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Combustion characteristics of tannery sludge and volatilization of heavy metals in combustion*

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Abstract: Incineration is considered one of the most readily available techniques for sewage sludge disposal, including tannery sludge, which often contains significant amounts of volatile heavy metals. The combustion characteristics and kinetic analysis of tannery sludge were investigated using thermogravimetric analysis (TGA) at a heating rate of 30 °C/min in 50–950 °C. In addition to confirming that tannery sludge has a high content of volatile material and ash, it was further discovered that almost all the zinc (Zn) in tannery sludge is volatilized at 900 °C. The degree of volatilization for heavy metals at 900 °C followed the order of Zn>Cd>Cu>Mn>Pb>Cr. Moreover, the volatilization of these heavy metals increased with temperature. It is thus concluded that, to avoid heavy metal volatization during incineration disposal, 800 °C is a reasonable incineration temperature.

Key words: Tannery sludge, Combustion, Heavy metal volatilization, Thermogravimetric analysis (TGA), Combustion kinetics
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1 Introduction

Tannery sludge, the essential by-product of the tanning industry, contains chromium in amounts high enough such that is classified as hazardous waste. Landfilling and ocean dumping are the simplest methods for disposal of this waste sludge, but there is an increasing call to cease these methods because of environmental consequences (Saxena and Jotshi, 1996). Waste sludge has been widely used as agricultural organic fertilizer, but application of heavy metal-contaminated sewage sludge significantly increases the risk of soil contamination with toxic metals contamination and metals transfer to freshwater and plants. The use of sewage sludge for agricultural applications is thus less appealing and less often used (Jensen and Jepsen, 2005; Toribio and Romanya, 2006; Dai *et al*., 2007).

Organic sludge has generally good combustion characteristics, and sludge incineration technology has reached a commercial level of operation (Mahesh *et al*., 2006). Incineration has been well recognized as the most available pretreatment technique for waste sludge before landfilling, because of the effective volume reduction and detoxification. In addition, waste energy can be recovered by incineration for economic and environmental benefits, and the ash residue may be also used in the ceramic and building material industries (Mahesh *et al*., 2006).

The combustion characteristics of sewage sludge have been investigated (Ferrasse *et al*., 2003), and two maxima patterns of volatile matter and fixed carbon were found in sludge combustion (Ogada and Werther, 1996; Magalhães *et al*., 2008). Temperature and residence time were found as the dominant parameters determining the volatilization of heavy

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metals. The formation of gaseous metallic compounds is enhanced by high temperature (Bakoglu *et al.*, 2003), but the gaseous compounds and volatile metals have little chance to evolve completely, due to the limited residence time for sludge combustion in the incinerator (Corella and Toledo, 2000; Marani *et al.*, 2003).

It was found that the amount of heavy metal partitioned to the fly ash will be enhanced by a higher content of chlorine; therefore, the removal of chloride from the fuel would reduce the formation of toxic heavy metal chlorides in fly ash (Wang *et al.*, 1999). Sulfur content in sludge also plays a role in depression of the metal evolution in sludge combustion (Marani *et al.*, 2003; Yao *et al.*, 2006). Most volatile metals are enriched in fine particles in combustion, whereas the addition of sorbent in sludge induces a shift of metals from fine particles to coarse particles (Yao *et al.*, 2004; Yao and Naruse, 2005). The speciation of the metals emitted is changed by co-combustion of sludge with coal due to an interaction between alkali metals derived from sludge and sulfur from coal. Vapor-to-solid phase partitioning of metals is controlled by surface reaction with active surface sites (Seames *et al.*, 2002), and the addition of sewage sludge to coal combustion leads to higher heavy metals emissions (Amand and Leckner, 2004; Lopes *et al.*, 2004; Miller *et al.*, 2004).

Though there is an increase for some metals in their contents in bottom ashes compared to the original sewage sludge's content level, no higher leachability and/or ecotoxicity is apparent in those ashes, which suggests that there could be opportunities for their further use (Lopes *et al.*, 2003). After the elimination of the organic matter in sludge by combustion, the residue has been successfully tested as raw material for ceramic industry (Abreu and Toffoli, 2009), and the leachability studies on those products revealed that the concentration of metal present in the leachate meet the demand of the standards prescribed (Swarnalatha *et al.*, 2006).

