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Evaluation of lead recovery efficiency from waste CRT funnel glass by chlorinating volatilization process

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The current study was carried out to develop a novel process, namely chloride volatilization procedure for lead recovery from waste cathode ray tube (CRT) funnel glass. In the recovery system, the glass powder was first compressed into cylindrical pellet homogeneously with chlorinating agents, and then subjected to thermal treatment for solid-phase reaction. In this case, lead could be easily released from the silicon oxide network of the glass and it was recovered in the form of PbCl_2 . It was found that CaCl_2 was the most effective chlorinating agent, and the optimum operation temperature, holding time and system pressure were 1000°C , 2 h, 600 ± 50 Pa, respectively. The evaporated PbCl_2 could be easily recovered by a cooling device. The evaporation ratio of lead from waste CRT was 99.1% and the purity of the recovered PbCl_2 product was 97.0%. The reaction routes and lead recovery mechanisms of the process were identified. This study provides an efficient and practical process for waste CRT funnel glass detoxification and recycling.

Keywords: chloride volatilization; lead recovery; cathode ray tube; funnel glass; detoxification

1. Introduction

Waste cathode ray tube (CRT) funnel glass generally contains 21–24 wt.% PbO , which is a rapidly growing hazardous waste.[1] Lead exists in the form of $x\text{PbO}(1-x)\text{SiO}_2$ in CRT glass; thus, it is difficult to use conventional methods to extract the lead completely from funnel glass.[2–5] However, the boiling point of PbO is 1740°C and the boiling point of PbCl_2 is only 950°C . In addition, the Gibbs free energy of the chlorination reaction drops to negative value with the help of SiO_2 present in the CRT funnel glasses, which allows the chlorination–volatilization process to occur in a relatively low temperature. Therefore, the transformation of PbO into PbCl_2 by chlorination–volatilization process is thermodynamically preferred for detoxification of waste CRT funnel glass according to its unique composition. Thus far, chlorination–volatilization process has been applied to extract cadmium, zinc, lead and other metals from molten slag,[6] metal ores,[7–9] fly ash [10–13], etc.

A number of studies have been conducted on extracting or recovering lead from waste CRT funnel glasses, e.g. Tung-Chai Ling demonstrated that 3–5% concentration levels of nitric acid can effectively extract most of the lead from the crushed funnel glass surface.[14] Sasai et al. [15] extracted metal from spent lead glass using Na_2EDTA reagent by the wet grinding operation. Yot and Mear [16] investigated the effect of silicon carbide

and titanium nitride on reducing lead contained in waste funnel CRT glasses. Other reports concentrated on the encapsulation of lead inside the glass, e.g. Kim et al. [17] utilized minute amounts of biopolymers and a cross-linking agent to encapsulate CRT wastes into a concrete composite. And Nirut Lairaksa studied the encapsulation of funnel glass in a cement clinker.[18] Moreover, some pretreatment approaches, such as subcritical water treatment, mechanical activation and ultrasonically enhanced leaching were also developed to improve the lead extraction ratio.[19–21] Chen et al. demonstrated that pyrovacuum reduction with carbon is an effective way of lead recovery.[22] Comparatively, as Table 1 shows, the chlorination–volatilization process has many significant advantages over the foregoing techniques, including ease of breaking the chemical binding between SiO_2 and lead atom in CRT funnel glass, environmentally benign, use of cheap additives, low-energy consumption, ability to produce high purity product with high evaporation ratio (ER), etc., which makes it more efficient and profitable than other techniques.

Nevertheless, there are few reports on recovering lead from waste CRT funnel glass by chlorinating volatilization process. The main purpose of this research is to investigate the feasibility of using chlorine-based metallurgy technique for lead recovery to develop an environmental benign process for waste CRT funnel glass disposal. In a previous

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Table 1. Comparisons of different methods of lead removal from CRT funnel glass.

