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Treatment of high copper-containing wastewater by producing magnetic material

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ABSTRACT

The purpose of this study is to provide a materialization method for high copper-containing wastewater and compare it with conventional chemical precipitation process and ferrite process. In the first stage of mineralization, the copper-containing wastewater was added into a tank with NaOH solution followed by the addition of Fe^{3+} ion and heated to 70°C. The molar ratio between Fe^{3+} and divalent metal ions (M_t^{2+}) was given and the pH value of solution was controlled in the strong alkaline range (11.0–11.5). In the inverse mixing method, the whole precipitation process was carried out in the strong alkaline solution, which resulted in the simultaneous precipitation of Fe^{3+}, Cu^{2+}, Ni^{2+} and Zn^{2+} ions. The first reaction was complete when the ORP remained constant. Finally, the formation of the magnetic ferrite, Fe^{2+} ion was added into the solution accompanied by heating and aeration. The magnetic separation could be then used for solid–liquid separation. Therefore, these results reveal that not only the filtrate lies below the regulatory effluent standards, but also the solid products meet the toxicity characteristic leaching procedure standards. Moreover, the solid products are magnetic and have the characteristic of evenly distributed copper ions.

Keywords: Materialization; High copper-containing wastewater; TCLP; Magnetic material

1. Introduction

In Taiwan, the rapid expansion of the electronic and communication industries increasing amounts of heavy metals containing wastewater are produced in the electroplating factories and during the manufacturing processes of printed circuit boards (PCBs), such as electroplating, etching, and washing [1,2]. The more strict regulations for the concentration of heavy metal ions (i.e. Cu, Pb, Cr, Zn, and Cd) in effluents motivate the search of an efficient, cost-effective, and environmental friendly process to treat such high heavy metals containing wastewater [3,4].

Generally, wastewater is treated by the use of the chemical precipitation process and the coagulants are

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used to treat wastewater [5–7]. Besides the ferrite process for toxic and metal removal from wastewater consists of the precipitation of heavy metal ions from the alkaline solution containing ferrous ions and the alkalization for the formation of ferrous hydroxide, and the partial oxidation of Fe^{2+} to Fe^{3+} by aeration at a temperature above 50℃ [8–10]. The magnetic ferrite can be formed and also cause the co-precipitation of other metal ions present in the wastewater. The crystalline structure of the ferrite is the same as that of the cubic spinel (AB_2O_4) in which A^{2+} and B^{3+} are replaced by nonferrous metals M_t^{2+} and Fe^{3+}, respectively. Thus, MFe_2O_4 is generally defined as ferrite's chemical formula. Due to its high stability, ferrites form easily during the treatment of heavy metal-containing wastewater by wet oxidation [11–14]. The reactions of ferrite formation are as follows:

$$XM_t^{2+} + (3-X)Fe^{2+} + 6OH^- \rightarrow M_{tX}Fe_{3-X}(OH)_6 \tag{1}$$

$$M_{tX}Fe_{3-X}(OH)_6 + \frac{1}{2}O_2 \rightarrow M_{tX}Fe_{3-X}O_4 + 3H_2O \tag{2}$$

Nevertheless, Cu^{2+} cannot be incorporated into the crystalline structure of the ferrite generated from copper containing wastewater. This might be due to the early formation of copper oxide (CuO) in the alkaline solution during oxidation. For a CuO–H_2O system, it is apparent that the potential/pH diagram shows CuO is formed when the concentration of copper ions in the solution reaches 10^{-6} M, and pH value is greater than 7.0 [15,16]. Furthermore, the leaching concentration of copper in the residual solid (copper-containing sludge) from the chemical precipitation and ferrite processes does not meet the regulation standards defined by EPA of Taiwan.

In order to purify heavy metal containing wastewater which may pose serious environmental problems and recover heavy metals for industrial reuse and materialization, numerous hydrometallurgical technologies have been developed in the past. The purpose of the present work is to investigate the possibility of the materialization method (the homogenization, mineralization, and magnetization processes) for high copper-containing wastewater and compare it with conventional chemical precipitation process and ferrite process.