China's leather production accounted for one fifth of global production in 2007. Therefore, a large amount of tannery sludge is generated every year, and it is necessary for safe disposal of this waste. Although incineration is the most available technique for disposal of tannery sludge, there is little literature which focuses on the combustion characteristics and volatilization of heavy metals in tannery sludge. The combustion characteristics of tannery sludge was investigated in this study using thermogravimetric analysis (TGA) in the temperature range of 50–950 °C, and the emission potential of heavy metals during isothermal combustion in a tube furnace was also studied. The experiments focused on six targeted metals: Cr, Cu, Cd, Mn, Pb and Zn. The aim of this paper is to present observations on the volatilization tendency of metals during tannery sludge combustion.

2 Experiment

2.1 Sample preparation

The sample materials in this study were sludge from a tanyard in a city of Zhejiang Province, China. After being dried at 105 °C for 3 h, the sludge was crushed and pulverized before further analysis. Proximate analysis of tannery sludge was conducted to obtain moisture level, ash content, volatile content and fixed-carbon content. Ultimate analysis of the dried samples for carbon, hydrogen, nitrogen, and sulfur (%, w/w) were also determined.

The detailed characterization of tannery sludge is shown in Table 1, indicating that there is a large amount of ash and volatile matter in tannery sludge.

Table 1 Properties of tannery sludge

Property		Value
Ultimate analysis (%, w/w)	C	25.4
	H	1.7
	O	24.2
	N	1.1
	S	0.5
	Cl	0.7
Proximate analysis (%, w/w)	Moisture	1.5
	Ash	45.6
	Volatiles	45.1
	Fixed carbon	7.8
Heavy metals analysis (mg/kg)	Cr*	0.83
	Cu	65
	Mn	576
	Pb	20
	Cd	50
	Ni	40
	Zn	260
Higher heating value (MJ/kg)		8.5

* With the unit of (%, w/w)

The contents of heavy metals in tannery sludge are also given in Table 1. The X-ray diffraction (XRD) analysis of the original sludge is presented in Fig. 1, and $CaCO_3$ and SiO_2 were found to be the main inorganic components.

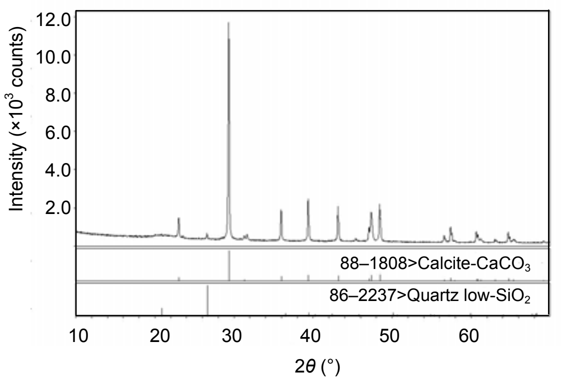
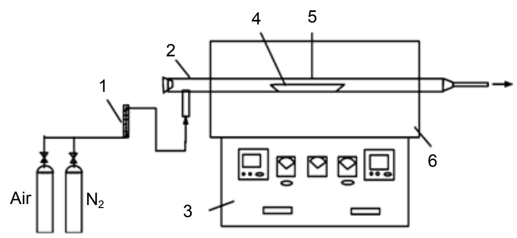


Fig. 1 XRD-spectrum of the raw tannery sludge sample
The spectrums in the bottom were the standard ones of $CaCO_3$ and SiO_2

2.2 Method

A Nicolet Nexus 670 spectrometer (USA) and a Mettler Toledo TGA/SDTA851^e thermo analyzer (Switzerland) were used in this study, coupled by a Thermo-Nicolet TGA special connector. Air was used as oxidizer with a flow rate of 60 ml/min. A heating rate of 30 °C/min was applied starting at 50 °C, with a final temperature of 950 °C. The sample material (about 15 mg) was used in TGA.

