

The behaviour of selected heavy metals in MSW incineration electrostatic precipitator ash during roasting with chlorination agents

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Received 1 November 1995; accepted 5 February 1996

Abstract

The thermal behaviour from 500–1050°C of selected heavy metals (Pb, Zn, Cu and Cd) in the electrostatic precipitator (ESP) ash from a municipal solid waste (MSW) incinerator was studied on a laboratory scale. The parameters studied included temperature, reaction time, type of chlorination agent, and amount of chlorination agent. The vaporization and removal of these metals are characterized by the formation and volatilization of the metal chlorides, and are dependent on temperature and time. As a result of volatilization, $\approx 90\%$ of Pb, Zn, Cu and Cd can be removed from the ESP ash under the conditions studied. The fraction of metal removed is determined mainly by the relative stabilities of the metal chloride and oxide. The results provide a basis for the development of a thermal treatment process to convert the ESP ash from potentially hazardous into non-hazardous material and to recover the selected metals from the ash.

Keywords: Chlorination; Electrostatic precipitator ash; Heavy metal; MSW incineration; Roasting

1. Introduction

This paper describes the investigation of the behaviour of selected heavy metals in the electrostatic precipitator (ESP) ash from a municipal solid waste (MSW) incinerator when heated to various temperatures (500–1050°C) with and without the addition of solid chlorination agents. The focus is on Pb, Cd, Zn and Cu contaminants in the ESP ash. Pb and Cd are the elements commonly detected in the ESP ash leachate, concentra-

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tions of which often exceed the limits set by legislation [1], and Zn and Cu are the heavy metals present in a relatively high concentration. The objective of this study was to identify the conditions under which heavy metals would be removed by chlorination volatilization. The parameters investigated in the experiments were heating temperature, heating time, and amount and type of chlorination agents.

ESP ash from the MSW incinerators contains heavy metals, such as Pb and Cd [2], which can be leached into soil and water bodies if the ash is disposed of in a landfill. Mitchell et al. [3] investigated ESP ash samples in the UK, and found that the median concentrations of Pb, Cd and Zn in the ESP ashes were 108, 113 and 387 times higher, respectively, than in soil. Graydon and Kirk [4] reported that the selected metals exist primarily on the surface of larger particles (mostly silicates) in the form of chlorides, and this is supported by the observation of Germani and Zoller [5]. The sources of Pb, Cd and Zn in MSW include plastics containing Cd stabilizers and pigments, and discarded Ni–Cd and Pb batteries [6,7].

Leaching of heavy metals from the ESP ash in landfill to the environment has received considerable attention and has been addressed in the literature [3,8,9]. According to Mitchell et al. [3], selected metals associated with MSW incineration ESP ash are likely to be more soluble in landfills than those associated with coal ESP ash. Germani and Zoller [5] studied the solubility in water of elements in ESP ashes, and obtained solubilities ranging from 63% for Zn to 100% for Cl. This mobility could be greatly enhanced if the ash is co-disposed with organic biodegradable materials found in ordinary domestic refuse [3]. To prevent leaching of heavy metals to the ground water and/or subsoil, the landfill sites may be equipped with liners or leachate collection/treatment facilities or heavy metals may be removed prior to the disposal.

A process which would decontaminate the ash so that it can be safely disposed of in a landfill would be desirable, especially if the process could simultaneously recover the contaminant metals. A wet process of extracting the metals from the ESP ash with acids has been used in some places [10,11] for these purposes. The drawbacks of this process are the difficulties of filtering the leachate owing to the formation of gelatinous silicate and aluminium compounds, and in recovering the individual metals from the leachate because of its complex matrix. On the other hand, a dry process, such as thermal treatment, is potentially attractive. In view of the volatile nature of metal chlorides at high temperature, it appears possible to separate some heavy metals from the bulk (Si, Al and Ca) of the ash by a thermal process.

Recently, Jakob et al. [12] studied the evaporation of Zn, Pb, Cd and Cu in an ESP ash in various atmospheres at temperatures between 670°C and 1300°C, and found that the evaporation was most effective at temperatures just below the melting range of the ash (1000–1100°C). Fray [13] reported that Zn, Pb and Cd could be separated from arc furnace dust, which was mainly iron oxide, when the dust was heated with chlorine in air. Deng and Li [14] roasted a complex gold ore at 1000–1050°C using calcium chloride as a chlorination agent and were able to recover the precious metals as chlorides. In fact, as a common practice, chlorination is an important first step in the extraction of zirconium, vanadium and titanium from their oxides [15]. Temperature is a key factor in chlorination. When chlorination is conducted in the temperature range 100–600°C, the metal chlorides formed are usually non-volatile, and the metals are

recovered by leaching with water. Chlorination can take place in the temperature range 600–1000°C such that the wanted metal chlorides are volatilized and recovered by condensation, as in the case of titanium recovery. Chlorination agents may be gaseous, liquid or solid. Gaseous reagents are chlorine, hydrogen chloride, and phosgene (COCl_2). Chlorine gas is the most common chlorination agent. Liquid reagents are carbon tetrachloride and sulphur chloride, and both are sometime used in the gaseous state. The most commonly used solid reagents are sodium chloride and calcium chloride [16].

