scaled applications because of its unacceptable treatment costs and high equipment investment. In an effort to realize sustainable beneficial metal recovery of the waste sludge, it is necessary to find a simple, reliable and cost efficient metal separation method that can not only be used for industrial applications, but also does not create any secondary pollution.

For decades, people have noticed when imposing ultrasound to a liquid–solid interface, acoustic cavitation occurs. The low pressure cavitation bubbles begin to burst later when crashed by higher pressure of the surrounding medium in liquid [12]. As the bubbles collapse, they shoot out high speed local jet streams with velocities measured sometimes over 100 meters per second [13], and the surfaces of solid particles of the slurry are activated by the high speed jet streams [14]. Moreover, applying ultrasonic to a liquid–solid mixture agitates liquid around the surfaces of the solids, speeds up cross-phase solid–liquid diffusion transfer and intensifies chemical reaction between the liquid and solid phases. As the result, both diffusion controlled and reaction rate controlled solid–liquid acid leaching processes are greatly enhanced by ultrasonic agitation. In addition to increasing the leaching reaction rate, ultrasonic also can enhance the precipitation reaction [15,16].

Many researchers have applied ultrasound in heavy metal leaching process to increase extraction rate of metal. Swamy and Narayana reported the rapidly extract metals using ultrasound [17]. Bese introduced the dissolution of copper from copper converter slag by acid leaching with ultrasound [18]. Kim et al. investigated the enhanced leaching of chromium from radioactive sludge [19].

☆ Patent-pending technique.

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Gil et al. reported the removal of mercury from contaminated water by using ultrasound [20]. There are also many reports about leaching and recovering single noble metals such as silver or manganese assisted by ultrasound [21,22]. However, these researches only investigate the ultrasonic enhancement effect on the leaching reaction rate of metal from its bearing sludge. They cannot enhance the separation of different metals from a multiple metals mixed sludge by using ultrasound, which is very important for the metal recovery from heavy metal waste sludge.

Only a few studies have analyzed the ultrasonically assisted separation of the different metals from waste sludge. Godinez investigated the ultrasonically assisted selective leaching of zinc over iron from electric arc furnace dust [23,24]. But his zinc recovery efficiency only achieved 80% when the iron was separated from zinc leachate by ultrasonically assisted selective leaching. In order to achieve separation of different metals in a multiple metals bearing sludge and realize metal recovery for beneficial utilization with minimum secondary environment pollution, two questions need to be answered. First, how to separate different metals within sludge; second, how to economically extract all metal components inside sludge (with high recovery efficiency).

This paper intends to provide a method to address these two questions and investigates an ultrasonically assisted approach for the separation of copper and iron from PCB waste sludge. In this work, a novel and practical technique that efficiently separates copper and iron with minimum cost and virtually zero waste discharge has presented. With assistance of ultrasound and appropriate leaching procedure, the study realized the total separation and high recovery of different metals from PCB waste sludge via acid selective leaching. It completely improves traditional acid leaching process and supplies a cost efficient method for heavy metal recovery from a multiple metal bearing sludge. This technique has been successfully applied to the industrial scaled applications in a heavy metal recovery plant in Huizhou city, Guangdong province, China for more than two years. To our knowledge, this work presents the first application of ultrasound for the separation of copper and iron from the waste sludges. Further investigations for applying the ultrasonically assisted acid leaching to separation of nickel, zinc, copper, chromium and iron in plating waste sludge have been performed and will be discussed in a coming paper.

2. Materials and methods

2.1. Experimental setup and materials

The ultrasonically assisted leaching experiments were performed in 1 l titanium tank with three ultrasonic transducers attached on the external wall of the tank. All transducers are connected to one ultrasonic generator which generates ultrasonic waves with a frequency of 20 kHz under powers of 80, 160, and 240 W. (The apparatus was manufactured by Guangzhou KEPU Ultrasonic Electric Technological Ltd., China.) A stirrer impeller and a glass heat exchanger were hanged inside the tank. The temperature inside the tank was maintained within ±2 °C around an expected temperature via a water circulating conduit of the glass heat exchanger. A schematic diagram of the experimental setup is shown in Fig. 1.

