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# Carbon dioxide sequestration in municipal solid waste incinerator (MSWI) bottom ash

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## Abstract

During bottom ash weathering, carbonation under atmospheric conditions induces physico-chemical evolutions leading to the pacification of the material. Fresh bottom ash samples were subjected to an accelerated carbonation using pure  $CO_2$ . The aim of this work was to quantify the volume of  $CO_2$  that could be sequestrated with a view to reduce greenhouse gas emissions and investigate the possibility of upgrading some specific properties of the material with accelerated carbonation. Carbonation was performed by putting 4 mm-sieved samples in a  $CO_2$  chamber. The  $CO_2$  pressure and the humidity of the samples were varied to optimize the reaction parameters. Unsieved material was also tested. Calcite formation resulting from accelerated carbonation was investigated by thermogravimetry and differential scanning calorimetry (TG/DSC) and metal leaching tests were performed. The volume of sequestrated  $CO_2$  was on average 12.5 L/kg dry matter (DM) for unsieved material and 24 L/kg DM for 4 mm-sieved samples. An ash humidity of 15% appeared to give the best results. The reaction was drastically accelerated at high pressure but it did not increase the volume of sequestrated  $CO_2$ . Accelerated carbonation, like the natural phenomenon, reduces the dangerous nature of the material. It decreases the pH from 11.8 to 8.2 and causes Pb, Cr and Cd leaching to decrease. This process could reduce incinerator  $CO_2$  emissions by 0.5–1%.

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## 1. Introduction

The objective of integrated waste management is to deal with society's waste in an environmentally and economically sustainable way. Moreover, the consequences resulting from the increasing concentration of human-induced greenhouse gases are central to the debate over global change. There is a considerable discussion at scientific and industrial levels about how to limit emission the best.

Under this framework of integrated waste management, thermal treatment represents an appropriate option for reducing the amount of waste to be landfilled. Besides it allows waste hygienization [1]. The major advantage of this process is the dramatic reduction of the waste volume, typically by 90% [2], as well as the energy recovery from burnable waste. However, the incineration of municipal solid waste generates solid residues, bottom ash and fly ash, as well as atmospheric emissions containing about 12% (v/v, dry gas) of  $CO_2$  [3].

These emissions are treated to reduce main pollutants content (HCl, HF, CO, NO<sub>x</sub>, etc.) but the totality of CO<sub>2</sub> is released to the atmosphere. As for solid residues, in some European countries bottom ash and fly ash are separated prior to management. About 50% or more of stockpiled municipal waste incinerator bottom ash is used as a secondary building material or for similar purposes, in road sub-bases and the construction of embankments, wind and noise barriers and other civil engineering structures [4–8]. In France, for 64% of MSWI bottom ash, a period of weathering is required before reuse and MSWI bottom ash is stocked during several months on weathering areas [9]. Actually, MSWI bottom ash is incinerated at high temperatures and thereafter cooled rapidly. The material is then unstable under

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atmospheric conditions. Weathering will therefore stabilize the material by changing its mineralogical characteristics. It will have some positive effects as decreasing heavy metal leaching [10–12]. Carbonation has been recognized to be an important step in weathering process [13–15]. It involves the CO<sub>2</sub> dissolution in water at initially alkaline conditions. This causes pH to decrease and calcite to precipitate until the material is in equilibrium with CO<sub>2</sub>. Moreover, heavy metals (Cd, Cr, Pb, Zn) can be trapped in the newly formed minerals. Calcite is the predominant newly formed mineral during bottom ash maturation, combined with aluminium hydroxides and various sulfates [11,12].

On weathering areas, the CO<sub>2</sub> source is the atmospheric CO<sub>2</sub>. However, it has been proved that the natural carbonation process can be accelerated, using different sources of CO<sub>2</sub> and a few methods have already been experimented at laboratory scale on different MSWI bottom ash and other calcic residue [14–19]. Moreover, accelerated carbonation may represent a proper pre-treatment stage prior to either recycling or landfilling bottom ash [1]. The carbon dioxide source can be either pure CO<sub>2</sub> or any other CO<sub>2</sub>-rich gas such as certain industrial emissions. Consequently, using bottom ash CO<sub>2</sub> sequestration capacity to treat industrial emissions could be another interesting possibility of ash utilization. This process could both help reducing the greenhouse effect and reducing bottom ash storage duration by accelerating weathering reactions [14–17].