A large scale thermal treatment process could consist of a rotary kiln to heat the ESP ash and a recovery system to collect the volatilized metal compounds. The set-up would be similar to the one, for example, used in Japan at the Tobata plant of the Kowa Seiko Co. [16], where a rotary kiln and a scrubber are used for roasting pyrite cinder and for recovering the volatile non-ferrous metals. The cost for disposing of the treated ESP ash in a landfill would be substantially lower than that for disposing of the untreated ash to a secure landfill should it be classified as a hazardous waste. Also, there is the opportunity to use the treated ash as a raw material in industrial processes, such as cement production.

2. Experimental

2.1. Materials and reagents

The ESP ash used in this study was from a municipal solid waste (MSW) incineration plant. The flue gas from the combustor was passed through electrostatic precipitators prior to discharge. Grab samples of the ash collected from the electrostatic precipitator were passed through a 20 mesh sieve and well mixed by a tumbling mixer in this laboratory.

All chemicals used were of reagent grade, and water was distilled and deionized. The reagents included nitric acid, hydrofluoric acid and perchloric acid for sample preparation. Stock 1000 ppm standard solutions of Pb, Zn, Cu and Cd were used to prepare the working standards for the calibration of analytical equipment. The chlorination agents used were NaCl, $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, $\text{MgCl}_2 \cdot 4\text{H}_2\text{O}$, $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ and $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$. The amounts of chlorination agents shown are based on the chlorine (Cl) content.

2.2. Apparatus

As shown in Fig. 1, a quartz tube (100 cm long \times 3 cm I.D.) furnace (Carbolite Model TZf 12/75) with a programmed temperature controller (Model 808 P) was used in this study. The heating zone, in which the temperature varied within 5°C at 1000°C, was about 15 cm long. A 10 cm long alumina boat, which contained the ESP ash sample, was placed in the heating zone of the quartz tube furnace.

2.3. Experimental procedure

Samples of ESP ash, which had been well mixed, were accurately weighed. For experiments with chloride addition, the chlorination agent, which was in powder form,

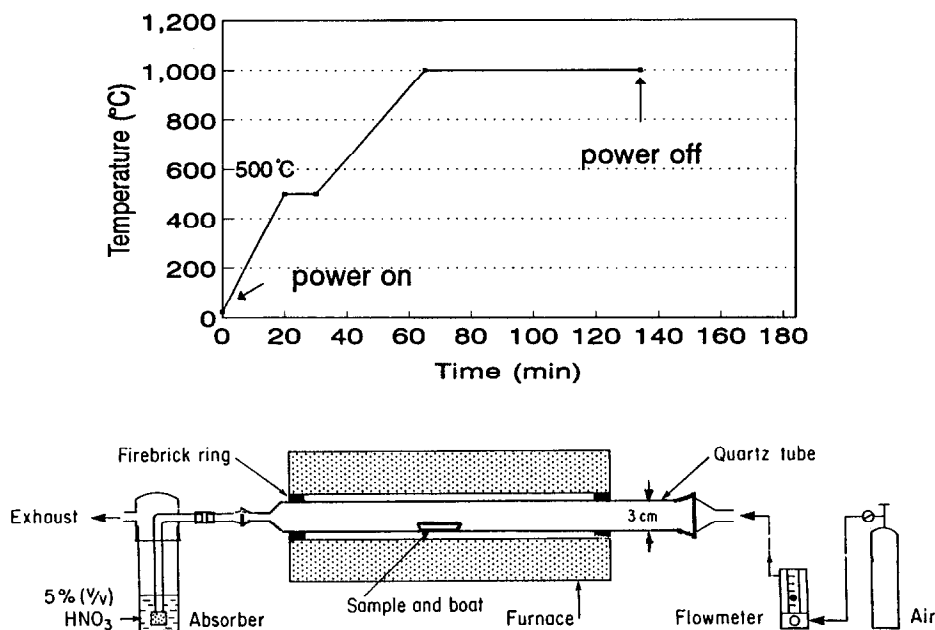


Fig. 1. Experimental apparatus and the heating sequence.

was thoroughly mixed with the ash in a weighing dish using a glass stirring rod before transfer to the alumina boat. During heating, dry air was supplied at $\approx 100 \text{ ml min}^{-1}$ to entrain the volatile matter. On leaving the furnace, the volatile matter first condensed partially on the wall of the quartz tube outside the heating zone and then on the condensation tube; the rest of the volatile matter was absorbed in 5% HNO_3 solution in a scrubber. A typical heating sequence is shown in Fig. 1. The holding time of 10 min at 500°C was to burn off the carbon in the ESP ash, so that the roasting took place under oxidizing conditions.