The PCB waste sludge tested in the study was supplied by Dingfu Electrical (Huizhou) Co. Ltd. The sludge appeared brownish, and its pH was 5.6 (measured by pH-meter), moisture content was 75.3% (measured by Infrared Moisture Meter). The main metals composition of the sludge was analyzed to be (wet content) 4.85% Cu and 4.23% Fe (analyzed by Atomic Absorption Spectrometry). The other metals wet content in the sludge was lower than 0.1% and was neglected. After stirring the sludge for 30 min at a speed of

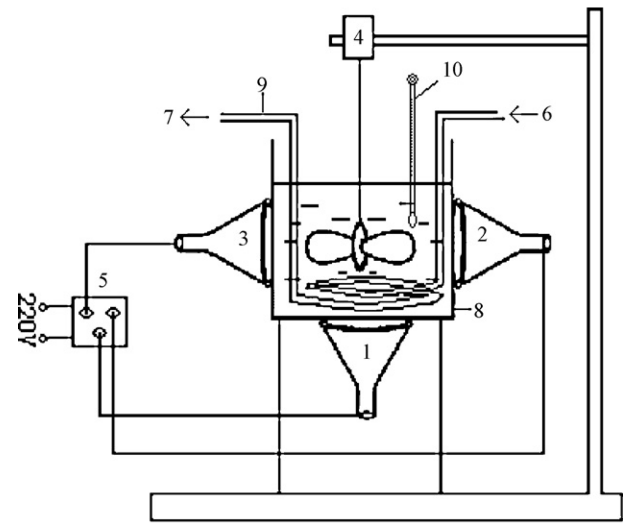

Fig. 1. The ultrasonically assisted leaching apparatus.

900 rpm under the condition of its water content up to 90% and filtering it through a 2-mm sieve, the mean particle size of the filterable sludge was measured to be 165 μm (analyzed by Scanning Electron Microscopy).

All other chemicals used were of either reagent grade or better.

2.2. The ultrasonically assisted leaching tests

In order to test the effect of leaching procedures on the separation efficiency, two leaching procedures were employed. The detail of the procedures is as follows:

2.2.1. Leaching procedure 1

Part 1 sludge preparation: 100 g sludge was transferred into a 500 ml beaker and mixed with proper amount of H_2SO_4 (98%), then stirred for 10 min as its pH value reached to 1.5 and almost all the sludge was dissolved. Next added 10 ml H_2O_2 (30%) into the beaker, then poured the acid dissolved mixture contained within the beaker into the titanium tank and retained at a pre-set temperature. The prepared acid dissolved mixture was referred as part 1.

Part 2 sludge preparation: 200 g sludge was transferred into a 1000 ml beaker, added water to dilute the sludge till its water content up to 90%, then added 20 ml H_2O_2 (30%) and stirred it for 30 min at a speed of 900 rpm. All mixture contained in the beaker was then poured on a 2-mm sieve to take out coarse residues, and the filterable sludge fraction was subsequently retained at a controlled temperature and referred as part 2.

Next, slowly added part 2 into part 1 in the titanium tank under constant stirring and ultrasonic application, a test without ultrasonic application was also conducted in the study as comparison. By adjusting the pouring rate of part 2 into part 1 while keeping a constant temperature inside the titanium tank, a desired pH value can be reached within a pre-determined time frame. The beginning time of the test (time zero) was set at the time when the ultrasonic system was turned on or started to add part 2 into part 1 in the tank in the test without ultrasonic application.

2.2.2. Leaching procedure 2

200 g sludge was transferred into a 1000 ml beaker, added water to increase sludge water content up to 90%. Next, added 20 ml H_2O_2

(30%) to the beaker and stirred for 30 min at a speed of 900 rpm. Then poured all the mixture contained in the beaker onto a 2-mm sieve to take out coarse residues and the filterable sludge fraction was retained in the titanium tank with a pre-set temperature. Slowly added H_2SO_4 (98%) into the sludge inside the titanium tank while keep stirring and exerting ultrasound on it. By adjusting the rate of pouring H_2SO_4 and keeping a constant temperature inside the titanium tank, a desired pH value can be reached within a predetermined time frame. The beginning time of the test (time zero) was set at the time when the ultrasonic system was turned on.

The reactions in both the procedures are:

$$2Fe^{2+} + H_2O_2 + 2H^+ = 2Fe^{3+} + 2H_2O \quad (1)$$

$$2Fe(OH)_2 + H_2O_2 = 2Fe(OH)_3 \quad (2)$$

$$Cu(OH)_2 + 2H^+ = Cu^{2+} + 2H_2O \quad (3)$$

$$Fe(OH)_3 + 3H^+ = Fe^{3+} + 3H_2O \quad (4)$$

$$Fe^{3+} + 3Cu(OH)_2 = 2Fe(OH)_3 + 3Cu^{2+} \quad (5)$$

2.3. Analytical methods

The pH value was measured by a calibrated PHB-3 pH-meter (Shanghai San-Xin Instrumentation, Inc.) within appropriate time intervals.

