

## Heavy metal vaporization and abatement during thermal treatment of modified wastes

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### Abstract

This study examines the vaporization percentage and partitioning of heavy metals Cd, Pb and Zn during thermal treatment of wastes with added PVC, heavy metals or phosphate, and the efficiency of sorbents for removal of these metallic compounds in flue gas of an industrial solid waste incinerator. Firstly, vaporization experiments were carried out to determine the behavior of heavy metals during combustion under various conditions (type of waste, temperature, presence of chloride or phosphate . . .). The experimental results show relatively high vaporization percentage of metallic compounds within fly ash and limestone matrix while heavy metals within sediments treated with phosphoric acid are less volatile. Vaporization of metals increases with increasing temperature and with chloride addition. The thermal behavior of the selected heavy metals and their removal by sorbents (sodium bicarbonate, activated carbon) was also studied in an industrial solid waste incinerator. These pilot scale experiments confirm that heavy metals are concentrated in fly ashes and cyclone residues, thus effectively controlling their release to the atmosphere.

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### 1. Introduction

Metal emissions from waste incineration plants are currently of great environmental and regulatory concern because of their toxicity. In fact, a continuous issue facing society is the disposal of municipal and industrial solid wastes. The limits in landfill sites, the growing amount of such wastes, the increasing disposal costs and the ever more stringent regulations have stimulated efforts to improve the efficiency and acceptability of the incineration technology through flue gas treatments and fly ash detoxification by water-washing followed by a sintering treatment [1]. Emission factors and removal efficiencies of heavy metals have been investigated [2] and the kinetics of heavy metal release during waste incineration were studied in the laboratory [3]. Heavy metals emission from solid waste combustion results primarily from the vaporization of elements during the combustion process. Heavy metals vaporization during municipal solid

waste and coal combustion has been studied mainly by direct analysis of the residues produced [4–7]. Other experimental studies on the metal behavior during thermal treatment of artificial solid wastes [4–6,8] showed that heavy metals with higher saturated vapor pressure and lower boiling point are most likely to be found in fly ash and/or in the flue gas. Heavy metals initially vaporize in the flame, the resultant metallic vapors then undergo homogeneous nucleation to form an ultra fine aerosol having a size of 10–30 nm [9,10]. In the post flame region, the combustion gases cool rapidly, the condensed aerosol grows continuously by heterogeneous coagulation; the resultant agglomerates have a mean size of about 1 to 2  $\mu\text{m}$ . The partitioning of heavy metals varies significantly with the element type. Metals of class I such as Ba, Ce, Mg, Mn, Cr are concentrated in the coarse ash (bottom ash), never vaporize [10,11], and rapidly undergo a transition to a various solid species. The class II metals such as Cu, Pb, Se, Zn and As are concentrated in particulate matter while the others such as Br, Hg, I and Cd are concentrated in the gas phase [12,13]. However, the partitioning of heavy metals in the incinerator discharges is affected by a number of factors such as combustion temperature, waste composition, moisture,

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Table 1  
Composition of fly ash (Solvay data)

	Al <sub>2</sub> O <sub>3</sub>	CaO	Fe <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	MgO	MnO	Na <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	
FA (wt.%)	9.1	24.3	8.4	0.9	2.7	0.2	1.6	1.9	18.0	1.8	
	C <sub>mineral</sub>	C <sub>orga</sub>	Cl	S	As (g/kg)	Cd (g/kg)	Cr (g/kg)	Cu (g/kg)	Ni (g/kg)	Pb (g/kg)	Zn (g/kg)
FA (wt.%)	0.8	0.3	0.6	6.9	0.071	0.250	0.420	0.880	0.077	5.400	11.000

Table 2  
Composition of phosphated sediment (Solvay data)

	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Ca	SO <sub>4</sub>	Al <sub>2</sub> O <sub>3</sub>	PO <sub>4</sub>	OM	Cd (g/kg)	Cr (g/kg)	Cu (g/kg)	Ni (g/kg)	Pb (g/kg)	Zn (g/kg)	Cl (g/kg)
Phosphated sediment (wt.%)	23.0	4.5	11.5	1.2	4.5	4.5	10.4	0.016	0.117	0.347	0.103	1.000	3.600	0.500

chlorides in waste, etc. [14–16]. Our previous work was centered on the stabilization of metals with phosphates [17–18]. Little is known on metal behavior and partitioning during waste incineration.

