Thermal behaviors and heavy metal vaporization of phosphatized tannery sludge in incineration process

TANG Ping[1], ZHAO Youcai[1,*], XIA Fengyi[2]

1. State Key Laboratory of Pollution Control and Resource Reuse, School of Environmental Science and Engineering, Tongji University, Shanghai 200092, China. E-mail: tpshe@163.com
2. Department of Life and Environmental Science, Wenzhou University, Wenzhou, Zhejiang 325007, China

Abstract

The high concentration of heavy metal (Cu, Cr, Zn, Pb) in tannery sludge causes severe heavy metal emissions in the process of incineration. In the present investigation, the tannery sludge was treated with 85% phosphoric acid before the incineration process in the tube furnace to control the heavy metal emissions. The thermal behavior and heavy metal vaporization of pre-treated tannery sludge were investigated, and X-ray diffraction (XRD) and scanning electron microscopy (SEM) analysis were also implemented to elucidate the chemical mechanisms responsible for the thermal behavior and weakening of heavy metal vaporization of pre-treated tannery sludge. The results obtained show that the differences in thermal behaviors between untreated and pre-treated tannery sludge are caused by the reaction of phosphoric acid and calcium carbonate. The vaporization percentage of heavy metal decreased efficiently with the increasing volumes of H_3PO_4, which indicated the important thermal stability of the water-insoluble metallic phosphates ($Ca_{18}Cu_3(PO_4)_{14}$, $Ca_9Cr(PO_4)_7$, $Ca_{19}Zn_2(PO_4)_{14}$, $PbMgP_2O_7$) formed during tannery sludge phosphatation.

Key words: tannery sludge; thermal behavior; heavy metal vaporization; phosphatation

Introduction

Tannery sludge contains a considerable amount of organic matter, heavy metals, as well as pathogenic or disease-causing micro-organisms, which are harmful to the environment and human health. Hence, tannery sludge treatment has become a serious environmental issue. At present, the conventional options for sewage sludge disposal, such as fertilizer and soil amendment material for the land and landfill, are widely applied. However, heavy metals in the tannery sludge often outweigh the soil's heavy metal content, and the application of sludge can indeed increase the concentration of heavy metals in the agricultural soil and affect the crop production owing to uptake. Moreover, landfilling also poses a potential environmental hazard, including the production of odor and release of methane gas, as well as contamination of groundwater by leachate. It is, therefore, of great significance to find a proper way to dispose tannery sludge to avoid secondary pollution.

Incineration is an alternative solution for disposal of sludge containing heavy metals, which has become the major disposal route for sludge in the European Union. Moreover, it is expected that the role of incineration will increase in the future, since it results in a large reduction of sludge volume to a small quantity of ash, which

accounts for only 10% of the volume of mechanically dewatered sludge; the thermal destruction of toxic organic constituents and pathogens; and the recovery of the flue gas energy (Werther and Ogada, 1999).

Although incineration has more advantages than other disposal methods, the disadvantages such as producing secondary pollutants (dioxin, NO_x, SO_2, and heavy metals) may inhibit the application of the technology. At present, most pollutants, except for heavy metals, can be effectively removed by Air Pollution Control Devices (Han *et al.*, 2008). Moreover, the heavy metals will badly endanger human health and the environment after entering the atmosphere. Metal emissions from sludge incineration process are currently of great environmental and regulatory concern because of their toxicity. Therefore, until now, the topic on how to control emissions of heavy metals during the incineration process has drawn more and more attention from scientists.

Recently, studies have been conducted on how to convert heavy metal present in sludge into chlorides and sulfates or other compounds to transfer heavy metals in fuel as much as possible into the flue gas, which can be captured by condensation on the filter-ash or in the scrubber water and then recovered as mineral ores rich in metals (Hoffmann *et al.*, 2008; Sun *et al.*, 2004; Zorpas *et al.*, 2001); thus, the heavy metal can be reused and emissions can be eliminated. Moreover, some effort has been made in this

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* Corresponding author. E-mail: zhaoyoucai@mail.tongji.edu.cn.

innovative concept (Wochele and Stucki, 1999). However, it is evident that more volatile metals such as Hg, Pb, and Cd are significantly present in the stack emission, despite the scrubber stages, but in line with the volatility of the heavy metals (Velden *et al*., 2008). Furthermore, it is reported that lithophilic metals such as Fe, Cu, Cr, and Al remained mainly in the bottom ash (BA) during municipal solid waste incineration (MSWI) (Feng *et al*., 2007) and about two thirds of Pb and Zn was found in the BA despite their high volatility (Jung *et al*., 2004).