As pH of the sludge mixture inside the titanium tank reached the pre-set desired value, the mixture was then filtered to separate into solid and liquid phases. The solid phase was then rinsed by water three times (200 ml each), and all rinsing water was collected and mixed with the liquid phases generated from the filtration. The weight of the rinsed solid phases and the total volume of liquid phases of mixed filtrate and rinsing water were measured, and indicated as M_S, V_L, respectively. Copper and iron contents of both solid and liquid phases were determined by an Atomic Absorption Spectrometry (Hitachi Ltd., Japan, Z-5000 Series), and indicated as $W_{Cu,S}$, $W_{Fe,S}$, $C_{Cu,L}$, $C_{Fe,L}$, respectively. As the result, the leaching efficiency is defined as following:

$$\text{Copper leaching efficiency}\,(\%) = \frac{C_{Cu,L}\,(g/l) \times V_L\,(l)}{C_{Cu,L}\,(g/l) \times V_L\,(l) + W_{Cu,S}\,(g/kg) \times M_S\,(g)/1000}$$

$$\text{Iron leaching efficiency}\,(\%) = \frac{C_{Fe,L}\,(g/l) \times V_L\,(l)}{C_{Fe,L}\,(g/l) \times V_L\,(l) + W_{Fe,S}\,(g/kg) \times M_S\,(g)/1000}$$

3. Results and discussion

3.1. The effect of ultrasound on leaching efficiency

In order to investigate the impact of ultrasound on leaching efficiency, lab tests were performed by the leaching procedure 1 under the following experimental conditions:

(i) Ultrasonic power of 160 W, temperature at 25 °C, stirring speed at 600 rpm, and ending pH of 3.0 were set during entire test period.
(ii) Without ultrasound, temperature at 80 °C, stirring speed at 600 rpm, and ending pH of 3.0 were set during entire test period.
(iii) Without ultrasound, temperature at 25 °C, stirring speed at 600 rpm, and ending pH of 3.0 were set during entire test period.

And the test results were presented in Figs. 2 and 3.

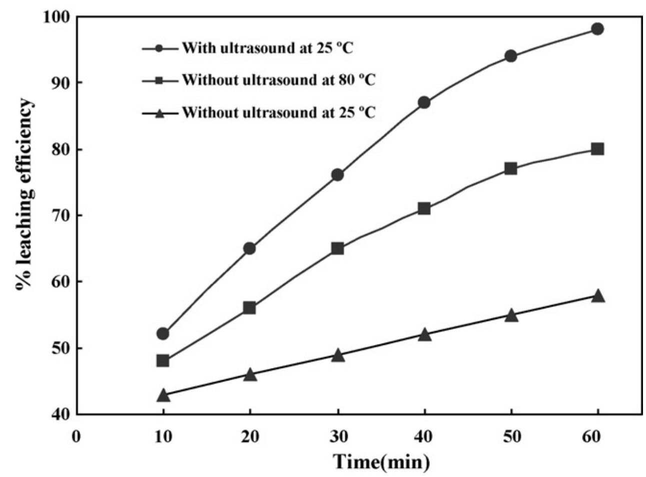

Fig. 2. The copper leaching efficiency varies with sonication time under different conditions.

As shown in Figs. 2 and 3, the copper leaching efficiencies were higher and iron leaching efficiencies were lower with ultrasound at 25 °C than the ones without ultrasound at 80 and at 25 °C. It showed that the ultrasonically assisted acid leaching, even with lower temperature, has higher separation efficiency between copper and iron than conventional acid leaching that does not use ultrasound. The solubility product constants of $Cu(OH)_2$ and $Fe(OH)_3$ are 2.2×10^{-20} and 4.0×10^{-38}, respectively. In theory copper and iron can be separated easily based upon their solubilities via pH adjustment. However, under real situation separation by either dissolution or precipitation via leaching process is difficult to be realized. Not only the ion movement from the surface of $Cu(OH)_2$ particles into liquid are limited by solid–liquid phase diffusion transfer process, but some parts of the surface of $Cu(OH)_2$ particles are also covered by insoluble metal components like $Fe(OH)_3$. So it is difficult for the $Cu(OH)_2$ particles contained within sludge to be fully dissolved into its leaching solution at appropriate pH value without over dosing acid liquid. Furthermore, $Fe(OH)_3$ always intend to forms colloidal particles that keep suspension and do not settle or separate from the liquid phase easily. Therefore, copper and iron in sludge are not easy to be separated in the real industrial practice.