This paper describes an experimental study of the vaporization of metals (Cd, Pb and Zn) during thermal treatment of wastes with added PVC, heavy metals or phosphate. Laboratory and pilot scale experiments are carried out to evaluate the effects of operating conditions on vaporization percentage of metals within wastes and to determine the efficiency of sorbent injection (sodium bicarbonate, activated carbon) in flue gas to remove these toxic compounds. The reactants used today in Europe for flue gas treatment are Ca(OH)<sub>2</sub> or NaHCO<sub>3</sub> together with activated carbons (Neutrec Process<sup>®</sup>).

## 2. Experimental

### 2.1. Industrial wastes

The wastes used are incinerator fly ash and dredged sediment treated with 5% of 85% phosphoric acid to stabilize the heavy metals in it (NOVOSOL Process<sup>®</sup>, Solvay group). The amount of phosphoric acid added was fixed according to previous studies [19,20].

The fly ash (FA) collected from a power plant has the chemical composition presented in Table 1. These mineral wastes contain 14% humidity and relatively high concentrations of heavy metals such as Cd (250 ppm), Pb (5400 ppm) and Zn (11000 ppm).

An organic waste, sediment treated with 5% of 85% phosphoric acid, is also investigated to evaluate the effect of phosphate reaction on metals contained in the raw sediments. Their heavy metal concentrations are lower than those of fly ash and equal to 16 ppm of Cd, 1000 ppm of Pb and 3600 ppm of Zn (Table 2), but their water contents are higher (40%).

Finally, artificial mineral waste made of limestone (35 wt.% humidity), with low heavy metal concentration (Table 3), doped with CdCl<sub>2</sub>, PbCl<sub>2</sub> or ZnCl<sub>2</sub> 2 wt.% is also used for the better understanding of the vaporization process.

Organic chloride (PVC) is added at 5% by weight (for the laboratory scale experiments) and at 1% by weight (for the pilot scale experiments) to the different wastes to simulate chloride source in the waste and determine its effect on metal vaporization.

### 2.2. Experimental procedure

#### 2.2.1. Vaporization (laboratory scale)

Vaporization experiments are firstly carried out at laboratory scale by combustion of few grams of waste (3–5 g), put in an alumina crucible pushed into a quartz tube in a preheated horizontal tubular furnace CARBOLITE 12/65/600 (diameter = 60 mm, length = 600 mm). During calcination experiments, air flow rate is fixed at 150 L/h. The influence of temperature (600–850 °C), combustion duration (10 min to 4 h from initial sample introduction to withdrawal from the temperature stabilized oven), composition of the matrix containing heavy metals and PVC addition, on the vaporization of metals is studied. Finally, the oven is connected to two impingers filled with a solution of 100 mL HNO<sub>3</sub> 0.5 M allowing the collection of the vaporized fraction of the heavy metals to be determined (Fig. 1).

#### 2.2.2. Adsorption experiments in industrial pilot plant waste incinerator (NOVOSOL)

Experiments in an industrial waste incinerator developed by SOLVAY (Fig. 2) and located at Dombasle sur Meurthe plant in France, are also carried out. 200 kg of waste (fly ash, limestone doped with CdCl<sub>2</sub> or PbCl<sub>2</sub> 0.5 wt.%) are calcinated in a rotary kiln at 850 °C during 1 h with a solid flow rate of about

Table 3  
Composition of limestone (Solvay data)

	Ca	Na	CO <sub>3</sub>	SO <sub>4</sub>	Cl	H <sub>2</sub> O	Fe	Cd (g/kg)	Cr (g/kg)	Cu (g/kg)	Ni (g/kg)	Pb (g/kg)	Zn (g/kg)
Limestone (wt.%)	25.2	1.5	36.8	0.1	0.1	35.0	0.3	0.001	0.006	0.006	0.001	0.006	0.007