2. Materials and methods

2.1. Copper-containing wastewater

In order to obtain the optimum reaction condition, four kinds of copper containing wastewater were synthetically prepared based on real process information. Real wastewater samples A, B, and C (WA, WB, and WC) were produced from a metal surface treatment factory, a electroplating factory, and a manufacturing processes of PCBs located at industrial areas in Taoyuan County for this research work. The heavy metal concentrations of wastewater and filtrate from the chemical precipitation process, ferrite process, and materialization process were determined using atomic absorption spectrometry (Perkin Elmer, AA 400). The stabilities of those solid products were tested by the toxicity characteristic leaching procedure (TCLP) and characterized by X-ray diffractometer (XRD, Bruker D8, Cu Kα radiation).

2.2. Experimental methods

Metal concentrations in those samples were analyzed by the above-mentioned method. TCLP test of samples were performed according to NIEA R201.12C (Taiwan EPA). The crystalline phases of the residual solids after being dried at 60℃ were further analyzed with an XRD. Crystalline phases were identified with database of International Center for Diffraction Data (JCPDS-ICDD). 2θ was set from 20^o to 80^o at a scan rate of 0.5˚/min. Apparatus for wastewater treatment by the materialization treatment method is shown in Fig. 1. It can be divided into three stages. The first stage is the pretreatment homogenization process (the wastewater tank and the pH condition tank), and the second one is the mineralization and magnetization process (the reaction tank and sedimentation tank), and the third one is separation, washing, and drying process (press, the filtrate tank the washing tank and the oven).

High copper-containing wastewater was stirred in the NaOH solution at 500 rpm for 20 min to form a homogeneous suspension. The main purpose of the first homogenization process is to compare with the conventional mixing method (adding NaOH solution to high copper ions containing wastewater). The opposite way (adding high copper ions containing wastewater to NaOH solution) seems more suitable for the formation of ferrite since the formation of CuO and α–Fe_2O_3 in ferrite was prevented effectively. The reason for this may be attributed to the different dissolution constants of the corresponding metal hydroxides [17].

The mineralization process had the following parameters: (1) a Fe^{3+}/M_t^{2+} (Cu^{2+}, Ni^{2+}, Zn^{2+}, etc.) mole ratio of 4, (2) the volume of the suspension at 1,000 ml, (3) the suspension stirred continuously at 400 rpm, (4) the pH of solution adjusted and

