

Microwave enhanced stabilization of heavy metal sludge

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Abstract

A microwave process can be utilized to stabilize the copper ions in heavy metal sludge. The effects of microwave processing on stabilization of heavy metal sludge were studied as a function of additive, power, process time, reaction atmosphere, cooling gas, organic substance, and temperature. Copper leach resistance increased with addition of aluminum metal powder, with increased microwave power, increased processing time, and using a gaseous environment of nitrogen for processing and air for cooling [N₂/air]. The organic in the sludge affected stabilization, whether or not the organic smoldered. During heating in conventional ovens, exothermic oxidation of the organic resulted in sludge temperatures of about 500 °C for oven control temperatures of 200–500 °C. After microwave heating dried the sludge, the sludge temperature rose to 500 °C. The reaction between copper ions and metal aluminum in the dried sludge should be regarded as a solid phase reaction. Adding aluminum metal powder and reaction temperature were the key parameters in stabilizing copper in the heavy metal sludge, whether heated by microwave radiation or conventional oven. The mass balance indicates insignificant volatilization of the copper during heating.

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1. Introduction

Heavy metal sludge produced from the sequence of flocculation, pH adjustment, and alkaline precipitation in printed circuit board plants are often regarded as hazardous material. In addition to metal ions and insoluble metal hydroxide, the heavy metal sludge contained several salts (CaSO₄, CaCO₃, NaCl, and NaHCO₃) and a polymer that is used as a chelating agent [1]. In Taiwan, the copper is recovered from waste metal sludge by sulfuric acid extraction and copper compound crystallization. TCLP leaching of the residual sludge still results in leachate copper concentrations above the legal limit for land disposal (<15 mg/L). Therefore, further treatment of the residual sludge is required before landfill disposal.

Compared with other conventional thermal treatment technologies, the microwave technique with the characteristics of polar oscillation and effect of dielectric losses offers the advantage of selective, uniform, and rapid heating. For solid phase

samples with heterogeneous composition, the phenomena of superheating and hot spot were encountered [2]. Furthermore, electromagnetic microwave has also been widely used in the treatment of environmental materials, such as heating and pyrolysis [3,4], assisted extraction and digestion [5–8], organic and inorganic synthesis [9–11], and sample preparation [12,13]. Moreover, growing interests in stabilization and immobilization of metal ions in soil and sludge through microwave radiation have also been reported [14–18]. Results indicated that microwave radiation inhibits leaching of metal ions from soil or sludge and that it makes these solid wastes acceptable for disposal or recycle.

In this study, the stabilization of heavy metal sludge enhanced by microwave radiation was investigated. The objects of this study were to: (1) determine the proper procedure for stabilization of heavy metal sludge by microwave energy, (2) discuss the effects of additives, reaction atmosphere, cooling gas, organic substances and temperature, (3) compare the difference between conventional oven and microwave radiation on sludge stabilization, and (4) conjecture the stabilizing mechanism of heavy metal sludge.

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Table 1
Operating conditions of different treatment

Experiment	Additive	Power (W)	Process time (min)	Atmosphere and cooling gas
Effect of additive	Al powder (0–3.09 g), α -Al ₂ O ₃ (0–2 g), γ -Al ₂ O ₃ (0–2 g)	600	12	Air
Effect of microwave power and process time	Al powder (0.39 and 0.58 g)	400, 600, 800	0–25	Air
Effect of atmosphere and cooling gas	Al powder (0, 0.39 and 0.58 g)	600	0–15	Air or N ₂
Effect of organics (Stage I)	No additive	600	12	Air
Effect of organics (Stage II)	Al powder (0.58 g)	600	0–15	Air

2. Materials and methods

2.1. Sample preparation and analysis

Raw heavy metal sludge from a Heavy Metal Sludge Treatment Plant in Taiwan had been treated by an acid extraction process. However, in terms of toxic characteristics leaching procedure (TCLP, R.1311, US EPA), copper ion concentrations of the raw sludge at moisture content 0% (132 mg/L) still do not meet the criteria of waste disposal in Taiwan (15 mg/L) and therefore a stabilizing treatment of heavy metal sludge is needed before disposal or reuse.