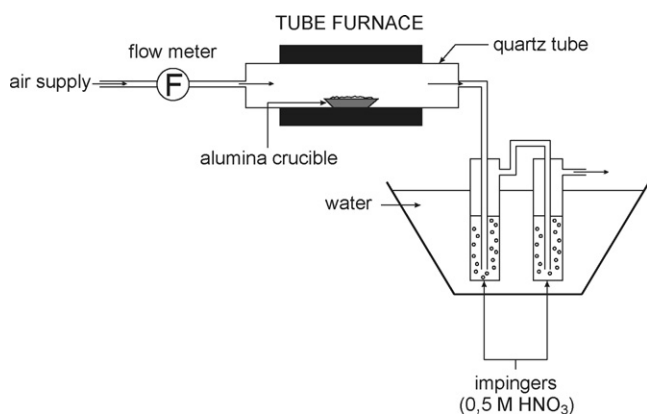


Fig. 1. Laboratory tube furnace.

30 kg/h. This industrial waste incinerator allows calcined waste, fly ashes produced and scrubber residues collected on the filter to be recovered and mass distribution to be estimated. During thermal treatment of these wastes, efficiency of sorbents (freshly ground sodium bicarbonate 4 kg/h, activated carbon 150 g/h) injected in the gas stream (2100 Nm<sup>3</sup>/h) to remove heavy metals from flue gas is also estimated.

### 2.3. Sampling and analysis of metals

To determine the quantities of vaporized metal, heavy metal concentration within solids (waste before and after thermal treatment, fines of cyclone, fly ash) are determined using AFNOR NF X31-147 standard method [21]. The mineralized samples

are then analyzed by ICP-AES. The vaporization percentage is calculated according to:

$$\% \text{ vaporisation} = 100 \times \frac{[M]_0 - [M]_f}{[M]_0}$$

with [M]<sub>0</sub>: initial metal concentration in solid sample; [M]<sub>f</sub>: final metal concentration in solid sample.

During experiments in the pilot, sampling trains were performed to determine the removal of heavy metals in gas phase by sorbents. Sampling duration was 3 h. The apparatus used was adapted from American standards US EPA method 5 [22] and USEPA method 29 [23] and French standard method AFNOR XP X43-051 [24] and allowed particulate and gaseous metal emissions to be withdrawn isokinetically from the source and collected. This equipment was installed above sorbent injection and downstream from filters (before evacuation of gas into the atmosphere) as shown on Fig. 2. The sampling train, presented on Fig. 3, consists in:

- Stainless steel probe nozzle;
- Probe liner with a heating system capable of maintaining a probe gas temperature during sampling of 120 °C;
- Borosilicate glass filter holder with glass frit filter support with temperature around filter of 120 °C during sampling, and tare weighed glass fiber filter (effective in the removal of 0.3 μm particles);
- Two impingers of the Greenburg–Smith design with the standard tip filled with a solution of 100 mL HNO<sub>3</sub> 0.5 M;