As prior mentioned, ultrasonic wave induced liquid cavitations that can blow out the surface of solids, which generates highly reac-

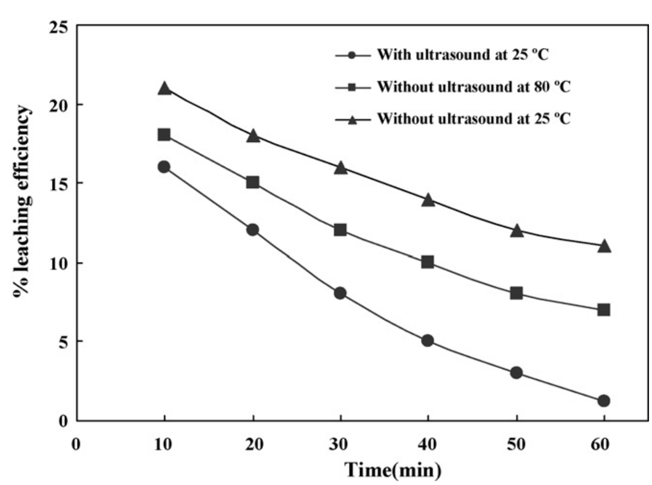

Fig. 3. The iron leaching efficiency varies with sonication time under different conditions.

tive surfaces, causes short-lived high temperatures and pressures at the surface, produces surface defects and deformations, forms fines and increases the surface area of friable solid supports, ejects material into solution [19], thus increasing ion movement from surface of $Cu(OH)_2$ into liquid phase and enhancing the leaching of copper into the leachate from the sludge. At the same time, ultrasonic wave can also enhance the settlement of $Fe(OH)_3$ which is presented by Fig. 3. Although the mechanism of this phenomenon is not very clear yet, it is already in good agreement with other experiments [23]. So the ultrasound dramatically raises the copper leaching efficiency, reduces the iron leaching efficiency and increases the separation efficiency of copper and iron from the PCB sludge.

3.2. The effect of leaching procedure

The leaching efficiencies of leaching procedure 1 and leaching procedure 2 were presented in Table 1 with all other parameters keeping the same for both procedures. The test times were all controlled at 60 min, ultrasonic powers were all selected at 160 W, the ultimate pH values were all set at 3.0, the temperatures were all controlled at 25 °C, and the stirring speeds were all 600 rpm. The tests of both procedures were repeated three times and the averaged leaching efficiencies were listed in Table 1.

By comparing the leaching efficiencies between two procedures in Table 1, it shows that the leaching procedure 1 has much better separation between copper and iron than the leaching procedure 2. In the leaching procedure 1 97.83% of copper and 1.23% of iron contained inside the sludge were dissolved into leaching solution, while in the leaching procedure 2 88.04% of copper and 10.16% of iron contained inside the sludge were dissolved into leaching solution. The leaching procedure 1 almost extracted all copper contained by the sludge with minimum iron, it virtually achieved copper and iron separation from PCB waste sludge. Leaching procedure 1 has a superior performance since pH value of its mixture was raised from 1.5 to 3.0 with pH lower than 3.0 most of the time in the leaching period. As results the leaching reaction of copper in the leaching procedure 1 proceeded more completely than leaching procedure 2 in which the pH of its mixture was from 5.6 down to 3.0 with pH higher than 3.0 most of the time in the leaching period, thus the copper leaching efficiency of the former is higher than the latter. Moreover, leaching procedure 1 in which the pre-prepared sludge with pH around 5.6 was slowly added into pre-prepared acid dissolved sludge solution with pH of 1.5 creates a smooth increased pH process. This enhances crystal heterogeneous nucleation, produces big crystal particles instead of colloidal particles, which makes the precipitation reaction of ferric ion towards settlement equilibrium and achieves lower iron leaching efficiency. Leaching procedure 2 in which acid was added directly into sludge often creates local temporary high acid zone where completely dissolution of ferric hydroxide occurs. As high acid zone quickly disappears during stirring, its pH arises promptly and produces settled ferric hydroxide again. This enhances homogeneous nucleation, produces small crystal particles that easy to form colloidal particles that seldom settles and results in higher iron leaching efficiency. So the leaching procedure 1 is more preferable to separation of copper and iron in PCB sludge.