The contents of heavy metals in the original sludge and their residue after being combusted at different final temperatures in a tube furnace were determined in this study (Fig. 2). When the quartz tube reaches the set temperature, the sample (about 1.5 g) paved on the quartz boat was pushed into the high temperature zone of the tube furnace quickly. An air flow rate of 500 ml/min was used, and the residence time of combustion was 25 min for complete reaction.



1: flow meter; 2: quartz tube; 3: temperature controller; 4: quartz boat; 5: thermocouple; 6: tube furnace

Fig. 2 Experimental systems of tube furnace reactor

The digestion of original sludge and residue generated from the tube furnace was accomplished using microwave digestion according to standard methods (US EPA3050, 1992). Contents of Cu, Mn, Pb, Cd and Zn were determined by atomic absorption spectrometer (AAS) (SOLAAR969, Thermo Spectronic, USA), and the content of Cr was determined by inductively coupled plasma atomic emission spectroscopy (ICP-AES) (IRIS Intrepid II XSP, Thermo Spectronic, USA), following microwave sample acid digestion (100 mg of solid sample in 6 ml HNO_3, 1 ml HF, 4 ml $HClO_4$; T=548 K, and P=5×10^5 Pa).

3 Results and discussion

3.1 Thermogravimetric data analysis

The thermogravimetry and differential thermogravimetric (DTG) curves of tannery sludge combustion as a function of temperature at a heating rate of 30 °C/min are shown in Fig. 3. A total weight loss of 52.3% (w/w) was observed in combustion. The combustion process can be subdivided into four stages (Fig. 3). The first stage is a loss in weight upon drying the sample before 150 °C. The second stage from 150 to 550 °C is the thermal decomposition and combustion of volatile materials. More than 27% (w/w) of raw materials is lost in this stage. The third stage is the combustion of products charring from the sludge and fixed carbon in tannery sludge and calcination of the calcite before 790 °C, corresponding to a mass loss of about 24.4% (w/w) of the raw sample. The last stage is the high temperature decomposition of inorganic materials in sludge; the mass loss in the last stage is no more than 0.85% (w/w) of the original sludge sample. The mass loss at 790 °C was 51.5% (w/w), while the total mass loss at 950 °C was 52.3% (w/w). Therefore, the extent of combustion reaction at 790 °C is 98.5% and combustion of tannery sludge was nearly complete at 800 °C.

3.2 Kinetic analysis of tannery sludge combustion

Kinetics analysis is an effective method for evaluating the thermal stability of materials in combustion. In this study, it is assumed that the process of combustion was subdivided into two steps (low and high temperatures). The degree of combustion reaction is defined as

$$\alpha = \frac{M_{\mathrm{I}} - M_{\mathrm{T}}}{M_{\mathrm{I}} - M_{\mathrm{F}}}, \tag{1}$$

where M_{I} is the initial mass of sample, M_{T} is the real time mass in combustion, M_{F} is the final mass when combustion finished.

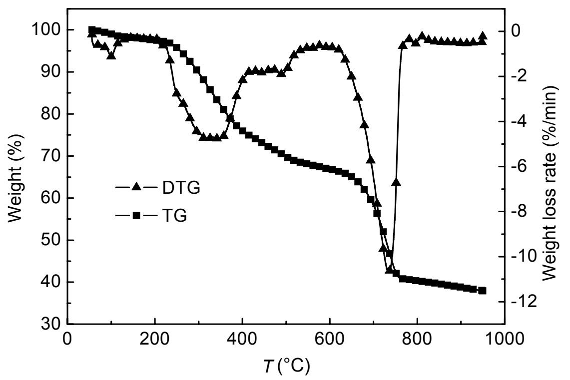


Fig. 3 TG and DTG analysis for tannery sludge combustion (heating rate of 30 °C/min)

The kinetics of combustion can be described as

$$\frac{\mathrm{d}\alpha}{\mathrm{d}T} = kf(\alpha), \tag{2a}$$

$$k = A\exp\left(-\frac{E}{RT}\right), \tag{2b}$$

$$f(\alpha) = 1 - \alpha, \tag{2c}$$

where k is the constant of reaction rate, T is the thermodynamic temperature (K), $f(\alpha)$ is the differential form of kinetic mechanism function, E is the activation energy, and A is a pre-exponential factor.