The pretreatment steps of the raw heavy metal sludge were as follows: (1) dry in an oven at 105 °C until the mass maintains a constant value within $\pm 1\%$ (about 12 h), (2) crush the dried sludge with a grinder, (3) sieve to a particle diameter between 100 and 400 mesh (0.15–0.063 mm), and (4) store the dried, crushed, sieved sludge in a 20 L bucket with a plastic cover. Worth note that the term “RS” used in this study means raw sludge after pretreatment.

The metal composition of the RS was determined by a modified microwave digestion method reported in previous studies [6,19]. Samples of raw heavy metal sludge (0.1 g) were mixed with a HNO₃–HCl–HF solution (3 mL). These acid slurries were processed using the following two-stage microwave program: (1) 650 W for 10 min (final temperature 160 °C) and (2) 800 W for 15 min (final temperature 200 °C). After the microwave-assisted digestion, the mixtures were cooled to room temperature, filtered, and the filtrate volume adjusted to 50 mL before analysis. The same microwave digestion method was also performed on the stabilized sludge, SS_L, after stabilization with 0.58/40 (g/g) aluminum powder/sludge at 600 W microwave radiation for 12 min.

In the experiments, 40 g of RS was put in a SiO₂ ceramic crucible (11 cm diameter) and the sludge moisture content adjusted to 50 wt.% with deionized water. An industrial microwave oven was used in this study with a microwave frequency of 2.45 GHz and variable power up to 1600 W. Treated samples were leached using TCLP and the leaching concentrations of Cu, Fe, and Al were analyzed by inductively coupled plasma-atomic emission spectrometry (ICP-AES).

2.2. Different orders of processing

The following four orders of processing were tested: (1) microwave radiation of 600 W and process time of 12 min with-

out adding any aluminum powder, (2) adding aluminum powder without microwave radiation, (3) with microwave radiation of 600 W and process time of 12 min then with the addition of aluminum powder, and (4) aluminum powder of 0.58 g was added with simultaneous microwave radiation.

2.3. Additives

Three aluminum series additives, aluminum powder, α -Al₂O₃, and γ -Al₂O₃, with different microwave adsorption properties were tested in the following range of masses added per 40 g of RS: (1) 0–3.09 g of aluminum metal powder, (2) 0–2 g of α -Al₂O₃, and (3) 0–2 g of γ -Al₂O₃. The operating conditions were microwave power of 600 W, process time of 12 min, and reaction atmosphere/cooling gas of air/air (see Table 1).

2.4. Microwave power and process time

In the microwave heating experiments, the higher the microwave power, the shorter the process time needed. Therefore, the effect of microwave power and process time were studied with the addition of aluminum powder and the operating parameters that are listed in Table 1.

2.5. Effect of reaction atmosphere and cooling gas

Two gases were used as the reaction atmosphere and cooling gas: air and nitrogen. The test procedure for establishing the ambient gas during the test follows: (1) put the sample in the microwave oven, (2) evacuate the oven with a vacuum pump, (3) fill the oven with the reaction atmosphere (air or nitrogen) to a pressure of nearly 76 cmHg, (4) perform the stabilizing experiment, and (5) vent the oven and blow the cooling gas (air or nitrogen) through the oven for 15 min.

2.6. Organics

A polymer used during heavy metal wastewater treatment made the organic content of the heavy metal sludge significant, almost 25% (w/w). A two-stage experiment was designed to better understand the influence of the organic content on stabilization. This experiment consisted of the following steps: (1) microwave RS at 600 W for 12 min to break down or decompose the organics (end of Stage I), (2) take 40 g of the sludge from Stage I and adjust its moisture content to 50% (w/w), (3) add

Table 2
Chemical composition of RS and SS_L

Element	RS (mg/g)	SS _L (mg/g)
Ag	N.D.	N.D.
Al	3.77	4.80
Ba	2.06	N.D.
Ca	66.91	66.60
Cd	N.D.	N.D.
Co	N.D.	N.D.
Cr	N.D.	N.D.
Cu	11.58	11.30
Fe	13.68	13.09
Ga	N.D.	N.D.
K	6.01	5.27
Mg	1.68	1.69
Mn	0.05	N.D.
Na	14.69	11.60
Ni	N.D.	N.D.
Pb	0.15	N.D.
Si	85.40	85.00
Zn	0.52	0.56

N.D., not detected.

0.58 g aluminum metal powder, and (4) microwave at 600 W for times ranging from 0 to 15 min (end of Stage II, see Table 1).