In addition, several efforts have also been made to decrease the heavy metal emissions during the incineration process by means of stabilization and absorption. Yao and Naruse (2005) and Naruse *et al*. (2003) reported that the addition of kaolinite powders into sewage sludge could capture Pb and Cd compounds, because of the reaction of Pb and Cd compounds with the kaolinite during combustion. Cheng *et al*. (2001) investigated the effects of limestone, $CaSO_4$, bauxite, kaolinite, and CaO on the emission of trace elements, SO_2 and NO_x during coal combustion. Rio *et al*. (2007) also claimed that the addition of sodium bicarbonate and activated carbon is an efficient neutralizing agent for gaseous streams, which eliminates most of the heavy metal emissions. Hsieh *et al*. (2007) demonstrated insignificant volatilization of Cu during heating after stabilization of sludge with microwave.

Although chemical stabilization as one of the methods to decrease the heavy metal emissions has been studied intensively (Eighmy *et al*., 1998; Bournonville *et al*., 2004; Crannell *et al*., 2000), the effect of chemical stabilization on the decrease of heavy metal emissions has not been studied. Therefore, in this study, the vaporization of heavy metals and the thermal behaviors in the incineration of sludge pre-treated with 85% phosphoric acid are studied. Moreover, the mechanisms and the reaction products resulting from chemical stabilization of tannery sludge with soluble are also investigated.

1 Materials and methods

1.1 Materials

Tannery sludge was collected from Wenzhou, China from March 2006 to April 2006. The water contained in tannery sludge was extracted by mechanical dewatering methods and the moisture content of the samples was decreased to about 73%. The incoming tannery sludge was characterized by a proximate and an ultimate analysis. The samples were stirred uniformly and then dried at 65°C. The metal concentrations of the tannery sludge were determined (Table 1).

Phosphatation was carried out by adding phosphoric acid (85% H_3PO_4) to tannery sludge containing sufficient water (50%) to exhibit the rheology of a thixotropic slurry, thereby achieving the best compromise between minimum water input and optimal contact between the reactant and the solids (Piantone *et al*., 2003), and this mixing process was followed by the formation of foam. After mixing sludge with H_3PO_4, the phosphatized sludge was put in a

Table 1 Analysis of the tannery sludge

Parameter	Tannery sludge
Proximate analysis (incoming tannery sludge) (wt.%)	
Moisture	73.25
Ash	13.11
Volatiles	13.61
Fixed carbon	0.03
Lower heating value (dry basis) (kJ/kg)	5,346.2
Ultimate analysis (dry basis) (wt.%)	
C	16.591
H	2.898
N	2.343
S	0.623
Heavy metal analysis (dry basis) (mg/g)	
Zn	0.38
Pb	0.28
Cd	0.003
Fe	9.12
Mn	0.50
Cr	20.49
Cu	0.07
Al	7.15

drying bed to avoid moisture interference.

1.2 Apparatus and experimental procedure

The apparatus used in this study was composed of an electric-heated tube furnace and an air supply (Fig.1). The heart of the furnace was a quartz tube burner of 600-mm length and 40-mm inner diameter. A porcelain boat moved by quartz rod was designed to feed the sludge into the combustion chamber. The combustion temperature at the center inside the burner tube was monitored by two thermocouples and controlled by a programmed temperature controller. Finally, the oven was connected to two impingers filled with 5% HNO_3, allowing the collection of the vaporized fraction of the heavy metals to be determined.

Experiments were carried out by combustion of 2–3 g wastes, put in a porcelain boat pushed into a quartz tube. During the incineration experiments, the air flow rate was fixed at 120 L/h. The influence of temperature (500–900°C) and phosphoric acid addition on the vaporization of heavy metals (Pb, Zn, Cu, and Cr) was studied.