Eq. (2a) can be written as

$$\frac{\mathrm{d}\alpha}{f(\alpha)} = \frac{k}{\beta}\mathrm{d}T, \tag{3}$$

where β is the heating rate.

An integration function of Eq. (3) is given as

$$G(\alpha) = \int_0^{\alpha} \frac{\mathrm{d}\alpha}{f(\alpha)} = \frac{A}{\beta}\int_0^T \exp\left(-\frac{E}{RT}\right)\mathrm{d}T = \frac{AE}{\beta R}\int_{\infty}^{u} \frac{-\mathrm{e}^{-u}}{u^2}\mathrm{d}u. \tag{4}$$

As u can be defined as

$$u = \frac{E}{RT}. \tag{5}$$

Eq. (4) can be written as

$$G(\alpha) = \frac{AE}{\beta R}\int_{\infty}^{u} \frac{-\mathrm{e}^{-u}}{u^2}\mathrm{d}u = \frac{AE}{\beta R}\int_{\frac{E}{RT}}^{+\infty} \frac{\mathrm{e}^{-u}}{u^2}\mathrm{d}u = \frac{AE}{\beta R}\cdot P(u). \tag{6}$$

Because integration by parts of $P(u)$ is

$$P(u) \approx \frac{\mathrm{e}^{-u}}{u^2}\left(1 - \frac{2!}{u}\right). \tag{7}$$

Eq. (7) can be written as

$$\ln P(u) = -u + \ln(u-2) - 3\ln u. \tag{8}$$

The value range of u is $20 \le u \le 60$, then we can obtain:

$$v = \frac{u-40}{20}, \tag{9}$$

and thus Eq. (8) can be written as

$$\lg P(u) = -2.315 - 0.4567E/(RT). \tag{10}$$

Based on Eqs. (6) and (10), then

$$\lg[G(\alpha)] = \lg\left(\frac{AE}{\beta R}\right) - 2.315 - 0.4567\frac{E}{RT}. \tag{11}$$

The most probable kinetic mechanism function and parameters were investigated by the Satava method (Hu, 2001). Because $\lg[AE/(\beta R)]$ is independent of temperature T, the term of $\lg[G(\alpha)]$ varies linearly with $1/T$ for a probable kinetic function of $\lg[G(\alpha)]$, whereafter, both the activation energy E and pre-exponential factor A can be determined by the slope and intercept of the line.

Based on the data of TGA, $G(\alpha)$ in Eq. (11) was substituted by the conventional kinetic mechanism functions in integral form (Hu, 2001). Table 2 presents the best kinetic mechanism function for tannery sludge combustion and activation energy E and

Table 2 The most probable kinetic function and kinetic parameters in tannery sludge combustion

Temperature range (°C)	Function	Reaction mechanism	$G(\alpha)$	E (kJ/mol)	A (s^{-1})	r
195–410	Z-L-T	3D diffusion	$\left[(1-\alpha)^{-1/3}-1\right]^2$	111.04	1.34E+10	0.99
610–740	P-T	Self-catalyzed reaction	$\ln\left(\frac{\alpha}{1-\alpha}\right)$	134.47	2.75E+09	0.99

Z-L-T: Zhuralev-Lesokin-Tempelmen; P-T: Prout-Tompkins; r: correlation coefficient

pre-exponential factor A for the two steps of combustion for volatile matters and fixed carbon in tannery sludge.

The result shows that the combustion of volatile matter in tannery sludge is controlled by the 3D diffusion model and the Zhuralev-Lesokin-Tempelmen (Z-L-T) equation is the most probable kinetic function, while the combustion of fixed carbon is controlled by self-catalyzed reaction and the Prout-Tompkins (P-T) equation is the most probable kinetic function. It was observed that the activation energy for tannery sludge combustion is high.

3.3 Tannery sludge combustion in tube furnace

The TGA is always carried out in heating rate-controlling step. However, the real combustion process occurs under a high heating rate for wastes after they are fed into high temperature incinerators in commercial applications. It is obvious that the combustion in tube furnace has a greater heating rate than the combustion in TGA, so the combustion in the tube furnace is closer to the industrial condition. The mass loss during tannery sludge incineration in the tube furnace at different final temperatures is shown in Fig. 4.