2.7. Temperature

Temperature is an important factor in thermal treatment of solid waste. A conventional oven gave a controlled temperature environment in which to place sludge for studying the effect of temperature. The difference between conventional heating and microwave heating is discussed. Each test used 40 g of RS with the moisture content adjusted to 50% (w/w). Some had 0.39 g of aluminum metal powder added and some did not. The samples were placed in a conventional oven at temperatures of 200, 300, 400, or 500 °C, and the sludge temperature recorded with time. Another oven experiment was also performed with RS without additive at stationary oven temperatures of 200, 300, 400, 500, 700, or 800 °C for a heating time of 6 h.

3. Results and discussion

3.1. Chemical composition of RS and stabilized sludge

Table 2 lists the extractable elemental composition measured in samples of the RS and the stabilized heavy metal sludge, SS_L.

Table 3
The TCLP concentration of metals from different treatments

Case	Treatment	TCLP concentration (mg/L)		
		Cu	Fe	Al
RS	None	132.0 ^a	N.D.	17.28
I	Microwave radiation only (600 W × 12 min)	445.1 ^a	N.D.	20.4
II	Al powder only (0.58 g Al powder/40 g RS)	55.1 ^a	22.71	92.44
III	1. Microwave radiation (600 W × 12 min). 2. Additive (0.58 g Al powder/40 g RS)	221.7 ^a	5.85	12.68
IV	Additive (0.58 g Al powder/40 g RS) + microwave radiation (600 W × 12 min)	3.6	230.3	131

^a Exceeds the TCLP criterion of 15 mg/L for Taiwan.

The major elements in the sludge were Si, Ca, Fe, Cu, and Al in decreasing sequence. The large amounts of Ca, Fe, and Al in the RS are attributed to the usage of lime, ferrous sulfate, and poly aluminum chloride, respectively, in wastewater treatment, while the Cu and Si came from the printed circuit board treatment. The copper concentrations in the RS and SS_L were 11.58 and 11.3 mg/g, respectively. The concentration difference is only $2.4 \pm 0.2\%$ between the RS and SS_L, meaning copper volatilization was negligible during the thermal treatment. The decrease of copper leaching concentration in stabilizing experiments may be attributed to the conversion of copper species. The instrument detection limit (IDL) of the liquid extract was of 0.1 mg/L, which translates into a detection limit for the sludge of 0.05 mg/g. Thus, N.D., or “not detected”, in Table 2 means <0.05 mg/g. All the values of metal concentration, mg/g, listed in Table 2 were corrected to the equivalent RS basis.

3.2. Choice of stabilizing processes

For Case I in Table 3, the RS with moisture adjusted to 50% (w/w) was in an aerobic condition and subjected to microwave radiation with no aluminum metal powder, resulting in a TCLP copper leaching concentration much higher than before treatment. The sludge dried after microwaving 12 min and the temperature rose to 500 °C, causing the organics (a polymer chelating agent) in the sludge to smolder. The polymer oxidized or decomposed, releasing the copper hydroxide trapped in the polymer to subsequently be leached during TCLP testing.

In Cases II and III, the TCLP copper leaching concentrations still exceeded the criterion for waste disposal in Taiwan (15 mg/L). Only Case IV decreased the TCLP copper leaching concentration below this regulatory limit. Therefore, the experimental process of Case IV was adopted as the stabilization process of choice.

3.3. Effect of additives, microwave power and process time

Fig. 1 illustrates the effect of different aluminum additives and their dosage by the following observations: (1) the TCLP copper concentrations dropped dramatically upon the addition of the aluminum additives up to an additive/RS ratio of about 0.39/40 (g/g), but remained relatively flat with further additions. (2) There was no obvious difference in microwave stabilizing tendency between γ -Al₂O₃ with higher microwave absorbance and α -Al₂O₃ with lower microwave absorbance. (3) Aluminum

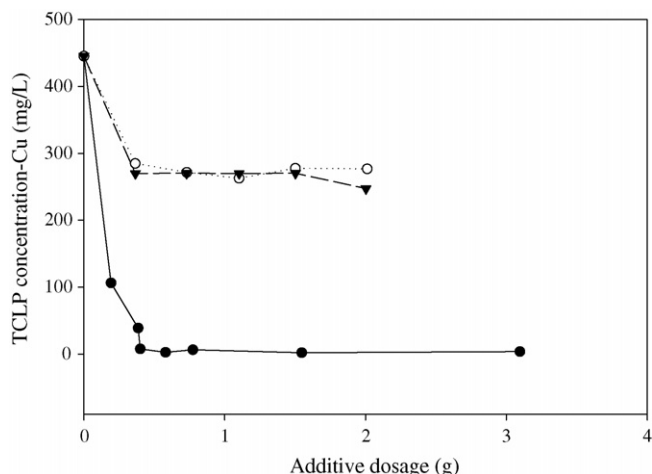


Fig. 1. The effect of additives: (●) aluminum powder, (○) α -Al₂O₃, and (▼) γ -Al₂O₃.