1.3 Analysis methods

The bottom ash and porcelain boat were weighted after the incineration test was complete and the boat was pulled out and cooled down, and the weight of the bottom ash was calculated by subtracting the weight of the boat from the total weight. Then, the ash was ground to fine
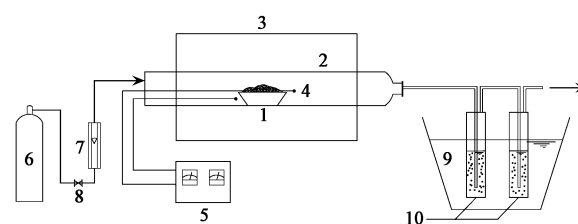

Fig. 1 Laboratory tube furnace. (1) porcelain boat; (2) quartz tube; (3) tube furnace; (4) thermocouple; (5) thermo controller; (6) air supply; (7) flow meter; (8) regulating valve; (9) water; (10) impingers (5% HNO_3).

powder, from which 400 mg was weighed and digested in concentrated HF, H_2O_2, and HNO_3 in pressure microwave. The concentrations of heavy metals in the aqueous solution were then analyzed by ICP (Optima2100DV, Perlin-Elmer, USA). Two parallel tests were carried out, and for each sample, two parallel digestions were conducted and their average values are obtained. The vaporization percentage (*V*, %) was calculated according to:

$$V = \frac{M_S - M_B}{M_S} \times 100\% \tag{1}$$

where, M_S was the initial metal content in sludge sample; and M_B was the final metal content in the bottom ash.

Under the test condition, the overall mass balance closure of heavy metals during combustion was in an acceptable manner, although the balance for all the heavy metals was not complete.

Powder XRD patterns of any possible crystalline phases and metal species formation were recorded on a D/max-γ βX-ray diffractometer (X'PERT MPD, Philips, Holand) with 50 mA and 40 kV, Cu K_α radiation. The thermal behaviors of samples were examined by DTA-TGA using a ZRY-2P simultaneous DTA-TGA analyzer (Q600sdt, TA, USA) while the samples were heated at a rate of 20°C/min from 50 to 900°C in air. Samples were weighed from 4 to 10 mg in mass, and were put into a Pt-Rh crucible with 20 taps. All curves were evaluated using the TA-instruments software. The mineral morphology images of bottom ash of untreated and pre-treated tannery sludge were obtained using ESEM (XL-30, Philips, Holand) operating with 30 kV accelerating voltage under high vacuum.

2 Results

2.1 Thermal behavior analysis

Thermogravimetric (TG) analysis provides a prior knowledge of the initial and final temperatures for the thermal degradation of sludge. The results of TG/DTG of tannery sludge are shown in Fig.2a. It can be seen from Fig.2a that the DTG curve of tannery sludge changes slightly relatively from 50 to 200°C and has 5.8% weight loss in TG analysis. Two obvious peaks are observed in the DTG curve from 200 to 400°C with 18.5% weight loss and from 660 to 780°C with 15.4% weight loss, respectively. After being finally held from 780 to 900°C, the rate of mass loss was approximately zero.

Figure 2b shows the TG and DTG measurements of tannery sludge pre-treated with 85% H_3PO_4, which accounts for 10% of dry basis of sludge (Sludge A). There is a small peak in the temperature range 120–220°C shown in the DTG curve of Fig.2b and the corresponding weight loss of the sludge is 5.3%. Two other obvious peaks are also observed in the DTG curve from 240 to 400°C with 14.0% weight loss and from 660 to 780°C with 11.5% weight loss, respectively.