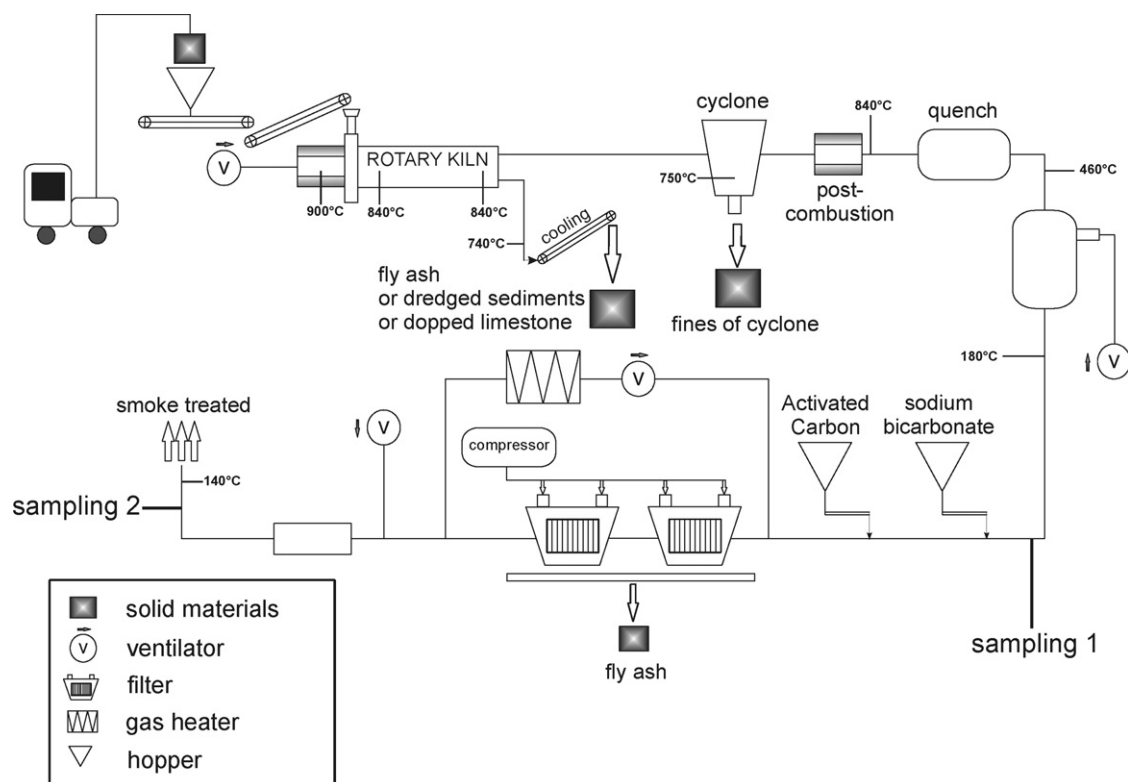


Fig. 2. Schematic diagram of industrial pilot waste incinerator and location of sampling trains.

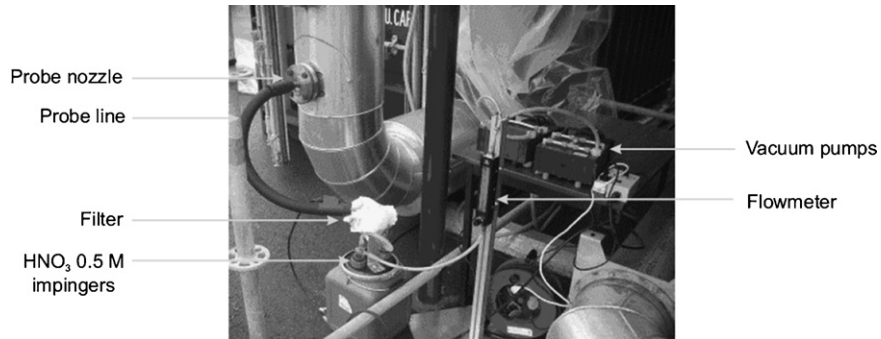


Fig. 3. System for the determination of metal emissions in flue gas.

- Two vacuum pumps and volume flowmeters to maintain isokinetic sampling.

### 3. Results and discussion

#### 3.1. Vaporization at laboratory scale

##### 3.1.1. Vaporization of heavy metals (HM) from fly ash

Results of vaporization percentage of heavy metals contained in fly ash between 600 and 850 °C, presented on Fig. 4, show firstly that vaporization equilibrium is reached in 120 min. We also observe that vaporization percentage increases with increasing temperature. Cd is the most vaporizable metal (55 wt.% at 850 °C) followed by Pb (50 wt.%) and Zn (28 wt.%). The vaporization percentage of Zn is in accordance with literature contrary to vaporization percentage obtained for Cd and Pb.

Jakob et al. [8] also described zinc vaporization percentage near 25% for similar fly ashes. However, these authors noted, at 850 °C, total vaporization of cadmium and lead, which is much more than that observed in our case.

Chan et al. [25] have reported vaporization equilibration times of less than 30 min for the three heavy metals concerned. The variations in vaporization percentage and equilibrium time between our experiments and the literature cited can be attributed

to the differences in experimental conditions used (device and sampling system).

##### 3.1.2. Vaporization of HM from limestone doped with Cd, Pb and Zn

During thermal treatment of doped limestone between 600 and 850 °C, vaporization percentage of heavy metals also increases with increasing temperature and Cd remains the most vaporized compound (38 wt.% at 850 °C) as shown on Fig. 5. The vaporization percentage of lead and zinc in limestone is lower than that in fly ash. It might be due to interaction between metal and this calcium matrix and important moisture of the limestone used. Durlak et al. [26] have shown the negative effect of moisture on the vaporization percentage of lead due to the decrease of free chloride. Increase in waste moisture would shift the speciation of lead from chloride to the oxide form, thus reducing the percentage of vaporized lead.