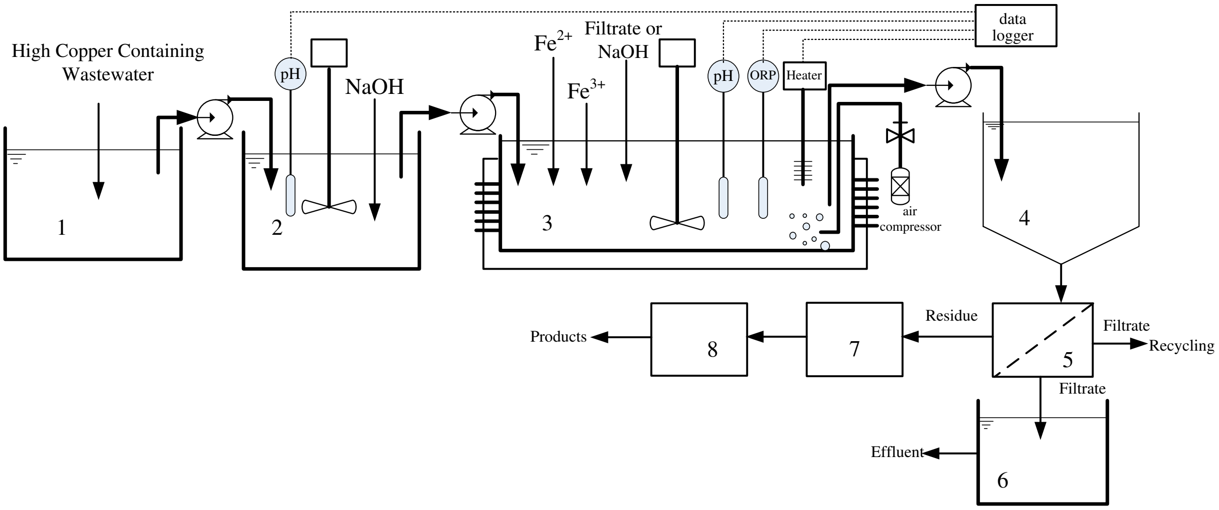

Fig. 1. Apparatus for wastewater treatment by the materialization treatment method (1: wastewater tank; 2: pH conditioned tank; 3: reaction tank; 4: sedimentation tank; 5: press; 6: filtrate tank; 7: washing tank; 8: oven).

maintained greater than 12.5 by adding 45% NaOH in the strong alkaline solution, and (5) a reaction temperature of 70˚C.

The Fe^{2+}/M_t^{2+} mole ratio of 4 was controlled. The pH of solution was adjusted to greater than 10.5. The suspension was then stirred continuously at 400 rpm, and the reaction temperature was set to 70˚C. After the temperature reached 70˚C, aeration with flow rate of 5 L air/min was initiated. The reaction time of the magnetization process was determined by monitoring ORP for about 50–90 min [18–20]. After the completion of the magnetization process, solid–liquid separation was adopted by filtration, and the residues were washed and dried at 60˚C for 24 h. Afterward, samples (filtrate and materialization products) were characterized. The schematic diagram of the experimental procedure is shown in Fig. 2.

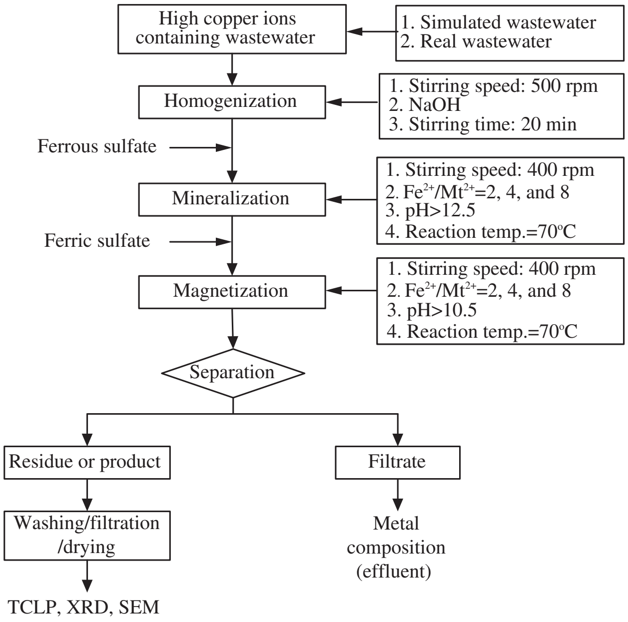

Fig. 2. Schematic diagram of the experimental procedure including the homogenization, mineralization, and magnetization processes.

3. Results and discussion

3.1. Characteristics of simulated wastewater and filtrate

The copper concentrations of simulated wastewater (SW) were 600, 2,000, 6,000, and 24,000 mg/L. The results of filtrate and TCLP by the traditional chemical precipitation process, ferrite process, and materialization process are shown in Tables 1 and 2. The residual heavy metal concentrations of SW after the chemical precipitation process, ferrite process, or materialization process were lower than the effluent standard and the removal efficiency of Cu^{2+} was higher than 99.5%. However, the pH value of SW after the ferrite process or materialization process was higher than the effluent standard. This is because the NaOH was added to the solution in order to enhance the formation of ferrite or materialization. The different ratios of Fe^{3+}/M_t^{2+} and Fe^{2+}/M_t^{2+} were also evaluated. In Table 1, the residual heavy metal concentrations of SW after the materialization process was not influenced by the Fe^{3+}/M_t^{2+} and Fe^{2+}/M_t^{2+} ratio.

