Carbonation of Municipal Solid Waste Incineration Fly Ash and the Impact on Metal Mobility

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Abstract: Fly ash from municipal solid waste incineration (MSWI) is considered as hazardous waste that calls for a robust, reliable, and reasonable treatment technique. This investigation aims to assess the impact of CO₂ partial pressure, water addition, time, and temperature on the stabilization of MSWI fly ash with particular emphasis on Pb, Zn, Cd, and Cr. Carbonation and element mobility were studied by applying thermal analysis and leaching assays on fly ash samples treated according to a 2⁴ factorial design. The relationship between the factors and the response variables was evaluated using partial least squares modeling. Chemical equilibrium calculations were performed so as to complement the experimental findings. Decalcification of carbonated fly ash in a typical Swedish landfill was estimated at 0.13 mm·yr⁻¹. Treatment through carbonation reduced the availability of Pb and Zn about 100 times and also the carbonate alkalinity of 7.4 eq·(kg·FS)⁻¹ (FS represents the fixed solids) was remedied successfully. However, shortcomings that need to be resolved are the remobilization of Cr with time and the mobilization of Cd.

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Introduction

Municipal solid waste incineration (MSWI), including air pollution control (APC), is a way to separate inorganic contaminants from bulk municipal solid waste and to collect them in a solid waste stream. The APC stream corresponds to only ~2.5% by weight of the incinerator feed, but contains ~90% of Hg, ~86% of Cd, ~40% of Zn, and 31% of Pb (Chandler et al. 1997). Once these pollutants are concentrated, they should be deposited with care.

Owing to the content of hazardous components, different countries have tightened up their legislation regarding the handling of APC residues. The European Commission (EC), for example, classifies APC residues from MSWI as hazardous waste [European Union (EU) 1991]. As such it has to be disposed of at a landfill that fulfills strict technical and monitoring requirements (EU 1999). In addition, the EC Landfill Directive (EU 1999) requires that all waste undergo pretreatment to reduce its hazardous nature. Japan's Waste Disposal and Public Cleansing Law [Ministry of Health and Welfare (MHW) 1991] stipulates that for all MSW incinerators with a capacity equal to or greater than 5 t·day⁻¹, APC residues must not be disposed of unless treated.

In spite of the need for APC residue treatment, no technique has gained wide acceptance. Thermal treatment in electric, burner, or blast furnaces strains resources, amounting to operation costs of up to \$500 per ton (Ecke et al. 2000). Cementitious stabilization and solidification almost triple the final waste mass while microencapsulation, which uses organic additives such as bitumen, paraffin, and polyethylene, requires expensive equipment and skilled labor (Chandler et al. 1997). Chemical stabilization with inorganic additives is still under development and assessment. Different additives were tested (Chandler et al. 1997), such as phosphates (Eighmy et al. 1997), sulfides, lime (Reardon and Della Valle 1997), clays, and carbonates. In the long term, the approach of stabilizing rather than solidifying APC residues might be superior, provided that the treated waste is placed in an environment not counteractive to the fixation of pollutants.

This work presents a new attempt, adopted from geochemical processes, to stabilize APC residues. Based on a treatment-oriented characterization of typical MSWI fly ash, it is suggested assessing carbonation as a stabilization method (Ecke et al. 2002). Using the same waste as for characterization, the objective of the present investigation was to study factors that possibly control carbonation and its impact on metal mobility. With reference to promising carbonation experiments on coal fly ash and spent shale (Reddy et al. 1991, 1994; Tawfic et al. 1995), the working hypothesis was that the availability of crucial metals (Ecke et al. 2002) such as Pb and Zn, and possibly Cr and Cd, can be lowered to meet the strictest EC standards for landfill leachate (Hjelmar et al. 1994).

Material and Methods

The fly ash used in this investigation was sampled from the MSW incinerator Ålidshemverket in Umeå, Sweden. The flue gas is treated in several steps. Ammonia is injected into the combustion chamber to reduce nitrous fumes. Sodium sulfide is added to remove Hg and calcium hydroxide is added to neutralize acid com-

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Fig. 1. Experimental setup used for the carbonation of fly ash

ponents. Bag fabric filters are used to remove particulate matter. This material is the research object called fly ash. During one week of regular plant operation, about 5 kg of fly ash were sampled three times per day and once per shift. The material was mixed and quartered to obtain subsamples.