Finally, vaporization equilibrium of metals in limestone is reached in 45 min. This shorter equilibrium time can be explained by the composition of the matrix. In the other wastes studied (fly ash and dredged sediments), heavy metals are present in the matrix without addition of metal chloride: these metals are fixed to the matrix whereas in limestone the metals are introduced as free chlorides.

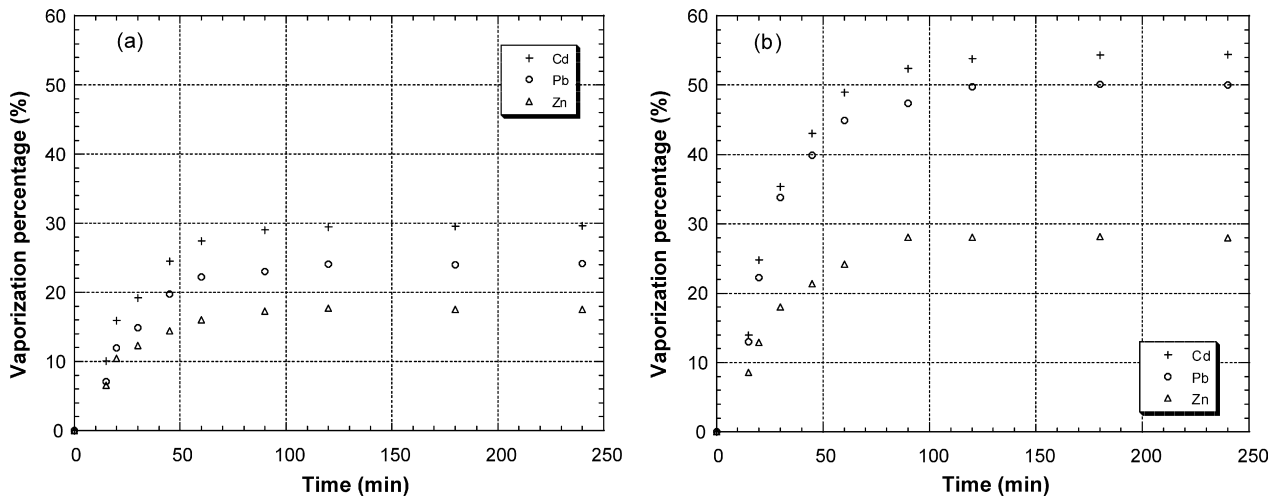


Fig. 4. Vaporization percentage of heavy metals during thermal treatment of fly ash at 600 °C (a) and 850 °C (b).