To this end, we first investigated accelerated carbonation reaction using pure  $CO_2$ . The objective of the present work was to quantify the volume of  $CO_2$  that could be removed and optimize the carbonation reaction in order to understand what the possibilities and limitations are with a view to technical application.

## 2. Materials and methods

#### 2.1. Bottom ash description

The bottom ash used in this study came from a MSWI facility in France. This plant is equipped with an energy recovery system. The facility is located in the metropolitan area of Lyon and began to operate in 1989. In 2003, it handled 180,000 t of mainly household waste stream and produced both electricity power (40,000 MWh) and heat to a district heating system (100,000 MWh). The resulting solid residues consisted in 43,200 t of bottom ash and 4140 t of fly ash collected by electrostatic filters. The temperature at the ESP filters is about  $250 \,^{\circ}$ C. At present the two residue streams are collected and managed separately. After combustion at  $850 \,^{\circ}$ C, bottom ash is dropped into a water quench tank. After quenching, a magnetic separation is performed.

Samples for experiments have been taken from open storage piles in a weathering area after only a few days of weathering. Their chemical composition in major elements was analyzed (Table 1). Previous studies carried out in our

Table 1	
Chemical composition in major elements of MSWI samples (w	vt.%)

Element	wt.%
SiO <sub>2</sub>	49.3
CaO	16.3
Al <sub>2</sub> O <sub>3</sub>	7.5
Fe <sub>2</sub> O <sub>3</sub>	7.6
MnO	0.1
MgO	2.6
Na <sub>2</sub> O	6.0
K <sub>2</sub> O	1.1
TiO <sub>2</sub>	0.6
$P_2O_5$	1.2
TOC	1.5
S tot	0.3
Cl	0.4

laboratory over a period of three years showed that this composition is quite constant [14,15].

#### 2.2. Determination of water content

The water content of bottom ash was tested by weighting a sample, drying the sample at  $105 \,^{\circ}$ C for 24 h and weighting it again.

#### 2.3. Accelerated carbonation of bottom ash

The experiments were performed at room temperature in a high pressure vessel. It consisted in a metallic cylinder of 150 mL internal volume connected to an industrial grade  $CO_2$ container.  $CO_2$  pressure in the reaction chamber was monitored through manometers with a pressure range of 1–20 bar.

About 100 g of MSWI bottom ash were weighted and introduced in the vessel. After removing the air initially present in the chamber, the chosen  $CO_2$  pressure was established. The sample was removed after determined reaction times, weighted and reintroduced in the vessel. Each experiment was stopped when the sample weight remained constant, that is to say when the carbonation reaction had ended.

Several parameters were tested to characterize carbonation efficiency:

- reaction time;
- carbon dioxide pressure (2, 3, 5, 7, 11, 17 bar);
- moisture content of ash.

Samples were sieved through a 4 mm-sieve. For a few experiments, unsieved material was also tested.

The initial humidity of bottom ash was 25% (w/w). Different samples ranging from 5% to 25% moisture content were prepared by drying fresh material at 105 °C. Drying durations had been determined by preliminary tests.

## 2.4. Leaching tests

The leaching tests were performed at an initial liquid to solid ratio (L/S) of  $10 L kg^{-1}$  with distilled water. Sam-

ples were rotated at 11 rpm during 24 h. After this leaching time samples were filtered at 45  $\mu$ m and the pH was measured. Metal concentrations in the leachates were analyzed by inductively coupled plasma and atomic emission spectrometry (ICP-AES) with a Jobin Yvon Horiba analyzer.

## 2.5. Thermal analyses

Using a Setaram LABSYS TG DSC 1600 thermal analyzer, two techniques of thermal analysis were applied, viz. thermogravimetry (TG) and differential scanning calorimetry (DSC). This apparatus allowed to record, as a function of temperature, both the mass sample evolution (TG), and the heat flow variation between the sample and a reference (DSC). Thermograms provide information about chemical reactions (e.g. oxidation and decomposition), physical processes (e.g. vaporization, sublimation, desorption), and thermal behavior (e.g. exothermic and endothermic reactions).