Methods	Equipment investment	Power consumption (W)	Additional agent	Treatment time (h)	Reaction temperature	Lead removal (%)
Pyrovacuum reduction [22]	1200 USD	4200	C	4	1000°C	98.6
Mechanical activation [20]	2700 USD	750	HNO ₃	3	95°C (1 h)	92.5
Ultrasonically leaching [21]	1000 USD	800	HNO ₃	1	Room temperature	91.0
Supercritical water [19]	5400 USD	2200	HNO ₃	18–22	355°C (3–7 h)	93.0
					100°C (15 h)	
SiC reduction [16]	900 USD	4000	SiC	1	950°C	40.0
This work	1200 USD	4200	CaCl ₂	2	1000°C	99.1

report,[23] a separation rate of 80% for lead from funnel glass was achieved through chlorinating volatilization process. Comparatively, lead separation rate of >99% was reached in this work by applying chlorinating volatilization process in vacuum system. Furthermore, this work focused not only on removal, but also on finding an effective way to recover high pure lead chloride powdered products without producing harmful off-gas. This work also evaluated the environmental impact of the chlorinating volatilization process for the first time and demonstrated that the residue of this process was no longer considered as hazardous waste. The optimum operating conditions were determined, the thermodynamic parameters were calculated and the mechanisms of the chlorinating process were identified.

2. Materials and methods

2.1. Sample preparation

Waste CRT funnel glass was provided by Huaxing Environmental Protection Corporation (Beijing). The glass was first broken into small pieces (3–9 mm) and cleaned by ultrasonic cleaning and wet scrubbing to remove coatings. The particles were then dry ball milled into powder and sieved through a 200-mesh sieve. Finally, the powder was dried at 105 °C for 24 h. All of the chemical reagents, e.g. NaCl and CaCl₂, were purchased from Chemical Reagent Company of Beijing in analytical grade.

2.2. Experimental procedure

The CRT glass powder was mixed with the solid-phase chlorination agent and then 1.0 g of the mixture was compressed into a cylindrical pellet using a hydraulic oil press machine at 5.0 MPa. The pellet was then loaded into the quartz crucible and placed in the process chamber.

The installation of the chamber was presented in a previous report.[24] Briefly, the chamber consists of four basic units, i.e. heating system, water cooling system, vacuum system and argon gas supply system. The chamber was air-tight, which was evacuated by a vacuum pump to a pressure of 600 ± 50 Pa when vacuum is needed. To

stabilize the atmosphere inside the furnace and draw out the volatilized gases, air was injected into the chamber at a rate of 200 ml/min. When no oxygen atmosphere was needed, a purity argon gas (99.9% purity) was blown into the chamber to remove the air. At subatmospheric pressures, the pressure in the chamber was controlled by a vacuum valve and measured by a pressure gauge (ZDM-1-20 capacitance diaphragm gauge). The chamber was heated by graphite heaters to the set temperature, and the water cooling device was opened during the process.

Considering potential formation of cracks at the surface of the quartz material, the highest temperature was 1100 °C in this study. According to the physical property of PbCl₂, [25] when the atmosphere pressure is below 5 mmHg, the boiling point of PbCl₂ is approximately 600 °C. Because of these preconditions, pellets with chlorinating agent ratios of 15, 25 and 35 wt.% were examined at temperatures 600 °C, 700 °C, 800 °C, 900 °C, 1000 °C and 1100 °C with a hitting rate of 20 K/min, and holding times were 0.5, 1, 1.5 and 2 h, respectively. The chamber was slowly cooled down to room temperature after each treatment. The residue and the product on the water cooling system were collected. Meanwhile, the off-gas was absorbed by 1.0 mol/L HNO₃.

2.3. Analysis

Chemical composition of the CRT funnel glass was carried out by X-ray fluorescence (XRF-1800). The product and the residue were digested by HNO₃–HClO₄–HF.[26] After dilution, the samples were analysed by an inductively coupled plasma optical emission spectrometer (ICP-OES, OPTIMA 2000). Measurement errors were controlled at around $\pm 1\%$. Mean values of duplicated experiments were reported. Lead leaching behaviour of residue was analysed based on the toxicity characteristic of the leaching procedure (TCLP) standard of U.S. EPA.[27,28] X-ray diffraction (XRD) measurements used the Ni-filtered Cu K α radiation on a RigakuD/MAX2500 diffractometer over an angle of $5^\circ < 2\theta < 90^\circ$. The results of the thermodynamic interactions between the glass and the chlorination agents were calculated using the software package HSC Chemistry 6.0.