The residual heavy metal concentrations were lower than the effluent standard. However, the leaching concentration of the residues after the chemical precipitation process, ferrite process, or materialization

Table 1
Characterizations of SW and filtrate from the chemical precipitation process, ferrite process, and materialization process

Items	Cu (mg/L)	Cr (mg/L)	Ni (mg/L)	Zn (mg/L)	pH
Initial SW1	600	100	200	100	3.06
Chemical precipitation process	0.19	0.17	0.11	0.08	7.84
Ferrite process	0.26	0.11	0.13	0.16	10.52
Materialization process 1[a]	0.36	0.19	0.11	0.19	9.95
Materialization process 2[b]	0.31	0.21	0.12	0.20	10.09
Materialization process 3[c]	0.16	0.12	0.10	0.13	10.43
Initial SW2	2,000	100	200	100	3.07
Chemical precipitation process	0.13	0.21	0.21	0.15	8.12
Ferrite process	0.29	0.08	0.17	0.10	10.74
Materialization process 1[a]	0.32	0.15	0.24	0.11	9.88
Materialization process 2[b]	0.30	0.15	0.22	0.04	9.81
Materialization process 3[c]	0.32	0.15	0.19	0.07	9.94
Initial SW3	6,000	100	200	100	2.95
Chemical precipitation process	0.21	0.16	0.15	0.14	7.88
Ferrite process	0.15	0.23	0.13	0.05	10.48
Materialization process 1[a]	0.41	0.13	0.24	0.11	10.2
Materialization process 2[b]	0.34	0.18	0.28	0.14	10.18
Materialization process 3[c]	0.40	0.13	0.22	0.18	10.61
Initial SW4	24,000	100	200	100	3.02
Chemical precipitation process	0.27	0.14	0.04	0.24	7.97
Ferrite process	0.21	0.09	0.18	0.20	10.79
Materialization process 1[a]	0.28	0.26	0.14	0.18	10.35
Materialization process 2[b]	0.31	0.17	0.17	0.17	10.27
Materialization process 3[c]	0.31	0.17	0.17	0.16	10.42
Standard of effluent	3.0	2.0	1.0	5.0	6.0–9.0

Notes: [a]$Fe^{3+}/M_t^{2+} = 2$ and $Fe^{2+}/M_t^{2+} = 2$. [b]$Fe^{3+}/M_t^{2+} = 4$ and $Fe^{2+}/M_t^{2+} = 4$. [c]$Fe^{3+}/M_t^{2+} = 8$ and $Fe^{2+}/M_t^{2+} = 8$.

process had a big variance (Table 2). The leaching concentration of the residues after chemical precipitation process was higher than the TCLP limitation, especially for Cu and Cr. These residues after the chemical precipitation process were belonged to hazardous wastes and needed further disposal. Additionally, the chemical precipitation process did not favor for the resource of copper.

In the ferrite process, the Cu leaching concentration of the residues was higher than the TCLP limitation, especially for SW4. Cu^{2+} might not be incorporated into the crystalline structure of ferrite and partial CuO was formed in the higher pH level (>10.5). In contrast, the Cu leaching concentration of the residues after materialization process was lower than the TCLP limitation. This is because the $CuFe_2O_4$ was formed and Cu^{2+} had been incorporated into the crystalline structure to resist the leaching. The results are demonstrated by the XRD pattern (Fig. 3).

The XRD pattern of the residues after the chemical precipitation process was amorphous material and the intensity of diffraction peak is relatively low. The chemical composition of the residues after the chemical precipitation process should be $Cu(OH)_2$.

3.2. Characterizations of real wastewater and filtrate

In the inverse mixing method of the homogenization process, the whole precipitation process was carried out in the strong alkaline solution ($pH > 12.5$), which resulted in the simultaneous precipitation of Fe^{3+}, Cr^{3+}, Cu^{2+}, Ni^{2+}, Pb^{2+}, Cd^{2+}, and Zn^{2+} ions. Furthermore, the species in the strong alkaline were $Cu(OH)_3^-$ and $Cu(OH)_4^{2-}$

Table 2
Leaching concentration (TCLP test) of the residues for the chemical precipitation process, ferrite process, and materialization process