For these analyses, a mass of about 20 mg of the ash sample was placed inside an alumine crucible. The samples were heated linearly at a heating rate of  $10 \,^{\circ}C/min$  to  $1000 \,^{\circ}C$  under an inert atmosphere of argon. The temperature of the sample and reference were recorded by a platinum/rhodium thermocouple (type S), and a high-precision balance registered the potential weight loss due to evaporation/decomposition of the sample.

Thermal analyses were repeated three times, to test samples homogeneity and also the reproducibility of the instrument.

#### 3. Results and discussion

#### 3.1. Carbonation

Carbonation mechanism is often described as an at least two steps process including a prior  $CO_2$  absorption in water, followed by the carbonation reaction in aqueous medium [19,20]. During the experiments,  $CO_2$  is dissolved in pore water and reacts with some components of MSWI bottom ash to produce carbonated species. The main reaction is known to be the portlandite (Ca(OH)<sub>2</sub>) carbonation in aqueous medium, which corresponds to the following expression [20–22]:

$$Ca(OH)_{2(aq)} + CO_{2(aq)} \rightarrow CaCO_{3(s)} + H_2O_{(l)}$$
(1)

The formation of solid calcite (CaCO<sub>3</sub>) induces an increase in the sample mass, which can be directly linked to the amount of trapped carbon dioxide. The experimental difference between the initial and the final mass ( $\Delta m$ ) can be converted in volume of absorbed CO<sub>2</sub> per kg of dry matter (DM) using the expression above:

$$V_{\rm CO_2} = \left(\frac{RT}{P}\right) \times \frac{\Delta m/M_{\rm CO_2}}{(100-h) \times m_{\rm i}} \tag{2}$$

Fig. 1. Gain of carbonate content as a function of carbonation time for five different  $CO_2$  pressures ranging from 2 to 17 bar (moisture content: 20%, w/w).

where  $V_{\text{CO}_2}$  is the volume of absorbed CO<sub>2</sub> (m<sup>3</sup>/kg DM) at room temperature and for a CO<sub>2</sub> pressure of  $1 \times 10^5$  Pa;  $R = 8.1345 \text{ J K}^{-1} \text{ mol}^{-1}$ ; *P* the CO<sub>2</sub> pressure ( $1 \times 10^5$  Pa); *T* is the ambient temperature (293 K);  $M_{\text{CO}_2}$  the CO<sub>2</sub> molar weight (kg mol<sup>-1</sup>); *m*<sub>i</sub> the initial mass of the sample (kg); *h* the moisture content of the sample (%).

## 3.2. Influence of CO<sub>2</sub> pressure

In order to evaluate the effect of  $CO_2$  partial pressure on the bottom ash carbonation, several experiments were performed at pressures ranging from 2 to 17 bar. The moisture content of samples was 20% (w/w). Fig. 1 shows the amount of formed carbonates as a function of carbonation time for several pressures during the first 3 h.

From the shape of these curves, it is obvious that  $CO_2$  pressure greatly influences the reaction kinetic. The duration to obtain a complete carbonation ranges from 3 h 30 min under 17 bar to more than 2 days under 2 bar. In a recent paper, van Gerven et al. [16] performed experiments of accelerated carbonation at  $CO_2$  partial pressures of 0.1 and 0.2 bar, and the reaction was completed in less than one week. On weathering areas, the  $CO_2$  partial pressure is only 0.00036 bar. In these conditions, a much longer duration will be required for the completion of the carbonation reaction. It has been demonstrated that after a 7 months weathering period bottom ash could still absorb  $CO_2$  [15].