Table 2. Chemical composition of the CRT funnel glass by X-ray fluorescence (XRF).

Oxide	SiO ₂	PbO	K ₂ O	Na ₂ O	Al ₂ O ₃	CaO	MgO	SrO	Fe ₂ O ₃	BaO	SO ₃	Other
Wt. %	51.46	24.59	8.00	5.20	3.20	2.92	2.25	0.91	0.47	0.40	0.37	0.23

3. Results and discussion

3.1. Chlorinating agent selection

The chemical composition of the CRT funnel glass is presented in Table 2. According to previous reports, the most commonly used chlorination agents are Cl₂ (g), HCl (g), NaCl, CaCl₂, MgCl₂ and AlCl₃. [29] Among which Cl₂ (g) and HCl (g) are environmentally hazardous. Meanwhile, MgCl₂ and AlCl₃ are relatively expensive and are normally used in a noble metal chloride refining process. Comparatively, NaCl and CaCl₂ are cost-effective and environmentally benign. Therefore, NaCl and CaCl₂ were employed as chlorination agents in the current study.

The chemical equation (1) shows that chlorination reaction does not happen directly between NaCl and PbSiO₃ even at a temperature of 1100 °C. However, according to Equation (2), it happens when SiO₂ and Al₂O₃ exist. When the reaction temperature was above 700 °C, the remaining residues melt and adhere to the crucible. Accordingly, it was deduced that NaCl was not a suitable chlorinating agent for lead recovery from waste CRT funnel glass.

$$\Delta G_{1100^\circ\text{C}} = +11.19 \text{ kJ} \cdot \text{mol}^{-1} \quad (1)$$

$$\Delta G_{1100^\circ\text{C}} = -15.68 \text{ kJ} \cdot \text{mol}^{-1} \quad (2)$$

Furthermore, a composite consisting of NaCl and CaCl₂ in the molar ratio of 1:1 was examined as a chlorinating agent in a ratio of 35 wt.%. In this case, the residue did not melt, but the evaporation of lead was relatively low, e.g. the highest ER was 79.8% at 1000 °C. Comparatively, using CaCl₂ alone as a chlorinating agent had the highest lead recovery ratio, which reached 99.1% at 1000 °C. An ER index of metallic lead was defined by Equation (3) to indicate the evaporation efficiency:

$$\text{ER}(\%) = \frac{((m_{\text{CRT}} \times \text{wt. \% of PbO} \times (\text{MW of Pb}) / (\text{MW of PbO})) - (m_{\text{r}} / m_{\text{a}} \times C_{\text{Pb}} \times V_{\text{a}} \times \text{MW of Pb}))}{(m_{\text{CRT}} \times \text{wt. \% of PbO} \times (\text{MW of Pb}) / (\text{MW of PbO}))} \times 100\% \quad (3)$$

where m_{CRT} is the weight of CRT funnel glass in the pellet, wt. % of PbO (Pb) is the amount of lead oxide (lead) in the glass, MW is the molecular weight, m_{r} is the weight of residue, m_{a} is the weight of residue used for analysis, C_{Pb} is the result from ICP-OES and V_{a} is the volume of diluents.

According to thermodynamic calculation, lead, sodium and potassium chlorides were released from the glass matrix at relatively low temperature because of the strong tendency of calcium to form silicates. Lead chloride was formed by the replacement of lead with calcium in the silicate glass phase:

$$\Delta G_{1000^\circ\text{C}} = -14.93 \text{ kJ} \cdot \text{mol}^{-1} \quad (4)$$

Negative ΔG values were obtained at a large range of temperatures, implying that lead was efficiently volatile in the presence of CaCl₂. The chlorinating reaction occurred in the condition of constant pressure and temperature, according to Van't Hoff isothermal formula, since a ΔG of $-14.93 \text{ kJ mol}^{-1}$ resulted in an equilibrium constant K of 4.10 in the following equation:

$$\Delta G = -RT \ln K \quad (5)$$

where T is the temperature and $R = 8.314 \text{ J} \cdot \text{mol}^{-1} \text{K}^{-1}$.