Items	Residues	Cu (mg/L)	Cr (mg/L)	Ni (mg/L)	Zn (mg/L)
SW1	Chemical precipitation process	124.50	25.30	35.70	24.90
	Ferrite process	78.60	1.50	1.10	0.60
	Materialization process 1[a]	2.24	1.02	0.55	0.64
	Materialization process 2[b]	2.11	0.91	0.82	0.48
	Materialization process 3[c]	1.87	0.85	0.64	0.63
SW2	Chemical precipitation process	178.80	135.80	67.90	15.60
	Ferrite process	217.20	2.40	0.60	0.70
	Materialization process 1[a]	4.45	1.87	0.78	1.02
	Materialization process 2[b]	4.31	1.85	0.68	0.98
	Materialization process 3[c]	3.42	1.34	0.59	0.93
SW3	Chemical precipitation process	875.31	389.42	42.77	11.53
	Ferrite process	647.22	1.20	0.37	0.30
	Materialization process 1[a]	6.03	1.05	0.82	0.57
	Materialization process 2[b]	5.83	1.07	0.63	0.42
	Materialization process 3[c]	5.68	0.97	0.57	0.38
SW4	Chemical precipitation process	978.10	798.30	58.99	17.60
	Ferrite process	1,145.50	1.94	1.01	0.70
	Materialization process 1[a]	6.72	1.21	0.93	1.20
	Materialization process 2[b]	5.81	0.98	0.88	0.84
	Materialization process 3[c]	6.07	0.82	0.85	0.78
TCLP limitation		15.0	5.0	—	—

Notes: [a]$Fe^{3+}/M_t^{2+} = 2$ and $Fe^{2+}/M_t^{2+} = 2$. [b]$Fe^{3+}/M_t^{2+} = 4$ and $Fe^{2+}/M_t^{2+} = 4$. [c]$Fe^{3+}/M_t^{2+} = 8$ and $Fe^{2+}/M_t^{2+} = 8$.

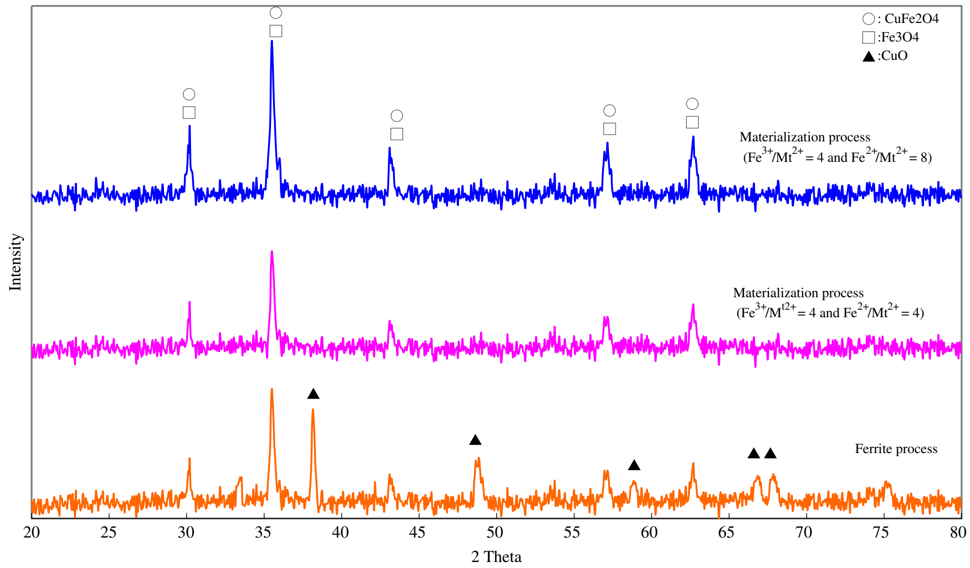

Fig. 3. XRD pattern of magnetic materials from SW after the ferrite process and materialization process.