powder combined with microwave radiation effectively stabilized the copper in the heavy metal sludge. These results indicate that the lattice arrangement of Al species is not a factor in the stabilization of heavy metal sludge, but the valence of the Al species is. Apparently, the aluminum metal powder effectively reduced the copper species in the sludge under microwave heating. Iron powder or iron wire of zero valence efficiently decreased the leaching concentration of metal ions in solid wastes with microwave radiation in prior studies [14–16]. In the preliminary studies of this work, the addition of Fe₂SO₄·7H₂O or FeCl₃ did not effectively stabilize the heavy metal sludge. Also, decomposing organic carbon during microwave heating not only did not reduce or stabilize the copper at the temperatures attained, but also released the copper ions chelated in the organic carbon to be more easily leached in subsequent TCLP testing. Apparently, the reducing ability of metal powders is required to stabilize the metals in heavy metal sludge at the levels of microwave power and temperatures used in this study. The role of the silicon, alkaline, and alkali species in this stabilization is uncertain, but likely result in thermodynamically stable metal-alumina-oxide species.

Fig. 2 shows that the leaching concentration of copper ions decreased with the increasing values of microwave power, process time, and aluminum quantity (0–0.58 g Al/40 g RS (g/g), no improvement above 0.58/40 (g/g)). The experimental data varied more at higher microwave power. The intermediate microwave power of 600 W was used in later experiments as a compromise between more efficient stabilization and reproducibility. The reducing ability of aluminum powder may be responsible for the decreasing of copper leachability during the first 3 min (see Fig. 2). Dissolved Cu(OH)₂ may be reduced to Cu⁰, which was not easily oxidized to CuO and hydrated back to dissolved Cu(OH)₂ during TCLP testing. During the 3–9 min period, the moisture of the heavy metal sludge gradually decreased, reaching 0 wt.% at the 9 min mark and the temperature of the sludge was below 150 °C. This temperature is too low to destroy the organic chelate and release more copper ions for reduction. Thus, the stabilizing efficiency decreased or did not improve

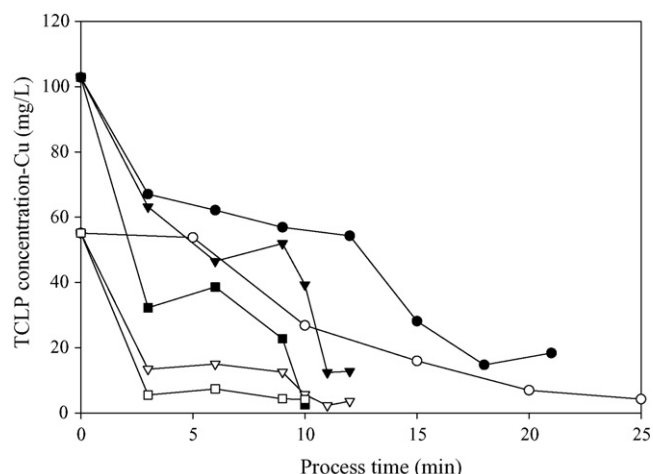


Fig. 2. The effect of microwave power and process time. Addition of 0.39 g Al powder with microwave radiation of (●) 400 W, (▼) 600 W, and (■) 800 W. Addition of 0.58 g Al powder with microwave radiation of (○) 400 W, (▽) 600 W, and (□) 800 W.

during this time period. Past 9 min, with the moisture vaporized, the temperature of the heavy metal sludge rose steeply up to 450–500 °C and the organic contents smoldered and were destroyed. The weight loss from organic destruction (about 25% loss) was not included in the calculation of weight loss for the samples. In the phase after 9 min, the stabilizing efficiency increased with time, because of the higher temperature of the heavy metal sludge from microwave radiation in the absence of water. The reaction between copper ions and additive is a solid phase reaction at this point. In a solid phase reaction, the reactants cannot migrate freely and the theories of Brownian motion do not apply. Therefore, the reaction mechanism at this stage cannot be interpreted entirely by the theories of aqueous chemistry.