A high speed mass loss stage in the temperature range of 600–800 °C and a low speed mass loss process in 500–600 °C and 800–900 °C were found in the tube furnace combustion. The mass loss with temperature in tube furnace incineration was larger than the value in TGA at the same temperature, mainly because of the long residence time in the tube furnace. But the variation trend of mass loss of tannery sludge with temperature in tube furnace combustion coincides well with the temperature evolution in TGA in the range from 500 to 900 °C. It can be concluded that the efficiency depends on the final temperature and residence time in tannery sludge combustion, while the parameter of heating rate has a weaker influence on efficiency of combustion. The mass of the residue at 800 and 900 °C is almost equivalent during combustion in the tube furnace; therefore, it is confirmed that 800 °C is sufficient for complete combustion of combustible materials in tannery sludge coupled with a long residence time, which coincides well with the results of the TGA study.

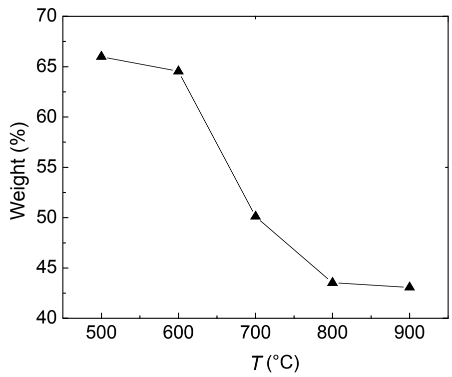


Fig. 4 Weight loss during tannery sludge combustion in tube furnace

3.4 Volatilization of trace elements in tannery sludge combustion

Base on the concentrations of heavy metals in the original tannery sludge and residue measured by ICP-AES and AAS, the volatilization of heavy metals in tannery sludge combustion was quantified. The degree of volatilization (V) (%, w/w) was calculated:

$$V=\frac{M_{S0}-M_{R}}{M_{S0}}\times 100\%, \tag{12}$$

where M_{S0} is the mass of metals in raw tannery sludge sample, and M_R is the mass of metals in combustion residue at different final temperatures. The degrees of volatilization according to temperatures for Cr, Cu, Mn, Pb, Cd, and Zn are shown in Fig. 5.

The degrees of volatilization of heavy metals at 900 °C followed the order of Zn>Cd>Cu>Mn>Pb>Cr. The volatilization of all the heavy metals increased with temperature, whereas there was a significant difference between them. There is a most notable enhancement of temperature on the volatilization of Zn from 7.8% at 500 °C to 96.1% at 900 °C. Furthermore, almost all the Zn was released at 900 °C, while the vaporization of Mn was almost unchanged (53.4% to 59.4%) in that temperature range. The weak volatilization tendency of Cr is shown in Fig. 5a, and the degree of volatilization of Cr is the least of the six heavy metals at each final temperature.

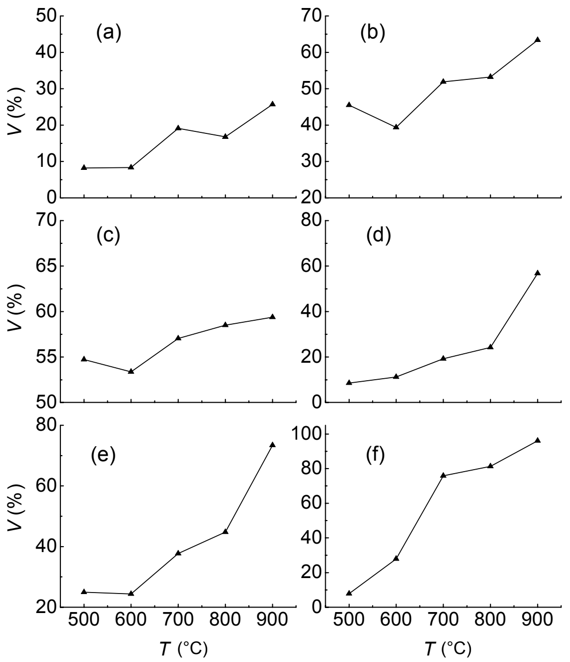


Fig. 5 Emission of heavy metals with temperature
(a) Cr; (b) Cu; (c) Mn; (d) Pb; (e) Cd; (f) Zn