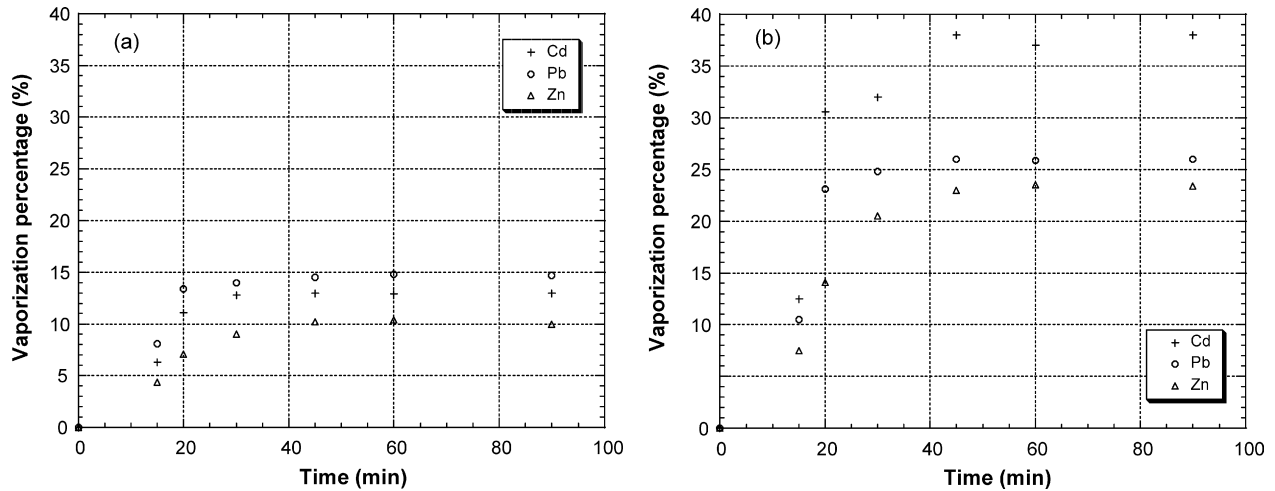


Fig. 5. Vaporization percentage of heavy metals during thermal treatment of doped limestone at 600 °C (a) and 850 °C (b).

### 3.1.3. Vaporization of HM from dredged sediments

In the case of phosphated sediment, vaporization percentages of Cd, Pb and Zn seem to be independent of temperature and are less than 20% at 600 and 850 °C (Fig. 6). This result reveals the effect of phosphate treatment on heavy metals in this organic waste. It was previously reported that phosphorous in refused derived fuel ash had the property of fixing the volatile metals in molten slag only under oxidizing conditions [27]. Kribi [28] has shown the important thermal stability of the metallic phosphates formed during sediment phosphatation. Within this waste, the HM show low vaporization, with Pb showing the highest value of only 20%. Vaporization equilibrium is reached in 90 min for each heavy metal.

### 3.1.4. Effect of chloride on the vaporization of HM

The effect of chloride addition (PVC) on the behavior of heavy metals during thermal treatment of doped limestone and fly ash has been also investigated. The results, presented in Fig. 7, clearly show the accelerating and increasing effect of PVC addition on vaporization percentages of Cd, Pb and Zn. When PVC

is added to wastes, vaporization percentages increase by about 10–15% for all heavy metals investigated at 850 °C. This result is in agreement with the literature [29–32].

Greenberg et al. [29] found that high levels of HCl in incinerator gas streams promote the volatilization of heavy metals by formation of metal chlorides with boiling points lower than 1000 °C. Wang et al. [32] also described the increasing of heavy metal contents in fly ash resulting from the combustion of waste with important Cl concentrations originating from the presence and decomposition of PVC during combustion.

### 3.2. Experiments with an industrial solid waste incinerator

During experiments carried out in industrial pilot, the behavior of heavy metals was evaluated by combustion of limestone doped with Cd<sup>2+</sup> (0.5 wt.%) and fly ash with PVC addition as well as efficiency of sorbents to remove heavy metals in flue gas.

The results of limestone combustion experiments are presented in Table 4. Firstly, Cadmium was mainly concentrated in fly ash and fines of cyclone with or without the addition of PVC.