3.4. Effect of reaction atmosphere and cooling gas

The effect of reaction atmosphere and cooling gas is essential for understanding a microwave system. Fig. 3 shows no stabilization occurred without the additive, whether the reaction atmosphere and cooling gas were air or nitrogen. The organics in the sludge was completely oxidized for air/air (reaction gas/cooling gas) within 9 min, making the copper ions more leachable in the TCLP test. The N₂/N₂ combination prevented this oxidation and kept the rising temperature moderate (under 150 °C). Therefore, it was supposed that under microwave radiation and an inert reaction atmosphere, the organic substance would consume little of the aluminum powder in later stabilizing experiments. Fig. 4 illustrates that the best combination was a N₂ reaction atmosphere coupled with cooling air, shortening the process time for stabilization of the heavy metal sludge. The microwave radiation may increase the energy and reactivity of the aluminum powder, but the inert atmosphere decreases the oxidation and destruction of the organic, implying that the activated Al powder was not consumed by the organic. In addition, the cooling air may improve the reaction between aluminum powder and copper ions, but the lower temperature prevented

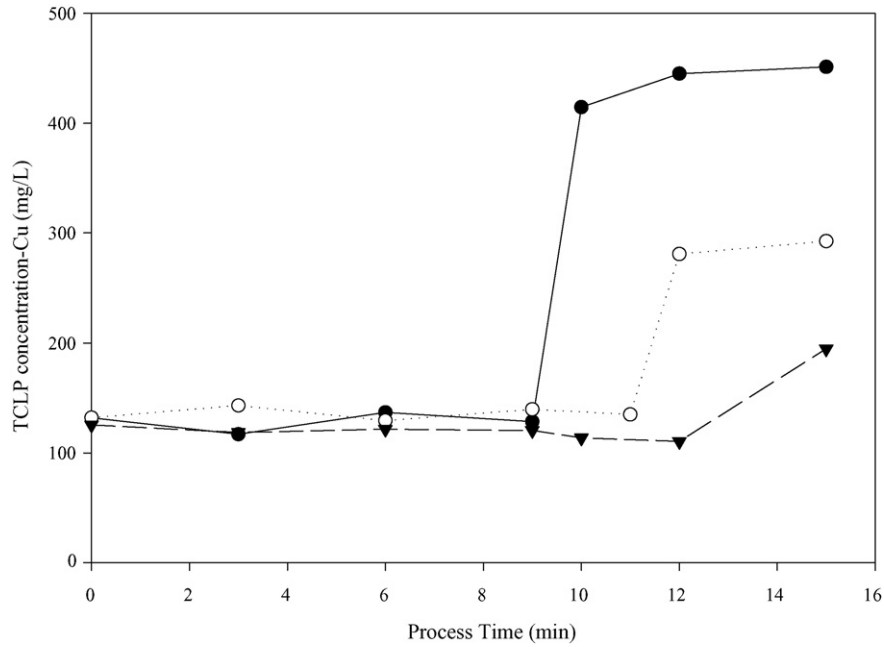


Fig. 3. The effect of atmosphere and cooling gas without additive: (●) air/air, (○) N₂/air, and (▼) N₂/N₂.

smoldering. Additive/RS ratios of more than 0.58/40 (g/g) minimized the effect of reaction atmosphere and cooling gas.

3.5. Effect of organic substance

During the process of microwave treatment, the organic substance played an important role in the stabilization of heavy metal sludge. In the preliminary study, similar experiments to Case IV in Table 3 were also performed. However, the results indicated that at the existence of aluminum powder whether the sludge smoldered or not, the leaching concentration of copper ions would still be affected (from 3.6 to 39 mg/L). Concentration of 3.6 and 39 mg/L represented the smoldering case and the non-smoldering case, respectively. As indicated in Section 3.2, it was supposed that the smoldering of sludge and destruction

of organics would release the copper ions chelated by organics which would offer a positive opportunity for the collision between copper ions and aluminum powder.