The six heavy metals have been volatilized to certain degrees at 500 °C (Fig. 5). The volatilization of the metals at 500 °C in the tube furnace is shown in Fig. 6. As investigated in TGA, the thermal decomposition and combustion of volatile matter in tannery sludge occurs from 150 to 550 °C, so some of the metals in the organic species are emitted in this stage. The degrees of volatilization for heavy metals at 500 °C followed the order of Mn>Cu>Cd>Pb>Cr >Zn.

The content of chlorine in the original tannery sludge was also determined in this study (Table 1), as the high content of chlorine will have an important influence on the volatilization of metals in real systems for tannery sludge incineration.

The metals emission at different stages in tannery sludge combustion, the variation of volatilization of heavy metals at each temperature interval of 100 °C from 500 to 900 °C is shown in Fig. 7. There is no Cr, Cu, Cd or Mn emitted in the temperature stage of 500–600 °C, whereas 20% of Zn was emitted in this stage. The degree of volatilization at each interval of 100 °C for Mn decreased with the temperature increasing above 600 °C. Pb and Cd have the maximum volatile ratios in the temperature range of 800–900 °C, whereas Zn has its most significant volatilization in the temperature stage of 600–700 °C. It was observed that there is little combustible materials for tannery combustion above 800 °C, while a large amount of heavy metals are released above 800 °C (Figs. 5 and 7). It is thus critical to control the combustion temperature at an optimal value for good combustion efficiency and minimization of heavy metals emission. Based on the investigation of the tannery sludge's combustion characteristics, almost all the combustible components in tannery sludge were burnt before 800 °C. Thus, 800 °C appears to be a reasonable temperature for safer (less toxic metal volatization) tannery sludge incineration.

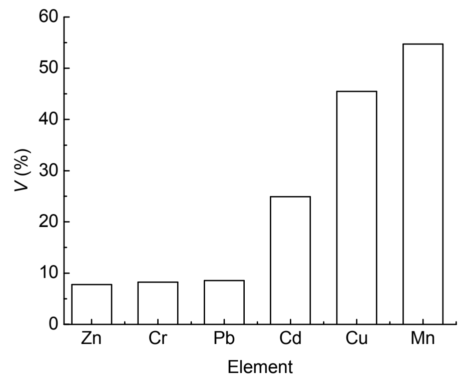


Fig. 6 Vaporization fraction of heavy metals at 500 °C

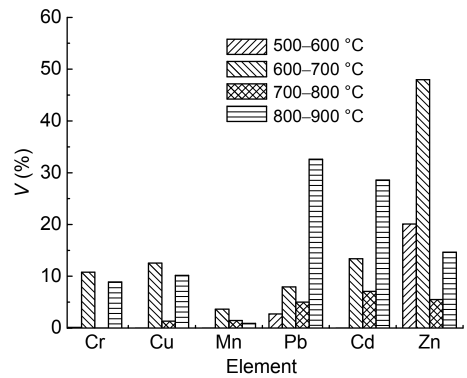


Fig. 7 Volatilization of heavy metals in different stages of tannery sludge combustion

4 Conclusions

Incineration is an available technique for disposal of tannery sludge, and the combustion of tannery sludge can be subdivided into two stages (low and high temperatures). The combustion efficiency depends on the final temperature and residence time in combustion, while the parameter of heating rate has a weaker influence on efficiency. The degrees of volatilization for heavy metals at 900 °C follows the order of Zn>Cd>Cu>Mn>Pb>Cr, and degrees of volatilization increase with temperature. Almost all Zn in tannery sludge is lost at 900 °C, while volatilization of Cr is the least at each combustion temperature. Most of Cr in tannery sludge is enriched in the residue during combustion. Although in real systems other phenomena may influence the emission of heavy metals, such as condensation and adsorption over fly ashes, the present study reveals that it is critical to control the combustion temperature for optimal combustion efficiency and minimization of heavy metals emission; wherein 800 °C is recommended in the case of this tannery sludge.

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