Figure 2c shows the TG and DTG measurements of tannery sludge pre-treated with 85% H_3PO_4, in which H_3PO_4 accounts for 20% of the dry basis of sludge (Sludge B). For Sludge B, the maximum weight loss is
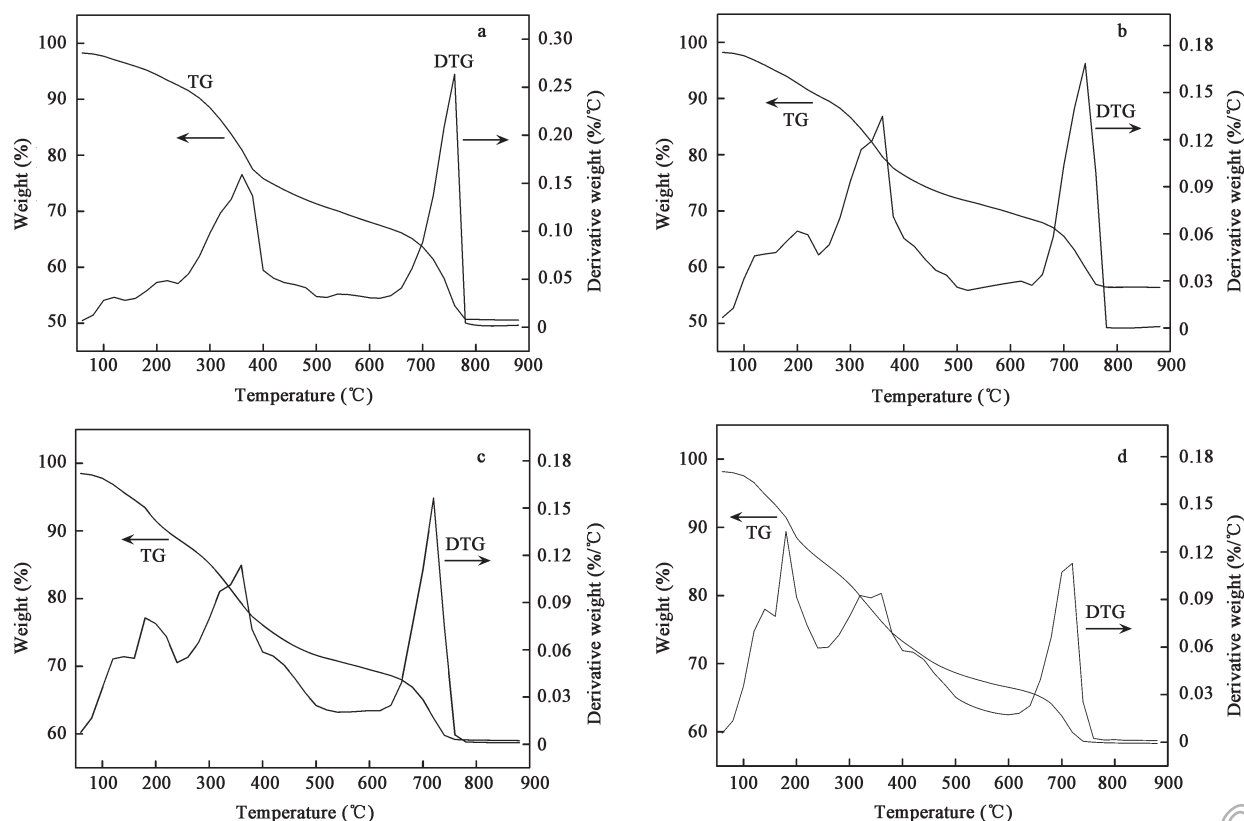

Fig. 2   TG and DTG curves of tannery sludge at the heating rate of 20°C/min. (a) tannery sludge; (b) Sludge A; (c) Sludge B; (d) Sludge C.

observed in the temperature range 140–440°C. In this temperature range, two different peaks are observed in the DTG curve. The first peak occurs in the temperature range 120–240°C and the corresponding weight loss is 6.8%, while the second occurs in the temperature range 240–440°C and the corresponding weight loss is 13.9%. Another main decomposition peak was observed between 660 and 780°C with the weight loss of 8.9% in TG analysis.

Figure 2d shows the TG and DTG measurements of tannery sludge pre-treated with 85% H_3PO_4, in which H_3PO_4 accounts for 30% of the dry basis of sludge (Sludge C). The main weight loss of Sludge C generally occurs over the temperature of 120–440°C. In this temperature, two distinct peaks are also observed as Sludge B. The first peak occurs in the temperature range 120–240°C with weight loss of 11.0%, while the second occurs in the temperature range 240–440°C with weight loss of 14.5%. Moreover, another peak in the DTG curve starts from 660°C and ends at 740°C with weight loss of 6.5%.