Table 1
The leaching efficiencies of the two procedures.

Type of procedure	Copper leaching efficiency (%)	Iron leaching efficiency (%)
Leaching procedure 1	97.83	1.23
Leaching procedure 2	88.04	10.16

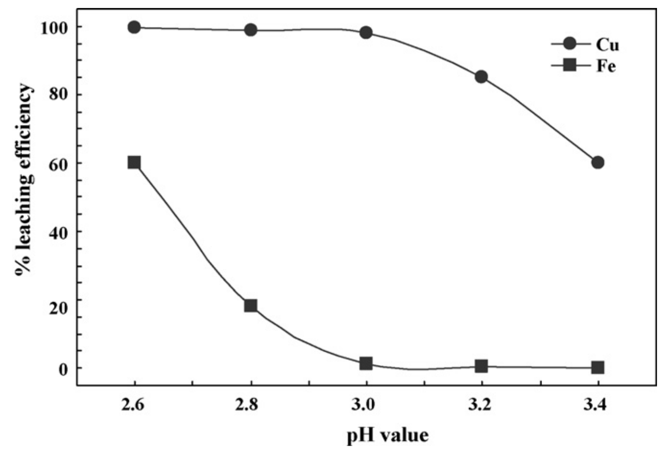

Fig. 4. The influence of pH on leaching efficiency.

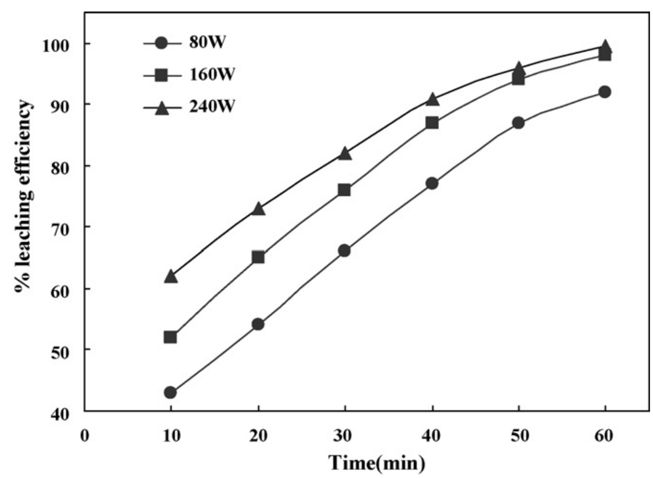

Fig. 5. The influence of ultrasound power and applying time on copper leaching efficiency.

3.3. The effect of pH value

The effects of pH value on the recovery of copper and iron from PCB sludge were shown in Fig. 4. The test time for each pre-set pH value was chosen as 60 min, the leaching procedure 1 was employed, ultrasonic power of 160 W, temperature at 25 °C, and stirring speed at 600 rpm were selected in the test.

As demonstrated in Fig. 4, both leaching efficiencies of the copper and iron increased as the pH value decreased and the leaching reactions were promoted by applied acid. At pH of 3.0, the leaching efficiencies of copper and iron measured as 97.83% and 1.23%, respectively, thus most copper has dissolved into liquid whereas most ferric particles still remain as solids. So the pH of 3.0 can be chosen as an optimal pH value during ultrasonically assisted acid leaching separation of copper and iron in PCB waste sludge.

3.4. The effect of ultrasound power and applying time

The leaching efficiencies of copper and iron among different ultrasonic powers (80, 160, 240 W) and different ultrasonic treatment time periods (10, 20, 30, 40, 50, 60 min) were shown in Figs. 5 and 6, respectively. The ending pH was set at 3.0 during the test by the leaching procedure 1 with a temperature of 25 °C and stirring speed at 600 rpm.