Accordingly, CaCl₂ was selected as the chlorinating agent. However, CaCl₂ could not react with PbO when the temperature was lower than 1100 °C in the absence of SiO₂ as Equation (6). It indicates that raw component in CRT glass decreases the temperature of chlorination process. On the other hand, the chlorination process utilizes the raw component from the CRT glass itself, which makes the chlorine-based metallurgy technique used in CRT funnel glass detoxification more efficient than in other materials that do not contain SiO₂.

$$\Delta G_{1000^\circ\text{C}} = +1.66 \text{ kJ} \cdot \text{mol}^{-1} \quad (6)$$

3.2. Optimization of the operating parameters

Based on Table 1, the required mass ratio of CaCl₂ was 48.8 wt.% to convert all the SiO₂ into CaSiO₃, and 10.9 wt.% of CaCl₂ was necessary to convert all the lead into PbCl₂. Therefore, adding percentage of CaCl₂ was examined within the above range. It was found that 35 wt.% of CaCl₂ was the optimum amount since the ER of lead was high enough at this ratio, and there was 22.3 wt.% SiO₂ left in the residues at the end of the process. This percentage is similar to that of SiO₂ in cement, implying that the residue was appropriate for application as fine aggregate in cement mortar. [30–33]

The lead evaporating ratio is closely related to temperature, holding time, system atmosphere and pressure.

Figure 1. Lead evaporation ratio as a function of treatment temperature (system pressure = 600 ± 50 Pa, air flow rate = 100 ml min^{-1} and holding time = 1 h).

According to Figure 1, lead evaporating ratio increased with treatment temperature and finally approached to almost 100% in 1 h at around 1100°C . Figure 2 illustrates the effects of holding time, system atmosphere and pressure on lead ER. It was clear that the optimum operation temperature, treatment time and system pressure were 1000°C , 2 h, 600 ± 50 Pa, respectively. The maximum ER of lead reached 99.1%. It was notable that the ER of this reaction within the first 1.5 h was lower in air than in argon gas, which could be attributed to the impact of H_2O (g) in air. H_2O (g) could react with CaCl_2 and form HCl (g) that weakened the effect of the chlorination reaction as the following

equation:

$$\Delta G_{1000^\circ\text{C}} = -1.732 \text{ kJ} \cdot \text{mol}^{-1} \quad (7)$$

Accordingly, dry reaction environment in the chamber could benefit the chlorination–volatilization process. Argon gas is much expensive than air; thus, it is considered that dry air is a preferable atmosphere in the process.

3.3. Product properties

Table 3 calculates the product collected by the cooling system containing 25.6 wt.% of lead, needing purification. The boiling point of PbCl_2 , NaCl and KCl were 950°C , 1465°C and 1500°C , respectively, showing that it was sufficient to separate these compounds by volatilization process in the same vacuum reaction system. Figure 3 shows the effect of temperature on the purification of the material through evaporating process for 30 min. The most efficient temperature was 650°C for product purification, at which the PbCl_2 content of the product reached 97.0 wt.%, close to the industrial grade of PbCl_2 (98.5–99.5 wt.%). When the temperature was 600°C , the evaporation rates of NaCl , KCl and CaCl_2 were lower than those at 650°C . The evaporation rate of PbCl_2 increased when the temperature was higher than 650°C , leading to the decrease of PbCl_2 in the residue. Figure 4 demonstrates that the purification process was effective to eliminate most of the crystal impurities, e.g. the product contains only two crystals, PbCl_2 and KPb_2Cl_5 (Figure 4(a)), but the raw product from the chlorination

Figure 2. Lead evaporation ratio as a function of treatment time. (a) argon gas, 600 ± 50 Pa; (b) air, 600 ± 50 Pa; (c) argon gas, atmospheric pressure; and (d) air, atmospheric pressure.

Table 3. Chemical composition of the powder in the cooling system by X-ray fluorescence (XRF).