2.2 Vaporization of heavy metal

Figure 3 show the relationship between the amount of phosphoric acid and the vaporization percentages of Zn, Pb, Cr, and Cu at 600 and 800°C. At 600°C, the vaporization percentage decreases rather slightly from 24% to 7% for Zn, from 21% to 12% for Pb, and from 17% to 11% for Cu when the phosphoric acid content increases from 0 to 30%, while the vaporization of Cr can be considered nearly unchanged at around 5%. At 800°C, the vaporization percentage decreases significantly from 43% to 11% for Zn, from 48% to 10% for Pb, and from 32% to 9% for Cu, while the vaporization percentage of Cr decreases slightly from 12% to 7%.

2.3 Crystalline phase and chemical composition analysis

The crystalline phase of the bottom ash of Sludge A, Sludge B, and Sludge C is as shown in Fig.4. The major crystalline phase of Sludge A at 600°C is $CaCO_3$, $Ca_5(CrO_4)_3O_{0.5}$, and $Ca_2Cr_2O_5$. As the content of H_3PO_4 increases, $Ca_2P_2O_7$, $Ca_{19}Cu_2(PO_4)_{14}$, $Ca_{19}Zn_2(PO_4)_{14}$, and $Ca_9Cr(PO_4)_7$ become the major crystalline phase as shown in Fig.4.
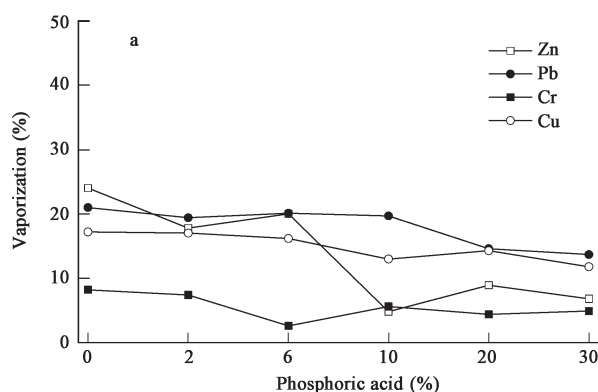
Figure 5 shows the X-ray diffraction pattern of the bottom ash of Sludge A, Sludge B, and Sludge C at 800°C. The pattern consists of strong intensity peaks corresponding to CaO and weak peaks of $Ca_2P_2O_7$, $Ca_{19}Cu_2(PO_4)_{14}$, $Ca_{19}Zn_2(PO_4)_{14}$, and $Ca_9Cr(PO_4)_7$. When the content of H_3PO_4 rises, the peaks of $Ca_2P_2O_7$, $Ca_{19}Cu_2(PO_4)_{14}$, $Ca_{19}Zn_2(PO_4)_{14}$, and $Ca_9Cr(PO_4)_7$ become sharper and these crystals become the major phase in the bottom ash.