2.4. Chemical analysis

After the thermal treatment, the ESP ash was separated into two parts: the condensed volatile matter and the ash residue. The volatile matter was collected from three places: (1) on the wall of the quartz tube, (2) in the connecting tube between the quartz tube and the inlet of the absorber, and (3) in the solution of the absorber. The condensed matter in locations (1) and (2) was dissolved by 5% HNO_3 . These solutions, and also the solution from the absorber, were analysed for their elemental content by inductively coupled plasma spectrometry (ICP) using a Spectro Analytical Instruments Model Spectroflame P spectrometer. Some elements were also analysed by atomic absorption spectroscopy (AAS) using a Perkin Elmer Model 703 AA spectrometer. The sum of the contents in these three solutions gave the total amount of elements removed from the ESP ash. The dissolution of the ash residue was accomplished by digestion with an acid mixture of HF , HNO_3 and HClO_4 (4:1:1) similar to that used by Cahill and Newland [17]. This

solution was also analysed for its elemental content using the same analytical techniques. The sum of the elemental content of the condensate and of the digested ash residue gave the total content of the elements in the ESP ash. For Pb, Cu, Zn and Cd, these sums were compared with the total content determined by digesting the raw ESP ash. The summed values account for 99.1% of Pb, 97.7% of Cu, 97.6% of Zn and 96.6% of Cd, respectively. To determine the data quality, experiments were replicated at 1000°C five times. The quantities of the four selected heavy metals volatilized were determined using AAS. The relative standard deviations for Pb, Zn, Cd and Cu were calculated to be 2.7, 2.4, 2.3 and 2.5%, respectively.

X-ray powder diffraction (XRPD), using copper $K\alpha$ radiation (34 kV, 20 mA), was also performed on a sample of the condensed volatile matter which was held on double-sided tape. The diffraction pattern captured on the film in a Guinier–de Wolff camera was digitized using a photodensitometer; the results were compared with the standard data sets using the Rigaku IBM search match program to identify the chemical phases.

3. Experimental results and discussion

Twenty metals were determined in the ESP ash by ICP, these included alkali metals (K and Na), alkaline earth metals (Be, Mg, Ca, Sr and Ba), transition metals (Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Mo and Cd), a Group IIIA metal (Al), and a Group IVA metal (Pb). It was found that the metals that make up > 1% of the ESP ash are Ca, Al, Na, K, Fe, Ti and Mg. A whole rock analysis of this type of ESP ash [4] showed that the ash was composed mainly of silica (36%) and oxides of calcium (13%) and aluminium (12%). The ESP ash contained about 1% Zn, 0.4% Pb, 0.1% Cu and 0.01% Cd, with which this study is most concerned. To quantify the volatility of the metals, the efficiency of volatilization R is used, which is defined as the percentage of a metal that is removed from the ESP ash.

3.1. Time dependence of the removal of Pb, Zn, Cd and Cu

Reaction time is one of the key parameters to be considered in process development. Samples were heated to 1000°C with a chlorination agent (CaCl_2 , 0.3 g Cl in 3 g of ESP ash) and held for different periods of time (0, 30, 60, 120 and 180 min) to study the effect of time on volatilization of metals. As shown in Fig. 2, the R values for Pb, Zn, and Cd reached a maximum after ≈ 30 min at 1000°C. The removal of Cu, however, was still taking place after 180 min. The reason for the different behaviour of Cu is proposed as being the low volatility of copper chloride at 1000°C. The boiling point of CuCl is 1450°C, which is much higher than those of ZnCl_2 , CdCl_2 and PbCl_2 (732°C, 960°C and 950°C). It should be pointed out that removal at time zero ($t = 0$) may not be zero, as some loss could occur during the heating period from 500°C to 1000°C, which took about 20 min.