Table 3
Characterizations of real wastewater and filtrate from the chemical precipitation process, ferrite process, and materialization process

Items	Cu (mg/L)	Cr	Ni	Cd	Pb	Zn
WA	2,789.1	855.4	389.5	31.3	141.2	1,282.5
Chemical precipitation process	0.24	0.27	0.18	N.D.	0.18	0.28
Ferrite process	0.28	0.24	0.22	N.D.	0.34	0.25
Materialization process[a]	0.34	0.22	0.11	N.D.	0.27	0.32
WB	3,635.4	392.3	535.5	11.1	53.0	1,308.4
Chemical precipitation process	0.21	0.11	0.09	N.D.	0.17	0.14
Ferrite process	0.31	0.09	0.07	N.D.	0.31	0.08
Materialization process[a]	0.24	0.14	0.17	N.D.	0.14	0.22
WC	6,164.2	207.2	738.3	15.5	62.0	321.2
Chemical precipitation process	0.40	0.32	0.24	N.D.	0.26	0.30
Ferrite process	0.47	0.25	0.34	N.D.	0.31	0.12
Materialization process[a]	0.54	0.19	0.41	N.D.	0.28	0.17
Effluent standards	3.0	2.0	1.0	0.03	1.0	5.0

Notes: [a]$Fe^{3+}/M_t^{2+} = 4$ and $Fe^{2+}/M_t^{2+} = 4$.

Table 4
Leaching concentration of the residues for the chemical precipitation process, ferrite process, and materialization process

Item	Products	Cu (mg/L)	Cr	Ni	Cd	Pb	Zn
WA	Chemical precipitation process	187.5	127.8	64.0	0.2	12.8	23.6
	Ferrite process	65.8	32.7	2.1	0.1	22.1	0.2
	Materialization process[a]	3.1	1.9	1.1	0.2	0.8	0.6
WB	Chemical precipitation process	197.8	80.4	44.6	0.1	15.4	17.1
	Ferrite process	120.5	15.3	1.7	0.1	8.9	0.8
	Materialization process[a]	4.9	1.5	1.8	0.2	0.4	1.0
WC	Chemical precipitation process	378.8	69.8	64.8	0.3	7.8	15.0
	Ferrite process	135.9	18.6	1.5	0.1	4.3	0.2
	Materialization process[a]	5.1	2.0	1.1	0.1	0.5	0.4
TCLP limitation		15.0	5.0	–	1.0	5.0	–

Notes: [a]$Fe^{3+}/M_t^{2+} = 4$ and $Fe^{2+}/M_t^{2+} = 4$.

according to the Cu-potential pH [17]. In contrast, in the conventional co-precipitation method, the pH value in suspension increased gradually from about 2.5–10.5 as the NaOH solution was added. Therefore, Fe^{3+} ions was precipitated first as the pH reached 2.1, and then, Cr^{3+}, Cu^{2+}, Ni^{2+}, Pb^{2+}, Cd^{2+}, and Zn^{2+} ions were precipitated simultaneously at pH greater than 4.5 due to the small difference in their precipitation pH range. The results of real wastewater and filtrate from the chemical precipitation process, ferrite process and materialization process are depicted in Table 3. It is obviously that all