Fig. 5 illustrates the two-stage stabilization with process time. The organic in the sludge was destroyed with a sludge mass loss of almost 25 wt.% during the first stage of processing. The second stage of processing stabilized the heavy metal sludge efficiently with little variation in the stabilizing curve and a temperature rose to only 200 °C even at a process time of 15 min. These results imply that the organic is a good microwave adsorbent and its presence is advantageous in controlling temperature in the absence of oxygen. A similar result was found for the pyrolysis of sewage sludge [4]. Fig. 5 shows decreased for Cu leaching and increased Fe and Al leaching. The following two hypotheses may explain this behavior for stabilization by microwave heating. Hypothesis I: (1) Al⁰ reduces Fe³⁺ forming

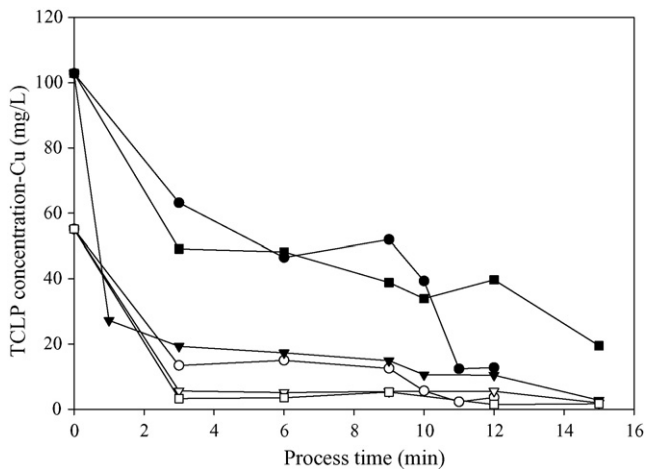


Fig. 4. The effect of atmosphere and cooling gas with additives. Addition of 0.58 g Al powder with (○) Air/Air, (▼) N₂/Air, and (□) N₂/N₂. Addition of 0.39 g Al powder with (●) Air/Air, (▼) N₂/Air and (■) N₂/N₂.

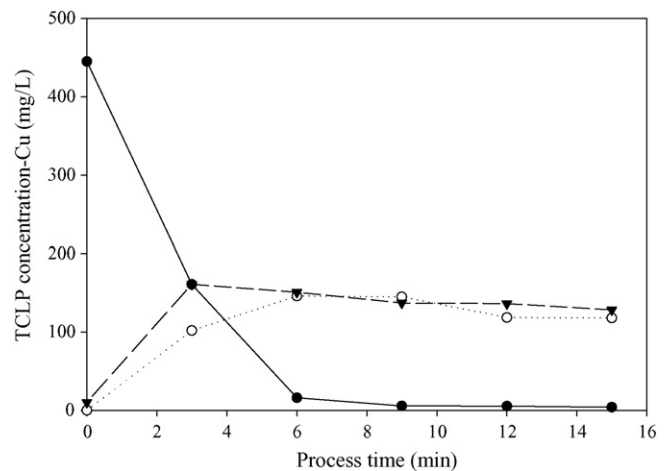


Fig. 5. Stabilizing curve of two-stage experiment: (●) Cu, (○) Fe, and (▼) Al.

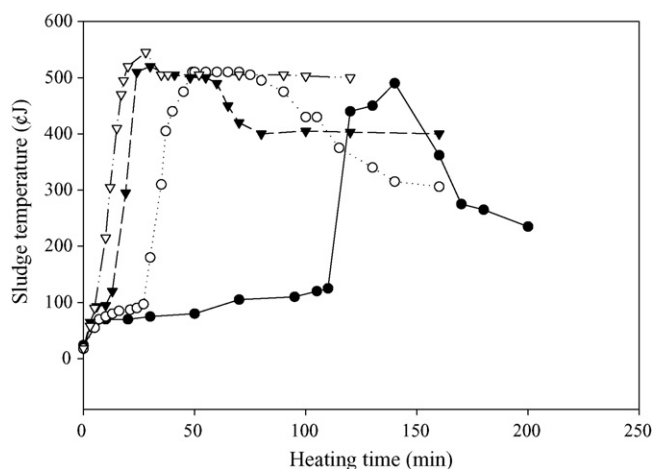


Fig. 6. Temperature variation of the heavy metal sludge without additive at conventional oven (●) 200 °C, (○) 300 °C, (▼) 400 °C, and (□) 500 °C.