Evidence showed that the volatile matter was indeed mainly chloride salts. Anion analysis of the condensate using ion chromatography indicated that the volatile matter

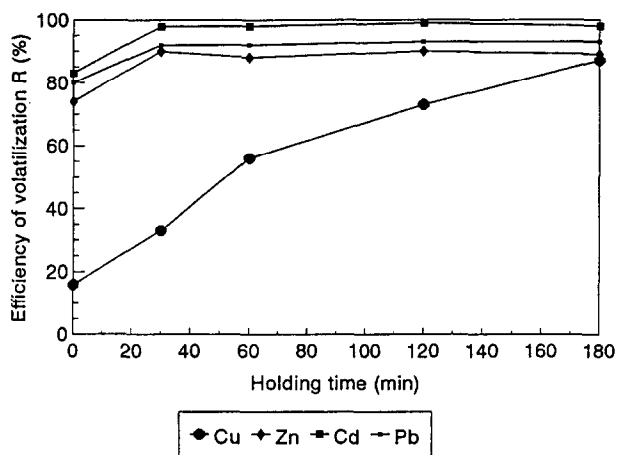


Fig. 2.

contained primarily Cl^- , as shown in Table 1. Its quantity was more than sufficient to account for all the heavy metals of interest. A large portion of the Cl^- , however, was from Na and K compounds. In the condensed volatile matter, XRPD identified two chemical phases, KPb_2Cl_5 and $\text{Na}_2\text{ZnCl}_4 \cdot 3\text{H}_2\text{O}$, which confirmed that the heavy metals, such as Pb and Zn, were in the form of chloride salts. The suggestion by Jakob et al. [12] that in air the heavy metals are probably evaporated as chlorides is supported by this work.

3.2. Effect of temperature on Pb, Cd, Zn and Cu removal

A 3 g ESP ash sample was treated for 180 min at various temperatures (500, 600, 700, 800, 900, 1000, and 1050°C) with and without calcium chloride (0.3 g Cl). Figs. 3 and 4 give the temperature dependence of the efficiencies of volatilization R for four selected heavy metals. As expected, increasing the temperature enhanced the volatilization of the selected heavy metals in both cases. As shown in Fig. 3, volatilization of Pb and Cd became significant when the temperature exceeded 600°C. Above 800°C the

Table 1

Anion analysis on the volatile matter (samples were treated at 1000°C for 1 h with and without a chlorination agent)

Anion	No Cl added		0.3 g Cl added	
	$\mu\text{g g}^{-1}$ in sample	$\mu\text{mol g}^{-1}$	$\mu\text{g g}^{-1}$ in sample	$\mu\text{mol g}^{-1}$
Cl^-	26600	750	31600	890
SO_4^{2-}	2760	39	4790	50
NO_3^-	< 10	< 0.2	< 10	< 0.2
Br^-	< 10	< 0.15	< 10	< 0.15

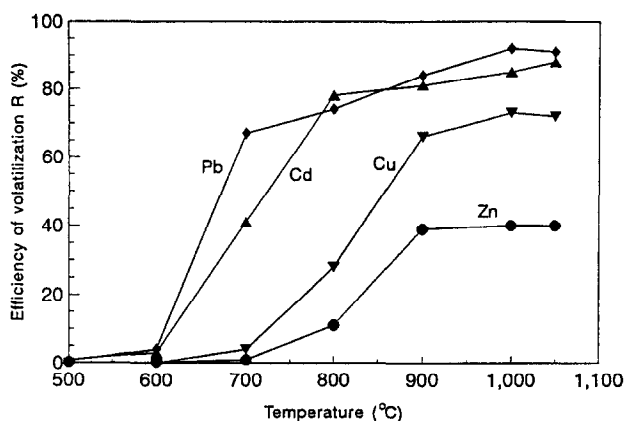


Fig. 3. Temperature dependence of heavy metal removal without chlorination agent (holding at target temperature for 180 min).

temperature effect became smaller. For Cu and Zn, the temperature range for a strong effect was 700-900°C.

The maximum R value achieved for each of the four metals was different. Without a chlorination agent, about 90% of the Pb and Cd was evaporated after 180 min at 1050°C, compared with 70% of Cu and 40% of Zn. Jakob et al. [12] reported that, after treatment for 180 min at 1030°C in air, the fractions of metals evaporated were \approx 90% for both Pb and Cd, 80% for Cu and 40% for Zn, which agree well with our observations. Unlike this study of complete recovery of condensable material, they used a water-cooled cold finger to collect volatile compounds.