For a better comparison of these two experiments (2 and 17 bar), another plot is proposed on Fig. 2 in which the time has been normalized. Each time value has been divided by the duration required to have a complete reaction (210 min for 17 bar and 3060 min for 2 bar) in order to plot the graphs on a same scale (ranging from 0 to 1) and show the entire reactions. We can see that  $CO_2$  pressure has no effect on the total amount of  $CO_2$  that reacts. Whatever the  $CO_2$  pressure, the maximum and limit change in mass is close to 3.2%, which corresponds to a volume of 24 L/kg DM of trapped  $CO_2$  Eq. (2). The  $CO_2$  pressure does not affect the carbonation equilibrium from a thermodynamic point of view, but it plays a major role on the kinetic of the whole process.





Fig. 2. Gain of carbonate content as a function of normalized carbonation time for CO<sub>2</sub> pressures of 2 and 17 bars (moisture content: 20%, w/w).

The limit of CO<sub>2</sub> solubility ([CO<sub>2</sub>]) in the pore water is directly related to its partial pressure ( $P_{CO_2}$ ) in the surrounding gaseous phase through Henry's law:

$$[\mathrm{CO}_2] = k_{\mathrm{CO}_2} \times P(\mathrm{CO}_2) \tag{3}$$

where  $k_{\text{CO}_2}$  is Henry's constant, only depending on the temperature ( $k_{\text{CO}_2} = 34.13 \text{ mol m}^{-3} \text{ bar}^{-1}$  at 20 °C).

Taking into account this relation, the calculation gives  $[CO_2]$  values ranging from 68 mol m<sup>-3</sup> to 580 mol m<sup>-3</sup>, respectively under 2 and 17 bar CO<sub>2</sub> pressure. The increase in CO<sub>2</sub> concentration in the aqueous phase obviously accelerates the carbonation reaction. However, it does not change the quantity of formed carbonates.

From our results, we can suppose that the slowest stage of the reaction is the CO<sub>2</sub> dissolution in the pore water and its diffusion. As far as the amount of formed carbonates is concerned, the limiting parameter seems to be the maximum available calcium ions in aqueous medium, which strongly depends on physico-chemical parameters of water (temperature, pH, ionic strength, etc.) and on calcium speciation (hydroxide, sulphate, chloride, phosphate, etc.). It seems that among the different calcium species, the portlandite (calcium hydroxide) is the main mineral that can be dissolved and carbonated [15]. Bertos et al. [19] gave similar results by submitting several waste materials of different Ca content to accelerated carbonation. It appeared that the degree of carbonation was strongly dependent upon the calcium content. However, some waste did not follow this tendency. This has been attributed to a lack of calcium hydroxide and/or the deposit of certain components that might influence the effective diffusivity and reactivity of the carbon dioxide.

## 3.3. Thermal analyses

Samples were analyzed by simultaneous TG/DSC before and after a complete carbonation. Curves showed a good repeatability whatever the carbonation conditions. Examples of obtained curves are shown on Fig. 3. The DSC curves are plotted in mW per mg of material on the first axis, and the mass losses (in percentage) are plotted on the second axis.



Fig. 3. TG/DSC curves of uncarbonated and carbonated bottom ash as a function of temperature.

The DSC curves first present an endothermic effect at 90 °C accompanied by a mass loss of about 20%. It corresponds to the sample dehydration.

Between 200 °C and 600 °C TG/DSC curves show a slight decrease and an exothermic phenomenon. This is attributed to the pyrolysis of the 1.5% of organic matter.

Then DSC curves present a small endothermic peak from  $600 \,^{\circ}$ C to  $750 \,^{\circ}$ C due to the dissociation of carbonates. It is accompanied by a mass loss corresponding to the sample content in carbon dioxide according to the following dissociation equation:

$$CaCO_{3(s)} \rightarrow CaO_{(s)} + CO_{2(g)}$$
 (4)

The latter is about 3.3% (DM) for fresh bottom ash and about 6.2% (DM) for carbonated bottom ash. With those results we can calculate the quantity of carbonates formed during accelerating ageing.

The mass difference is about 2.9%. This value is in agreement with the one obtained on the accelerated ageing set up, where the gain of mass was about 3.2%. It confirms that the gain of weight of samples was really due to a chemical carbonation and not to physical adsorption of CO<sub>2</sub>.