Al^{3+} and Fe^{2+} or Fe^0 . (2) Fe^{2+} or Fe^0 reduces Cu^{2+} forming Cu^0 and Fe^{2+} , contributing to the stabilization of Cu and the leaching of Fe. Hypothesis II: Al^0 reduces Fe^{3+} and Cu^{2+} simultaneously forming Fe^{2+} and Cu^0 . The amorphous nature of the heavy metal sludge prevented detection of the compounds formed. Analytical identification of the chemical species is needed to better understand the chemical reactions occurring during microwave heating of the heavy metal sludge mixed with aluminum metal powder.

3.6. Effect of temperature

Samples of 50 wt.% sludge solids, 50 wt.% moisture, and no aluminum additive were placed in a conventional ovens held at a constant temperature ranging from 200 to 500 °C for 6 h and the sludge temperatures were monitored with time to study the effect of temperature. Fig. 6 illustrates the temperature of the heavy metal sludge with time at different conventional oven temperatures. The temperature curves show that the sludge temperature rose up to 500 °C, even with the oven temperature at 200 °C. Exothermic oxidation of the sludge organic accounts for the temperatures exceeding those of the oven. The sludge temperatures eventually declined to the oven temperatures in all the tests. Fig. 7 illustrates the resulting TCLP copper leaching concentration plotted against the oven temperatures, with and without the aluminum metal additive. The temperature curves including the aluminum metal powder additive (not displayed in Fig. 6) were no different than those without the additive. The results indicate that effective stabilization of the heavy metal sludge with the additive occurred at oven temperatures of 200 °C and above. The TCLP copper leaching concentration maximized at 400.2 mg/L for the sludge without additive held at 300 °C and declined with oven temperature to 28.1 mg/L at 800 °C. Without additive, the leaching copper concentration declined gradually as the oven temperature increased above 400 °C. Just as with the microwave oven, the conventional oven effectively stabilized the sludge at 200 or above, when the aluminum metal powder addi-

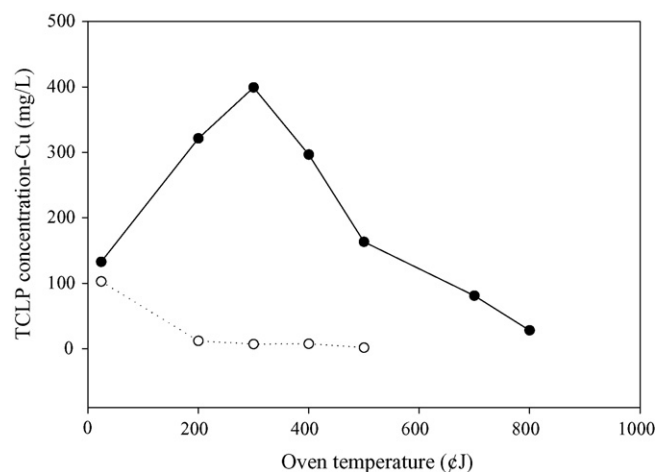


Fig. 7. TCLP concentration of copper ions from heating in the conventional oven (●) without additive (○) with additive.

tive was used. Higher temperatures do appear to stabilize the copper, even without additives. Extrapolating the plot in Fig. 7, a temperature of 850 °C must be used without the additive to meet the Taiwan criterion of 15 mg/L. This temperature can be achieved with either conventional or microwave heaters. Clearly, the aluminum metal powder allowed more effective stabilization of the heavy metal sludge at lower temperatures.

4. Conclusion

The study of stabilization of a heavy metal sludge generated by printed circuit board plants enhanced by microwave radiation led to the following conclusions:

1. The major elements of the raw heavy metal sludge are Si, Ca, Fe, Cu, and Al in decreasing sequence.
2. Microwave heating experienced no significant mass losses during treatment.
3. Adding aluminum metal powder made the thermal copper stabilization much more efficient and effective.
4. Increasing the microwave power and process time also improved copper stabilization. The reaction between copper ions and aluminum metal is a solid phase reaction, because the moisture vaporizes before the higher temperatures are achieved.
5. An inert reaction atmosphere (N_2) during heating and oxidizing atmosphere (air) for cooling gave better performance.
6. The organic content in the heavy metal sludge affected the stabilizing curve, whether or not the organic was destroyed (smoldered) during treatment.
7. Exothermic oxidation of the organic results in a sludge temperature higher than the temperature in a conventional oven.
8. The reducing ability of aluminum metal and higher temperatures stabilized the copper in the heavy metal sludge heated by microwave radiation or in a conventional oven.

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