Thermal behaviour of heavy metals may reflect the forms of these metals and other major species in the ESP ash prior to treatment. Many studies indicate that heavy metals

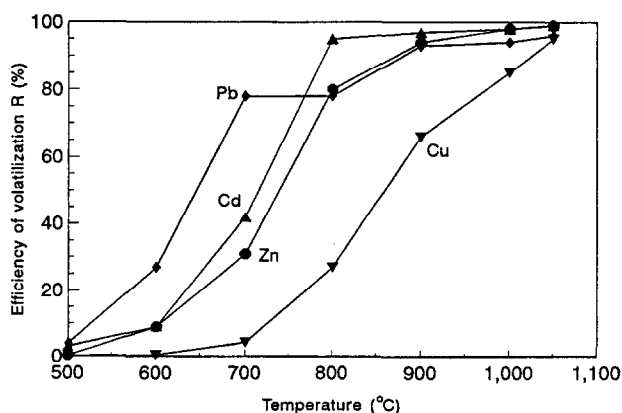


Fig. 4. Temperature dependence of heavy metal removal with 0.3 g Cl added (holding at target temperature for 180 min).

exist as chlorides, oxides and other salts in the ESP ash. The opinions sometimes diverge. For example, McKinley et al. [10] and Jakob et al. [12] suggested that a large portion of the Zn was in an oxide form, whereas Eighmy et al. [18] identified many salt forms of Zn, such as K_2ZnCl_4 . Depending on the initial form of the heavy metals in the ESP ash, their removal may include direct vaporization of chloride or both chlorination and vaporization. Increasing the temperature can enhance vaporization by raising the vapour pressure of the heavy metal chlorides, by improving the rates of diffusion, and by changing the contact pattern of reactants, e.g., from solid–solid to liquid–solid.

With a chlorination agent, as shown in Fig. 4, the trend of temperature dependence of R was similar to that without a chlorination agent. However, the R value for Zn increased significantly with calcium chloride added. Interestingly, the R value for Cu did not change at temperatures below 900°C, but the addition of calcium chloride led to higher R at temperatures above 900°C. Details of the effects of chlorination agents will be discussed later.

Increasing the temperature may accelerate the direct volatilization of alkali metal chlorides (NaCl and KCl), which would dilute the concentration of the valuable metals. It was found that when the temperature was raised from 1000°C to 1050°C, the R values of Na and K increased from 62 to 92% and from 76 to 95%, respectively; it was noted that the initial contents of Na and K in the ESP ash were ≈ 50 and 20 mg g^{-1} respectively. If the objective is only to remove the hazardous metals Pb and Cd, 900°C is sufficient.

3.3. Effect of chlorination agents

Effects of the quantity, type, and addition method of solid chlorination agents on metal removal were studied. To be useful as a chlorination agent, the chlorides MCl_2 must be able to react directly with metal oxides or to release a chlorine-containing gas (Cl_2 or HCl) when they react with oxygen or water according to the following general reactions



The oxide MO should be stable chemically and have low vapour pressure, so that the metal initially associated with the chlorination agent will remain in the ESP ash and will not cause any environmental problem when the treated ESP ash is disposed of in a landfill. Such chlorides as $CaCl_2$, $MgCl_2$, $FeCl_2$ and $AlCl_3$ meet these requirements.

The ESP ash contains about 0.5% moisture, which is readily removed when the temperature is over 100°C. Some of the chlorination agents contain not only moisture but also water of crystallization. Both calcium chloride and magnesium chloride may contain six H_2O units per molecule. Upon heating, however, calcium chloride loses all water at 200°C [19]. Hence, chlorination by HCl, as shown by Eq. (2), is unlikely to occur in this system where dry air is used.

Fig. 5 shows the effect of the amount of chlorination agent on the removal of the four metals. The R values of Pb, Cd and Cu clearly increased with the first 0.1 g of Cl added