2.4 Scanning electron microscope (SEM) analysis

Figure 6 shows the morphology of bottom ash of tannery sludge and Sludge A at 800°C. SEM analysis revealed the differences in the gross microstructure between the bottom ash of untreated tannery sludge and pre-treated tannery sludge with phosphoric acid. The SEM micrograph of bottom ash of untreated tannery sludge shows the presence of floc-like material as seen in Fig.6a. This floc-like material is believed to be the crystal grain of CaO. The micrograph of the bottom ash of pre-treated tannery
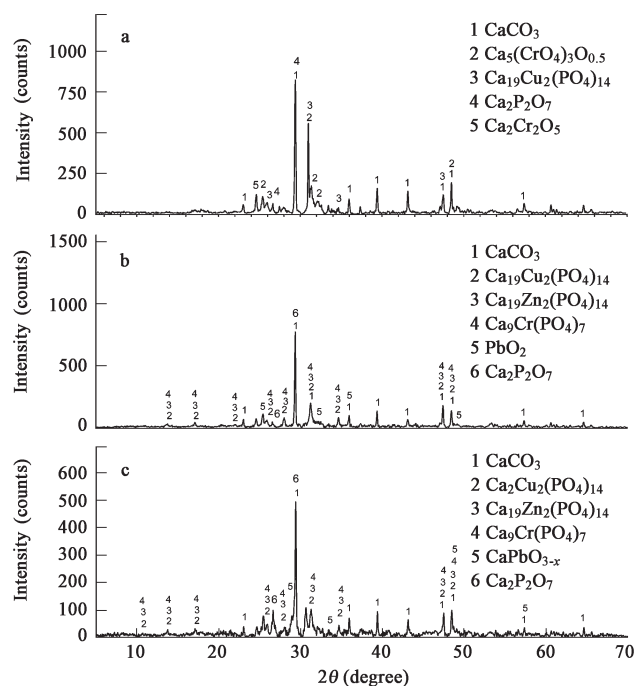
Fig. 4 X-ray diffraction patterns of the bottom ashes of Sludge A (a), Sludge B (b), and Sludge C (c) at 600°C.
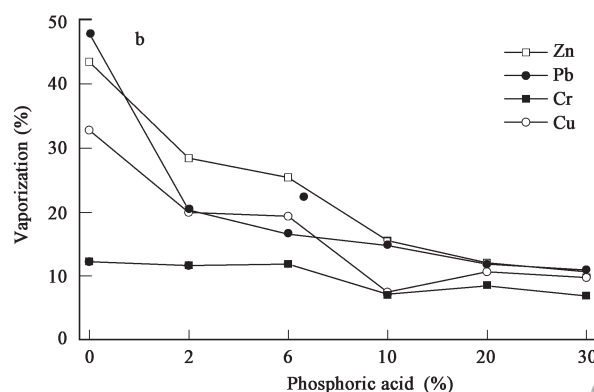
Fig. 3 Vaporization percentages of heavy metal during thermal treatment of phosphated sludge at 600°C (a) and 800°C (b).
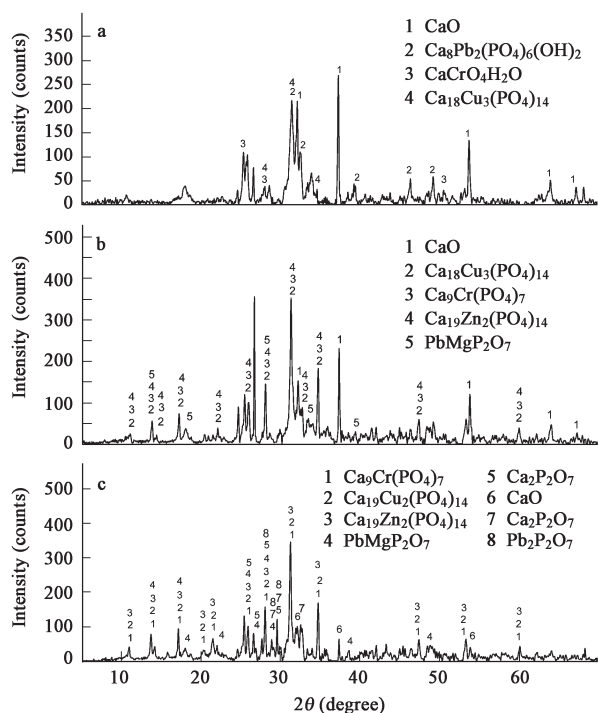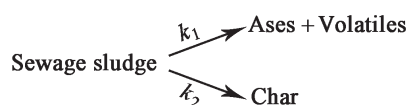
Fig. 5 X-ray diffraction patterns of the bottom ashes of Sludge A (a), Sludge B (b), and Sludge C (c) at 800°C.
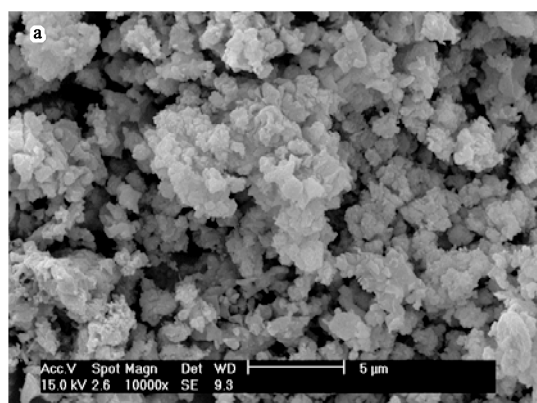
sludge (Sludge A) shows irregularly shaped mineral crystal grain covered on the CaO crystal (Fig.6b), which is believed to be the apatite-like mineral phase corresponding to $Ca_8Pb_2(PO_4)_6(OH)_2$ and $Ca_{18}Cu_3(PO_4)_{14}$ peaks in the XRD graph (Fig.5).