Elements	Cl	Pb	O	Na	K	Other
Wt. %	31.79	25.57	18.13	13.80	9.24	1.47

Figure 3. Lead content in the product after purification as a function of treatment temperature (system pressure = 600 ± 50 Pa, air flow rate = 100 ml min^{-1} and holding time = 30 min).

Figure 4. XRD results of the product before and after purification. (a) After purification at 650°C , argon gas, 600 ± 50 Pa and (b) before purification.

process contained many impurities such as NaCl, KCl, $\text{Pb}_2\text{O}_2\text{Cl}$, PbCl_2O_4 and Pb_2OCl_2 (Figure 4(b)).

3.4. Mass balance of lead

During the chlorinating volatilization process, lead could be distributed to the cooling system, the residue and the exhausted off-gas, and the percentages of the optimum operation were 95.6%, 0.9% and 3.5%, respectively, as Equation (8). Cooling system, namely lead recovery system,

captured most of the volatilized PbCl_2 . After purification, 94.9% of lead as the final product was collected from the cooling system. This system prevented the volatilization process from secondary pollution.

$$\begin{aligned} \text{Total Pb (100\%)} &= \text{Cooling system (95.6\%)} \\ &+ \text{Residue (0.9\%)} + \text{Off-gas (3.5\%)} \end{aligned} \quad (8)$$

There was no waste water produced during the process. And the concentration of lead in the off-gas was found to be 7.0 ppb by analysing the off-gas absorption liquid, which was below the limited value specified by GB-25466-2010 standard for a new industrial facility (8.0 ppb).

3.5. Characterization of the residues

Figure 5 illustrates metal contents in the residues. Monovalent metals were easy to evaporate from the pellet, indicating that NaCl and KCl were not suitable to be used as chlorination agents. On the other hand, multivalent metals, such as calcium, magnesium and aluminum, are generally left inside the residues. These multivalent metals are easy to react with silicate as in the following equations:

Therefore, CaCl_2 , MgCl_2 and AlCl_3 are suitable for utilization as chlorination agents in lead recovery process. Multivalent metals in CRT funnel glass were stable while monovalent metals tend to evaporate with PbCl_2 , especially at 600 ± 50 Pa. Hence, it is necessary to remove monovalent metal impurities from the PbCl_2 product.

Figure 6 shows the XRD results of the residues. Main crystal phases in the residues were CaSiO_3 and SiO_2 . The amorphous waste CRT glass powder composed by plenty of irregular particles turns into crystal structure after chlorination process. Lead leaching concentration of the residues was evaluated according to the TCLP of USEPA. The lead leaching concentration of the residue from the optimum operation condition was $1.69\text{--}2.11 \text{ mg L}^{-1}$, below the regulation threshold 5 mg L^{-1} . After the recovery process, lead leaching amount of the residue were largely reduced compared with the original CRT funnel glass (coarse size is 3–9 mm), whose leaching amount was 18.8 mg L^{-1} . Accordingly, the residue was safe to be disposed in landfills or be used as construction material.

Figure 5. Residue metal contents as a function of treatment time. (a) Argon gas, 600 ± 50 Pa; (b) argon gas, atmospheric pressure; (c) air, 600 ± 50 Pa; and (d) air, atmospheric pressure.

Figure 6. XRD patterns of the glass residues (air, 600 ± 50 Pa).

4. Conclusions

The results of the current study demonstrated that chlorinating volatilization process was efficient and practical for detoxification and recycling of waste CRT funnel glass. During the process, 99.1% of lead could be volatilized from the waste glass and 94.9% of lead was recovered by the cooling system. The optimal processes operating temperature, holding time and system pressure were 1000°C , 2 h and 600 ± 50 Pa, respectively. Among the most commonly used chlorination agents, CaCl_2 was the most efficient agent. After the treatment, the residues met USEPA specifications for non-hazardous waste and can be disposed in landfill or recycled as fine aggregates. Moreover, this process could recover a valuable product with a purity of 97.0 wt.% PbCl_2 ,

showing a considerable economical profit for CRT funnel glass disposal.

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