## 3.4. Influence of humidity

The effect of initial material moisture content on carbonation was studied by testing 4 mm-sieved samples under a  $CO_2$  pressure of 2 bar at ambient temperature. Moisture content of samples was measured before and after the experiment to make sure that it did not vary during carbonation.

Fig. 4 shows the volume of absorbed CO<sub>2</sub> as a function of bottom ash moisture content after a complete carbonation.

Moisture content is an important parameter of the reaction. We can see that the reaction does not occur for totally dried samples. This result was expected since the carbonation reaction occurs in aqueous phase. An ash humidity of 15%(w/w) seems to be the best option.

This result is particularly of interest and should be taken into account on the bottom ash weathering areas during summer when bottom ash piles are very dry and for covered areas,



Fig. 4. Volume of absorbed  $CO_2$  for carbonated samples at 2 bar as a function of bottom ash moisture content.

that are developing, where piles are protected from rain and not regularly humidified.

Above 15% there is a decreasing tendency of volume of trapped CO<sub>2</sub>. This could indicate that the reaction mainly occurs in the pores of the solid matrix. Above this value, the excess water is probably intergranular water. Then there is less contact between bottom ash and dissolved CO<sub>2</sub>, whose concentration in the aqueous medium only depends on external CO<sub>2</sub> pressure and temperature.

#### 3.5. Influence of sieving

An experiment with unsieved MSWI bottom ash was carried out in order to compare the results with 4 mm-sieved samples. Both samples were tested at a pressure of 1 bar until a complete carbonation. They had the same initial moisture content of 20%.

The volumes of absorbed  $CO_2$  were 24 L/kg DM for the 4 mm fraction of bottom ash and 12.5 L/kg DM for the unsieved sample. The sieved sample absorbs a larger quantity of  $CO_2$ , nearly twice. Indeed the elementary composition of bottom ash fractions varies according to size particles. It has been shown that the Ca content increases and the Si content decreases in bottom ash fine fractions [23–25]. During bottom ash sieving, a part of glass, ceramics and other materials that do not react with  $CO_2$  is removed and the proportion of Ca increases, which enhances the carbonation reaction.

#### 3.6. Chemical changes of leachates

These experiments were performed in order to assess briefly the effects of accelerated carbonation on leachates and compare them to the natural phenomenon effects.

## 3.6.1. pH evolution

The leaching tests performed on both fresh and carbonated samples gave pH values of 11.8 and 8.2 for the respective leachates.

The pore water of fresh bottom ash has generally very basic pH values, mainly due to the presence of dissolved portlandite  $(Ca(OH)_2)$  [26,27]. Portlandite is formed in bottom ash as a result of calcium oxide (CaO) hydrolysis at the exit of the furnace during the water quenching step. pH values

around 12 are in agreement with those of saturated calcium hydroxide solutions.

It has been also suggested that gibbsite  $(Al(OH)_3)$ , gypsum (CaSO<sub>4</sub>, 2H<sub>2</sub>O) and ettringite  $(Ca_6Al_2(SO_4)(OH)_{12}, 26H_2O)$  have an influence on the alkalinity of fresh bottom ash [26–28].

As already written (Section 3.1), during ageing treatment, portlandite reacts with dissolved  $CO_2$  to form calcite (CaCO<sub>3</sub>). This phenomenon leads to a decrease in pH by consuming the alkaline compounds. The final pH value is governed by the dissolved hydrogenocarbonates (HCO<sub>3</sub><sup>-</sup>).

In the case of naturally aged bottom ash, the pH of the material is reasonably well described by the presence of solid calcite in equilibrium with dissolved atmospheric  $CO_2$  [29].

## 3.6.2. Trace element leaching

The effect of the accelerated ageing treatment was also evaluated on the mobilization of some hazardous heavy metals (Cr, Pb, Cd) in the leachates at their natural pH (11.8 and 8.2).

Cr, Pb and Cd concentrations in fresh bottom ash leachates were 0.022, 0.593 and 0.012 mg L<sup>-1</sup>, respectively. In carbonated bottom ash, Cr and Pb leachates concentrations decreased to 0.005 and 0.026 mg L<sup>-1</sup>, respectively. The Cd concentration was below the analytical detection limit ( $4.6 \,\mu g \, L^{-1}$ ). Accelerated carbonation had then a positive effect on the leaching of these elements.