3 Discussion

The reaction scheme of sludge pyrolysis is very complex. Ahuja *et al.* (1996) proposed the following global reaction scheme for the pyrolysis of sewage sludge.

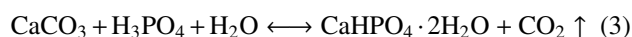As seen in Fig.2a, the pyrolysis of tannery sludge

took place in two stages. Prior to the first stage, the TG diagrams for tannery sludge show a slight decrease in weight at a temperature in the range of 50–220°C, owing to the evaporation of adsorbed water. The most abundant release of volatile matter was observed in the first stage. In this stage, the peak of DTG curve had a shoulder at 220°C, which can be attributed to the decomposition and devolatilization of less complex organic structures, which is a small fraction, while the peak was caused by the decomposition of more complex organic structures corresponding to a larger fraction.

In the second stage of sludge pyrolysis, decomposition of inorganic materials mainly took place. According to the literature, pure $CaCO_3$ showed an endothermic peak and the corresponding decrease in weight at 720°C (Geysen *et al.*, 2006). Thus, the weight loss in the second stage is mainly attributed to the decomposition of $CaCO_3$ following the reaction:
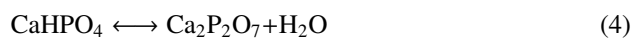
$$CaCO_3 \longleftrightarrow CaO + CO_2 \qquad (2)$$

When tannery sludge were mixed with phosphoric acid, $CaCO_3$ that contained in tannery sludge can react with H_3PO_4 following the reaction:

$$CaCO_3 + H_3PO_4 + H_2O \longleftrightarrow CaHPO_4 \cdot 2H_2O + CO_2 \uparrow \quad (3)$$

which lead to the formation of foam.

Thus, for Sludge A, Sludge B, and Sludge C, the peak that started from 120°C and ended at 240°C was a result of the dehydration of crystal water of $CaHPO_4 \cdot 2H_2O$ and the evaporation of absorbed water. Compared with the DTG curves of Sludge A, Sludge B, and Sludge C, the more the amount of H_3PO_4 was mixed, the higher was the peak. Furthermore, the DTG curves of Sludge A, Sludge B, and Sludge C showed peaks at 360°C having a shoulder at 420°C. The peaks were a result of the decomposition of organic matter while the shoulders can be assigned to the decomposition of $CaHPO_4$ following the reaction:

$$CaHPO_4 \longleftrightarrow Ca_2P_2O_7 + H_2O \qquad (4)$$

which can also be proved in the crystalline phase analysis (Figs.4 and 5). Owing to the reaction with H_3PO_4, the amount of $CaCO_3$ decreased, which led to weaken the
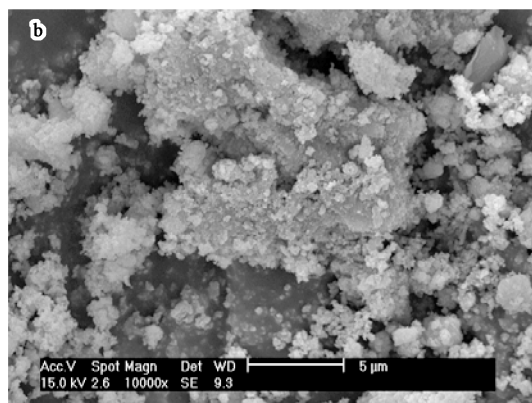

Fig. 6 SEM images showing the bottom ash of tannery sludge (a) and Sludge A (b) at 800°C.
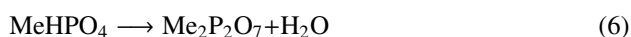
peaks of the DTG curves of Sludge A, Sludge B, and Sludge C at 720°C.