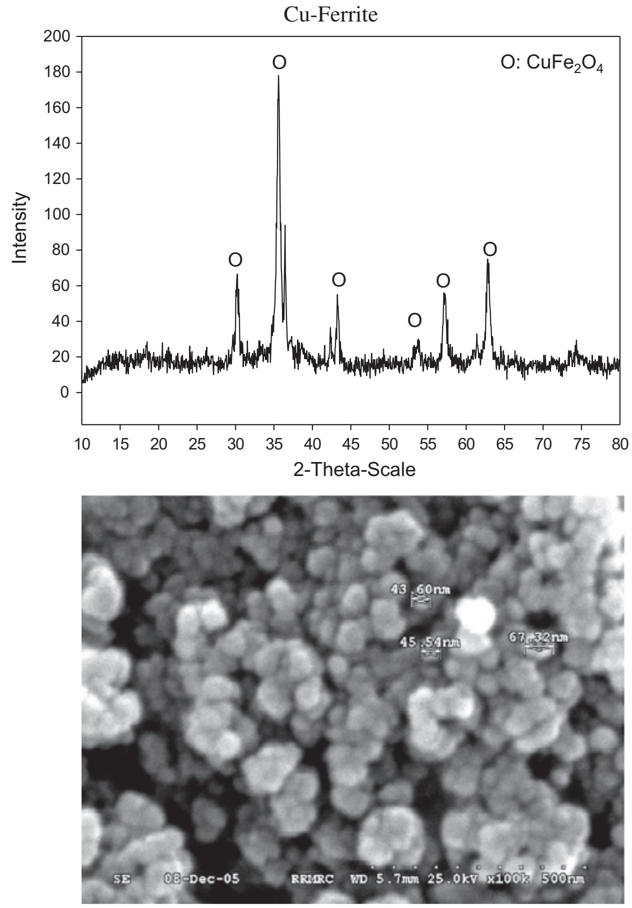

Fig. 4. XRD pattern and SEM image of magnetic materials after the materialization process from real wastewater.

heavy metal concentrations of the filtrate from chemical precipitation process, ferrite process and materialization process lie below the regulatory effluent standards stipulated by the EPA of Taiwan.

The results of TCLP are listed in Table 4. The heavy metal leaching concentrations of the residues from the materialization process are below the regulatory standards stipulated by the EPA of Taiwan. The XRD pattern implies that the materialization process is able to treat high copper-containing wastewater and produces non-toxic materialization products (Fig. 4). The nonferrous metals can be immobilized in the spinel structure. The XRD pattern of the solid products matches the pattern of $CuFe_2O_4$ and magnetite (Fe_3O_4) and can be categorized to be the spinel structure. The high intensities of the reflections indicate the high crystalline structure of the magnetic material.

3.3. Characterizations of magnetic materials

The SEM image demonstrated that the size of the magnetic material particles was 45–70 nm (Fig. 4). The saturated magnetization (Ms) and the BET analysis are shown in Table 5. It is obviously that the Ms and BET of magnetic materials after the materialization process is better than that after the ferrite process. The Ms would increase when the ratio of Fe^{2+}/M_t^{2+} increases. However, the higher Fe^{2+}/M_t^{2+} ratio might cause greater consumption of Fe^{2+}. Moreover, the difference of BET was lower in different Fe^{2+}/M_t^{2+} ratios. Thus, the Fe^{3+}/M_t^{2+} ratio of 4 and Fe^{2+}/M_t^{2+} ratio of 4 are suggested for the further application to ensure the leaching of heavy metals from magnetic materials is lower than the TCLP limitation.

4. Conclusions

The inverse mixing method of homogenization process seems more suitable for the formation of ferrite, since the formation of CuO and α–Fe_2O_3 in ferrite was prevented effectively. It is obviously that the heavy metal ions concentrations of the filtrate from the chemical precipitation process, ferrite process and materialization process lie below the regulatory effluent standards. However, the leaching concentrations (TCLP test) of the residues from the materialization process are below the regulatory standards stipulated by the EPA of Taiwan. The XRD pattern of the solid product matches the pattern of Cu-ferrite ($CuFe_2O_4$) and magnetite (Fe_3O_4). The main advantages of high copper-containing wastewater by the materialization process here are reducing disposal cost, creating reclamation profits, and producing the stable and environ-

Table 5
Ms and BET of magnetic materials from real wastewater

Items	Ms (emu/g)			BET (m^2/g)		
	WA	WB	WC	WA	WB	WC
Ferrite process ($Fe^{2+}/M_t^{2+} = 4$)	9.64	10.35	10.52	55.83	69.74	142.24
Materialization process ($Fe^{3+}/M_t^{2+} = 4$, $Fe^{2+}/M_t^{2+} = 4$)	17.71	18.18	20.36	56.12	78.64	156.74
Materialization process ($Fe^{3+}/M_t^{2+} = 4$, $Fe^{2+}/M_t^{2+} = 8$)	26.56	30.64	31.43	59.58	82.92	160.87

mentally friendly magnetic materials. On this basis, it is necessary to emphasize that high copper-containing magnetic material compounds may have an economically viable applications, such as magnetic materials, absorbents for toxic gases (hydrogen sulfide), frits, and pigments, etc.

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