Two combined phenomena can explain those results. First, pH is a dominant parameter in metal leaching. After accelerated ageing, the natural pH of bottom ash (8.2) is closed to the minima of Cr, Pb and Cd solubility. Furthermore, it has been shown that trace elements leaching is also reduced by sorption in new formed minerals: Cd and Pb have a strong affinity with calcite. They also form complexes with Fe and Al (hydr)oxides. Some other metal-bearing new formed minerals have also been found in weathered bottom ash such as oxides, phosphates and sulphates of Cr and Pb [11,20,21,30].

These experiments concerning both pH and trace elements leaching confirm that the accelerated carbonation treatment has the same positive effects as natural weathering. Consequently, accelerated carbonation could contribute to upgrade some properties of bottom ash.

## 3.7. Discussion and potential application

Taking into account that the incineration of 1 t of municipal solid waste generates approximately  $600 \text{ Nm}^3$  of residual pure CO<sub>2</sub> and about 250 kg of bottom ash, we can estimate the CO<sub>2</sub> sequestration potential of solid residues. Calculations give a result of 0.5–1% of CO<sub>2</sub> reduction by mineralization under carbonates forms. This ratio can appear relatively low, but the resulting short stabilization time of MSWI bottom ash can give a benefit in the whole treatment process, before reuse. If such a treatment is developed, the contact between bottom ash and emissions must be the last

step of gas treatment, in order to prevent the adsorption of gaseous pollutants that could ban bottom ash from recycling.

To optimize the process, it would be better to keep a quite constant moisture content at around 15% (w/w) and to grind the material in order to increase the exchange surface.

The Kyoto Protocol will commit the Community and its Member States to reducing their emissions of greenhouse gases by 8% compared to 1990 levels in the period from 2008 to 2012. Member States should ensure that the operators of certain specified activities hold a greenhouse gas emissions permit and that they monitor and report their emissions of greenhouse gases specified in relation to those activities. The excess emissions penalty shall be  $\in$  100 for each tonne of carbon dioxide equivalent emitted by that installation for which the operator has not surrendered allowances. The emissions allowance trading is allowed under certain conditions [31].

It can be precised that CO<sub>2</sub> emissions from waste thermal treatment represent about 2% of French global CO<sub>2</sub> emissions [3]. The French MSWI bottom ash production is around 3 Mt (DM) per year [9]. According to our result, this would represent a sequestration potential of about 73,000 t of carbon dioxide equivalent. This could correspond to  $\in$ 7,300,000 savings if operators exceed their allowance or a benefit if they can sale a part of it to other installations.

Another major advantage of this process would be a better bottom ash stock management for the production of secondary raw construction material.

Indeed, on weathering areas, maturation duration is long and variable (it depends on bottom ash characteristics evolution). Accelerated carbonation would significantly reduce storage duration. Moreover, it would ensure that the supplied bottom ash is totally carbonated and stabilized before reuse.

## 4. Conclusion

Two important effects of accelerated carbonation have been identified. This process has the major advantage of chemically binding  $CO_2$  and at the same lowering the hazardous nature of bottom ash. A correctly conducted accelerated carbonation process can chemically block the leaching of Cd, Cr and Pb, which are the main regulated heavy metals.

The CO<sub>2</sub> volume that can be trapped in the material has been quantified and is on average 12.5 L/kg DM of MSWI bottom ash. Tests performed to improve the efficiency of the process showed that the CO<sub>2</sub> partial pressure can drastically modify the kinetic of the reaction: the higher the CO<sub>2</sub> partial pressure, the faster the reaction. However, it does not increase the volume of trapped CO<sub>2</sub>. As far as humidity is concerned, a moisture content of 15% (w/w) seems to be the best option. The volume of trapped CO<sub>2</sub> can also be optimized by sieving the material or recycling glass and ceramics before incineration.

Accelerated carbonation using the stack gas of MSW incinerators that contains around 12% CO<sub>2</sub> or landfill bio-

gas that contains 35%  $CO_2$  could be a realistic technique for the treatment of bottom ash.

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79

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