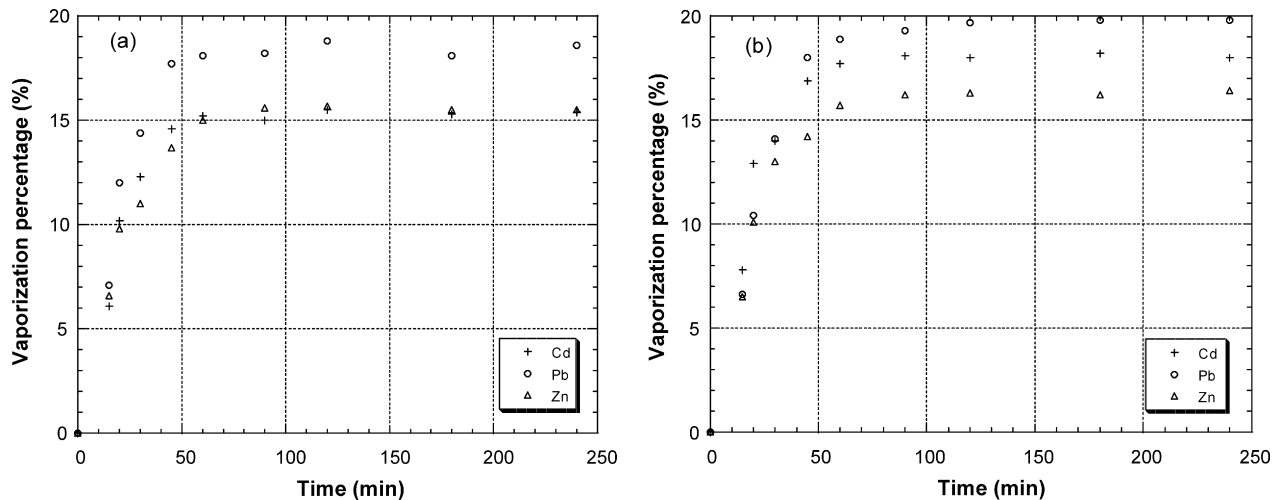


Fig. 6. Vaporization percentage of heavy metals during thermal treatment of phosphated sediments at 600 °C (a) and 850 °C (b).

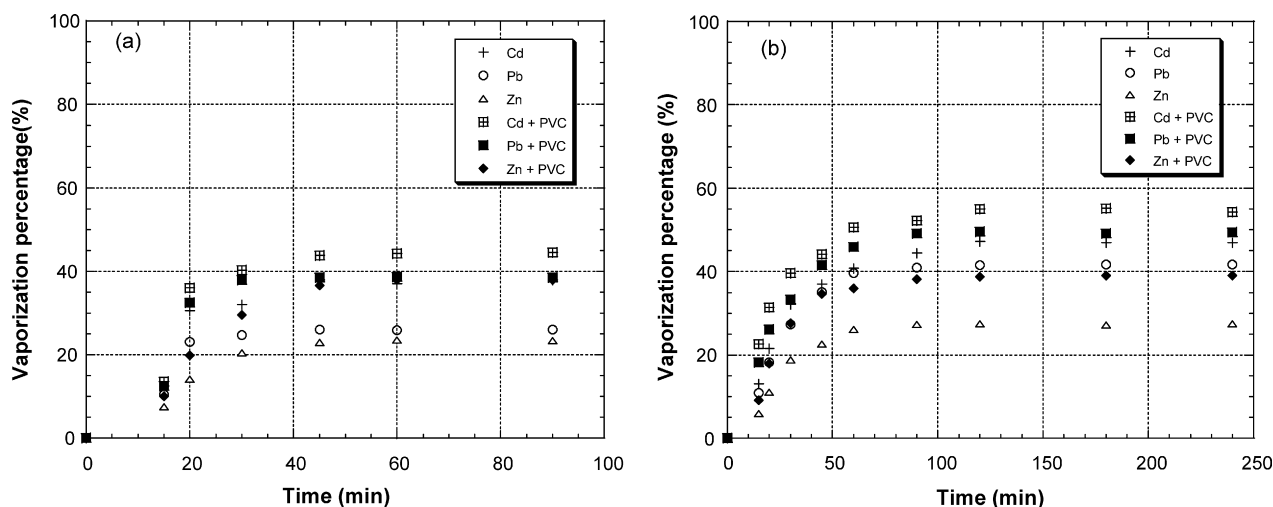


Fig. 7. Effect of PVC addition on vaporization percentage of heavy metals during thermal treatment of doped limestone (a) and fly ash (b) at 850 °C.

Table 4  
Results of combustion of limestone doped with CdCl<sub>2</sub> (Cd<sup>2+</sup>)

Feed	Sorbents	Calcinated residue		Fines of cyclone		Fly ash	
		Mass (kg)	Cd <sup>2+</sup> (g/kg)	Mass (kg)	Cd <sup>2+</sup> (g/kg)	Mass (kg)	Cd <sup>2+</sup> (g/kg)
Limestone 200 kg + Cd <sup>2+</sup> 0.5 wt.%	NaHCO <sub>3</sub> 4 kg/h + AC 150 g/h	82	0.602	20	11.116	24	3.277
	NaHCO <sub>3</sub> 4 kg/h	74	0.457	22	8.061	16	2.307
Limestone 200 kg + PVC 2 kg + Cd <sup>2+</sup> 0.5 wt.%	NaHCO <sub>3</sub> 4 kg/h + AC 150 g/h	87	0.720	21	2.015	20	2.505
	NaHCO <sub>3</sub> 4 kg/h	75	0.799	19	2.173	16	2.307

Table 5  
Results of flue gas sampling during combustion of limestone doped with CdCl<sub>2</sub>