Although most of the phosphoric acid reacts with $CaCO_3$, addition of phosphoric acid also allows the formation of other insoluble metal phosphates as shown in Figs.4 and 5, which makes heavy metals be trapped and fixed into stable crystal phases following the general expression:
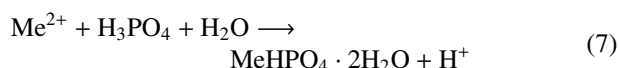
$$Me^{2+} + H_3PO_4 + H_2O \longrightarrow MeHPO_4 \cdot 2H_2O + H^+ \qquad (5)$$

where, Me stands for Pb, Cu, Zn, Mg, Fe.

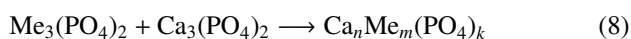In the process of incineration, the heavy metal crystal phases are also decomposed following the reaction:

$$MeHPO_4 \longrightarrow Me_2P_2O_7 + H_2O \qquad (6)$$

Therefore, $Mg_2P_2O_7$, $Pb_2P_2O_7$ also can be identified by XRD as the minor crystalline phase besides the major phase of $Ca_2P_2O_7$ and $CaCO_3$. Owing to the low content of phosphoric acid, most Cr was stabilized as the compound of $Ca_2Cr_2O_5$ and $CaCrO_4H_2O$ as shown in Figs. 4a and 5a.

Besides this, owing to the presence of excessive $CaCO_3$ in tannery sludge, the following reaction also occurred, which was in agreement with the minor crystal phases in the bottom ash of Sludge A, Sludge B, and sludge C identified by XRD, such as $Ca_8Pb_2(PO_4)_6(OH)_2$, $Cu_5(PO_4)_2(OH)_4$, and $Ca_5Al_{12}(PO_4)_8(OH)_{22}$.

$$Me^{2+} + H_3PO_4 + H_2O \longrightarrow \\ MeHPO_4 \cdot 2H_2O + H^+ \qquad (7)$$

where, Me stands for Ca, Al, Mg, Cu, Cr, Pb, Zn, etc.

When the addition of phosphoric acid increased, the product of phosphate of metals in the tannery sludge also increased. These phosphates of metals were apt to bond with each other as complex in the combustion process because of the following reaction:

$$Me_3(PO_4)_2 + Ca_3(PO_4)_2 \longrightarrow Ca_nMe_m(PO_4)_k \qquad (8)$$

thus, heavy metals, including Cr, were fixed into more stable crystal as apatite-like minerals as shown in Figs.4b and 4c and Figs.5b and 5c. Scanning electron microscope studies also provided conclusive proof on the presence of apatite-like mineral in the bottom ash, which can fix heavy metals to avoid transfer to the environment. However, most of Pb is stabilized as the compounds $PbMgP_2O_7$, $Pb_2P_2O_7$ according to XRD analysis.

After being treated by phosphoric acid, the heavy metals showed low vaporization in general agreement with the formation of insoluble phases, with Pb showing the highest value of only 17% (Fig.3), which was probably because the phosphate of Pb was not easy to bond with other phosphate to form the considerably stabler complex. For Cu, Cr, and Zn, the vaporization percentages seemed to be independent of temperature and were less than 15% at 600 and 800°C, which shows a good correspondence with the XRD and SEM analysis. Meanwhile, it can be seen form the Fig.3 that 10% is the optimal dosage to control the heavy metal emissions. Moreover, the stabilization agent also reduced the leachability of several divalent cations (Pb^{2+}, Cu^{2+}, Zn^{2+}, Cd^{2+}) even under aggressive leaching conditions (Crannell *et al*., 2000).

4 Conclusions

The study deals with the detailed investigation on the thermal behaviors and heavy metal vaporization of tannery sludge pre-treated with 85% phosphoric acid. In the TGA experiments, three degradation steps were observed for pre-treated tannery sludge, which include the decomposition of $CaHPO_4 \cdot 2H_2O$, the pyrolysis of organic matter, and the decomposition of $CaCO_3$. In the presence of H_3PO_4, the vaporization percentage of heavy metal in tannery sludge decreased efficiently with the increasing volumes of H_3PO_4. The chemical mechanisms governing the volatilization behaviors of heavy metals in pre-treated tannery sludge in the incineration process were proposed through XRD and SEM analysis. Pre-treatment of tannery sludge with proper amount of H_3PO_4 is considered among the measures of controlling heavy metal emissions in the process of tannery sludge incineration.

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