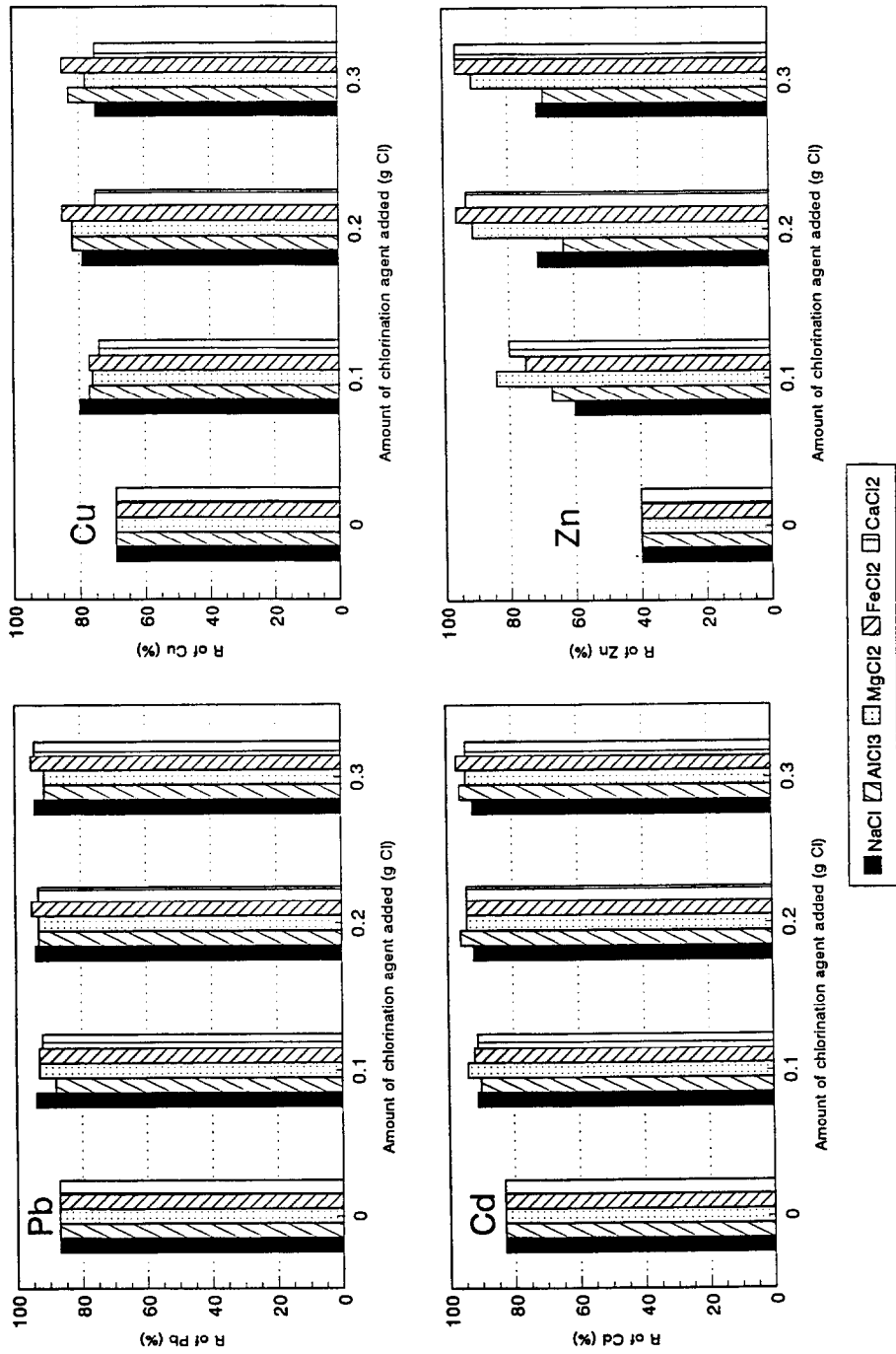


Fig. 5. Effect of amount and type of chlorination agent on heavy metal removal (holding at 1000°C for 60 min).

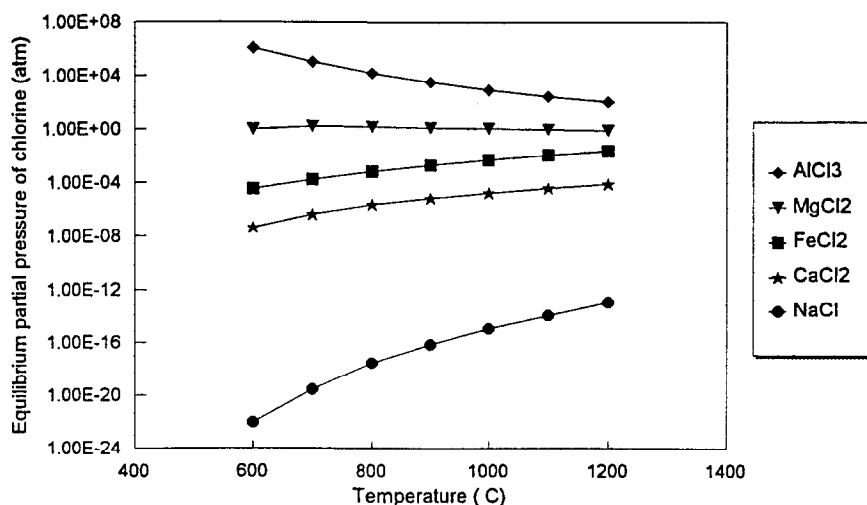


Fig. 6. Temperature dependence of equilibrium partial pressure of chlorine over different chlorination agents (assuming $P_{O_2} = 0.21$ atm).

into the 3 g of ash, regardless of the type of chlorination agent. However, there was no significant change when more chlorination agent was added. The addition of a chlorination agent will not appreciably affect the vaporization of existing metal chlorides. Thus, evidence from the high recovery of Pb, Cd and Cu with no additional chlorination agent and from the increase in recovery with addition of a chlorination agent, suggests that a very large fraction of the Pb, Cd and Cu is already in the form of chlorides. On the other hand, the strong dependence of the removal of Zn on the addition of chlorination agents indicates that a significant fraction of the Zn is in forms other than chlorides.