Feed	Sorbents	Sampling 1 (μg/Nm <sup>3</sup> )	Sampling 2 (μg/Nm <sup>3</sup> )	Removal (%)
Limestone 200 kg + Cd <sup>2+</sup> 0.5 wt.%	NaHCO <sub>3</sub> 4 kg/h + AC 150 g/h	0.654	0.003	99.5
	NaHCO <sub>3</sub> 4 kg/h	0.548	0.010	98.3
Limestone 200 kg + PVC 2 kg + Cd <sup>2+</sup> 0.5 wt.%	NaHCO <sub>3</sub> 4 kg/h + AC 150 g/h	1.087	0.011	99.0
	NaHCO <sub>3</sub> 4 kg/h	0.814	0.030	96.3

Results of flue gas sampling (Table 5) show very low Cd concentration in flue gas before exhaust to the atmosphere. When sodium bicarbonate is injected 96.3–98.3 wt.% of Cd is removed. If activated carbon is also injected, efficiency slightly increases and reaches 99.5%.

### 3.2.1. Results of fly ash combustion experiments (Table 6)

The results presented show a much higher vaporization of Cd in fly ash than in limestone doped with Cd. This result is in

agreement with that obtained at laboratory scale and presented in Figs. 4 and 5.

### 3.2.2. Results of flue gas sampling (Table 7)

It can be seen from the results presented that the injection of sodium bicarbonate is efficient for the neutralization of Pb and Cd (89.6 and 86.2%). For Zn, the removal rate of 62.2 wt.% is obtained. Nevertheless, the injection of activated carbon in addition to that of sodium bicarbonate enhances the

Table 6  
Results of combustion of fly ash

Feed	Sorbents	Calcinated residue		Fines of cyclone		Fly ash	
		Pb <sup>2+</sup> (g/kg)	Cd <sup>2+</sup> (g/kg)	Pb <sup>2+</sup> (g/kg)	Cd <sup>2+</sup> (g/kg)	Pb <sup>2+</sup> (g/kg)	Cd <sup>2+</sup> (g/kg)
FA 200 kg + PVC 2 kg	NaHCO <sub>3</sub> 4 kg/h + AC 150 g/h	5.113	0.383	7.893	0.612	12.932	1.027
	NaHCO <sub>3</sub> 4 kg/h	5.890	0.457	5.642	0.520	11.665	0.975

Table 7

Results of flue gas sampling during combustion of Fly Ash

Feed	Sorbents	Removal Pb <sup>2+</sup> (%)	Removal Zn <sup>2+</sup> (%)	Removal Cd <sup>2+</sup> (%)
FA 200 kg +PVC 2 kg	NaHCO <sub>3</sub> 4 kg/h + AC 150 g/h	91.9	94.3	97.3
	NaHCO <sub>3</sub> 4 kg/h	89.6	62.2	86.2

removal of Cd, Zn and Pb (varying from 92–97 wt.%) from flue gas.

#### 4. Conclusion

Vaporization experiments conducted in the laboratory tube furnace with fly ash, doped limestone and dredged sediments have showed that equilibrium was reached in 120 min.

The vaporization percentage of heavy metals increased with increasing temperature. The phosphate treated sediment showed the lowest vaporization percentage of 20% for lead. Except for dredged sediments treated with phosphate (in which heavy metals are stabilized), cadmium was the most vaporizable metal, followed by lead and zinc. These experiments showed also that the addition of organic chloride (PVC) increased the vaporization percentage of heavy metals about 10–15%.

Results obtained in industrial solid waste incineration with limestone or fly ash as feed waste were in agreement with those obtained in the laboratory tube furnace. Results revealed that heavy metals were mainly concentrated in fly ash and cyclone fines (with or without PVC addition). Flue gas sampling showed very low heavy metal concentrations released into the atmosphere. Sodium bicarbonate is an efficient neutralizing agent for gaseous streams which contributes to heavy metal reductions from 62.2% (Zn) to 89.6% (Pb). The addition of activated carbon further eliminates most of the heavy metal emissions, up to 97.3% (Cd), contributing to process safety and reliability. Despite important variations in HM vaporization depending on the presence of chloride or phosphate, the adsorbents used are quite efficient in purifying the gaseous emissions.

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