The results of Figs. 5 and 6 showed that the leaching efficiency of Cu increased with the rise of ultrasonic power and

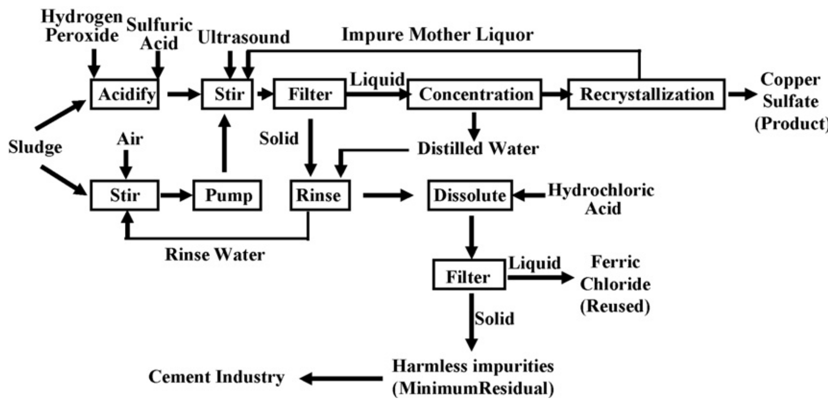

Fig. 7. The industrial treatment process flow sheet.

ultrasonic treatment time period while the leaching efficiency of Fe decreased, therefore the separation efficiency of copper and iron from PCB waste sludge increased with same scenario. Also shown in Figs. 5 and 6, after test time reached 60 min there was no significant change of the leaching efficiency between 240 and 160 W. Therefore 160 W (for 1 l tank) and 60 min can be chosen as an optimal power and applying time for the ultrasonically assisted leaching to separate copper and iron in PCB waste sludge.

3.5. The industrial application of the ultrasonically assisted separation procedure

This ultrasonically assisted separation procedure has been applied industrially in a heavy metal recovery plant in the city of Huizhou of Guangdong province in China. The whole industrial treatment process was shown in Fig. 7.

The process has been used for industrial application for more than two years. The annual quantity of PCB waste sludge treated by the novel cleaner process of the plant was 5800 tons in 2007. From it 1000 tons 98% copper sulfate and 3500 tons 20% liquid ferric chloride were produced. The produced copper sulfate was all sold in market and ferric chloride was all reused at local PCB manufacturing factories. There was no second pollution produced by the novel cleaner process. Only final remains from the process were 120 tons harmless inert acid-insoluble solids that were used as raw material by a local cement plant. The industrial application result has proved that the ultrasonically assisted separation procedure provides a cost effective way to recover heavy metal with high recovery efficiency from industrial waste sludge and reduces public health risk. It reduces added material consumption, recycles and reuses all process water in the process. It has higher separation and recovery efficiencies, lower recovery cost, higher end product quality, and zero process waste emission.

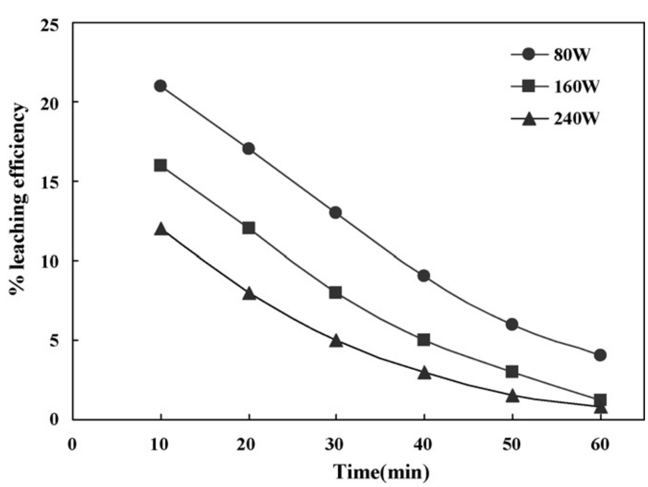

Fig. 6. The influence of ultrasound power and applying time on iron leaching efficiency.

4. Conclusions

Ultrasonic induced fluid cavitations which can enhance both dissolution of copper hydroxide into liquid phase and precipitation of ferric hydroxide into solid phase. Copper and iron can be separated much more efficiently and completely from a mixture by ultrasonically assisted acid leaching than the conventional heated acid leaching without ultrasound. The leaching procedure, pH value, ultrasonic power and applying time, all have impacts on the metals separation efficiency. With the appropriate leaching procedure, ending pH of 3.0, ultrasonic power of 160 W (in 1 l tank), leaching time of 60 min, leaching efficiencies of copper and iron reached 97.83% and 1.23%, respectively. Therefore separation of copper and iron in PCB waste sludge was virtually achieved. The separation approach presented by this paper has characters of low cost, low energy consumption and zero discharge of wastes, it has a great prospective on industry applications. The approach has been successfully applied to the industrial scaled applications in a heavy metal recovery plant in Huizhou city, China for more than two years, and industrial scale recovery of valuable metals from PCB waste sludge has been achieved. It has a great prospect on applications in heavy metal recovery industry and environment protection practices.

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