The effectiveness of the chlorination agents for Zn removal was found to be $MgCl_2 \approx FeCl_2 \approx CaCl_2 > NaCl \approx AlCl_3$. Based on their stability in air, the tendency of different chlorination agents to release chlorine varies. This tendency can be quantified using the equilibrium partial pressure of chlorine over the chlorination agent under a constant partial pressure of oxygen (0.21 atm). As shown in Fig. 6, the equilibrium partial pressures of chlorine decrease according to the sequence $AlCl_3$, $MgCl_2$, $FeCl_2$, $CaCl_2$ and $NaCl$ in the temperature range 600-1200°C. When chlorination is a necessary step for removal of metal, such as Zn in this case, there should be some dependence of R on the type of chlorination agent. Fig. 5 shows that $NaCl$ and $AlCl_3$ were less effective at 1000°C than other chlorination agents in the removal of Zn. In air, $NaCl$ has less tendency to supply chlorine than other chlorination agents. Another consideration is the volatility of the chlorination agent. The vapour pressure of $NaCl$ at 1000°C is two orders of magnitude higher than that of $CaCl_2$. As a result, it is easier for $NaCl$ to evaporate without reacting with oxygen or metal oxides, and to end up with the volatile matter. In other words, less $NaCl$ will be available as a chlorination agent. Analysis of the sodium content of the condensed matter revealed that 62% of the sodium salt ended up with the volatile matter after 180 min of heating at 1000°C, compared with less than 1% of Ca, Mg and Fe salts. The ineffectiveness of $AlCl_3$ is thought to be due

to its high tendency to release chlorine at relatively low temperatures at which the rate of chlorination of Zn is not significant. In fact, AlCl_3 is the only chlorination agent tested which has a very high equilibrium partial pressure of chlorine at low temperatures (see Fig. 6). However, to remove Zn via chlorination, the temperature has to be $> 600^\circ\text{C}$ (see Fig. 4). Thus, aluminium chloride may have released chlorine before the reaction with zinc oxide can occur.

According to Fig. 5, there was no significant difference among MgCl_2 , CaCl_2 and FeCl_2 . When these chlorides are used, the partial pressure of chlorine in the system may be about the same. The reaction between the chlorination agent and oxygen may be controlled by the oxygen partial pressure in the air, which is close to constant in an open system like that used in this study.

The addition of a chlorination agent may enhance the removal of metal by some mechanism other than supplying chlorine. Added chloride may lower the melting point of some species in the ash so that the contact pattern is changed and the reactions are accelerated. For example, NaCl and CdCl_2 can form a eutectic phase with a melting point of 397°C [20]. Added chloride can also lower the viscosity of a molten phase so that the rate of diffusion increases. KCl can lower the viscosity of CdCl_2 from around 200 P to ≈ 150 P at 650°C [21].

So far, the results presented have been based on mixing the dry powder of a chloride with the ESP ash. As the contact of the added chloride salt with the ash phase containing the extractable metal may be a limiting step in the reaction rate, more intimate mixing was performed. To achieve better contact, the chlorination agent and the ash were thoroughly mixed and wetted with water to form a slurry. After being dried in an oven overnight at 105°C , the sample was heated at 1000°C for 60 min. As shown in Fig. 7, metal removal from the wetted samples was improved, particularly for Cu. The maximum increase in R was 10% for Cu with 0.1 g of Cl added as CaCl_2 . As mentioned before, the removal of Cu was a relatively slow process. After the wet treatment, the distribution of both added chloride salt and heavy metal chloride was possibly more uniform. Thus, the contact of the added chloride salt and the targeted metal-containing phase was improved. Again, no significant difference was found between CaCl_2 and MgCl_2 .

4. Conclusions

The behaviour of selected heavy metals in the ESP ash from a municipal solid waste incinerator was studied on a laboratory scale under heated conditions to determine the technical feasibility of a process to detoxify the ESP ash for its safe disposal in a landfill and to recover heavy metals. The findings may be summarized as follows.

1. Heavy metal removal by volatilization from the ESP ash was dependent on time and temperature.
2. Without a chlorination agent, the efficiency of Zn removal was low. After treatment at 1050°C for > 180 min, only 40% of Zn was removed, compared with 90% for Pb and Cd and 70% for Cu. The cause of the low Zn recovery was that a large fraction of the Zn in the ESP ash was in forms other than chlorides.

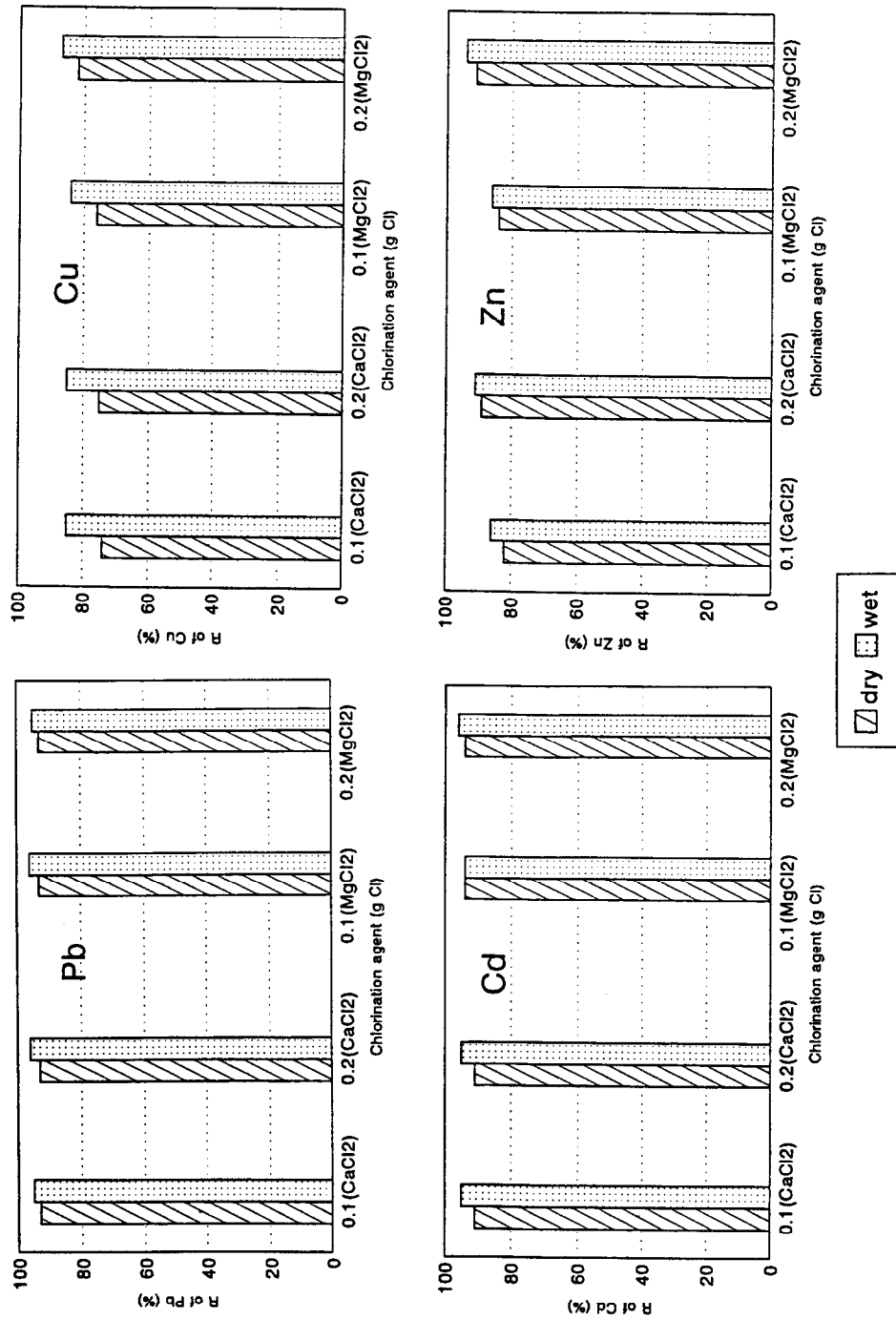


Fig. 7. Effect of contact pattern on heavy metal removal (holding at 1000°C for 60 min).

3. With a chlorination agent (calcium chloride) added, about 90% of the Pb, Cd and Zn was removed from the ESP ash after treatment at 1000°C for 30 min. The removal of Cu under the same conditions was slower than for the other metals. The suspected cause was the low volatility of CuCl at 1000°C.

4. The removal of heavy metals was the result of evaporation of heavy metal-containing chloride salts. Chlorination is important when the heavy metal is in a form other than chloride, which is probably the case for a large fraction of the Zn. As chlorination agents for Zn removal, CaCl₂, MgCl₂ and FeCl₂ were more effective than NaCl or AlCl₃.

Acknowledgements

The authors thank Professor M.P. Gorton for helpful discussions.

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