Carbonation

In the laboratory, 10 g of fly ash were carbonated in 16 experimental runs. They were varied according to a two-level full factorial design (Box et al. 1978), where the impacts of four factors were studied, viz., addition of water, partial pressure of CO_2 , temperature, and time. The fly ash was used as received or mixed with water. The material was placed in glass reaction tubes (Fig. 1). By applying a peristaltic pump, the daily total gas flow through the tubes was set at 2.2 L. The gas became saturated with moisture by passing through a washbottle. The water in the wash-bottle was in chemical equilibrium with the reaction gas (open system), either air or air mixed with CO_2 , before running the experiments. During carbonation, the temperature was kept constant using laboratory ambient conditions (20°C) or an oven (60°C). The experiments were terminated after either 4 or 40 days. The factor levels are summarized in Table 1.

Carbonated samples were crushed and homogenized with a pestle before analysis.

Table 1. 2^4 Full Factorial Design for the Carbonation of Fly Ash

Experiment number	Time (days)	Temperature ($^\circ\text{C}$)	CO_2 (% by volume)	H_2O addition (kg/kg)
1	4	20	0.03	0.0
2	40	20	0.03	0.0
3	4	60	0.03	0.0
4	40	60	0.03	0.0
5	4	20	50	0.0
6	40	20	50	0.0
7	4	60	50	0.0
8	40	60	50	0.0
9	4	20	0.03	0.5
10	40	20	0.03	0.5
11	4	60	0.03	0.5
12	40	60	0.03	0.5
13	4	20	50	0.5
14	40	20	50	0.5
15	4	60	50	0.5
16	40	60	50	0.5

Analytical Methods

Thermal analysis (Netzsch STA 409C) was performed in air by applying thermogravimetry (TG) and differential thermal analysis (DTA) in a temperature range of 20 – 1200°C at a heating rate of $10\text{ K}\cdot\text{min}^{-1}$. The crucible material was aluminum oxide ceramic. Differential thermal analysis and the first derivative of the TG curves were used to determine the temperature range for the decomposition of different phases.

pH_{stat} titrations (Cremer and Obermann 1992) were performed under Ar atmosphere using an initial liquid-to-solid (L/S) ratio of $40\text{ l}\cdot(\text{kg fixed solids})^{-1}$. One molar nitric acid, used as titrant, was added by a computer-controlled automatic titrator (Radiometer ABU 900). The titration time was set at 12 h. The titration levels were pH 8.3 and 4.5. Total titrant addition was recorded. All suspensions were filtered ($0.45\text{ }\mu\text{m}$).

Zero-headspace water leaching assays were performed on a laboratory roller mixer. The L/S ratio, leaching time, and sample treatment were identical to the pH_{stat} titrations. The final pH of the leachate (denoted here as pH_0) was determined.

The elemental compositions of the leachates from both pH_{stat} titrations and zero-headspace leachings were analyzed using inductively coupled plasma mass spectrometry at an accredited laboratory (SGAB Analytica, Luleå, Sweden).

Chemical equilibrium calculations were performed using the computer program *PHREEQC-2* (Parkhurst and Appelo 1999) and applying the LLNL thermodynamic database developed by the Lawrence Livermore National Laboratory, Livermore, Calif.

Statistics

Partial least squares (PLS) modeling (Cooley and Lohnes 1971; Wold 1989) was used to find the relationships between the response variables and the factors. Cross validation was applied to identify the appropriate number of principal components (Wold et al. 1987; Wold 1989).

Results and Discussion

A former characterization (Ecke et al. 2002) of the same MSWI fly ash showed that the material was both representative and homogeneous. The total contents of abundant elements were within the minimum-maximum ranges of dry/semidry APC residues as compiled by the International Ash Working Group (IAWG) (Chandler et al. 1997). For elements at lower concentrations, the deviations from the IAWG data were less than one order of magnitude. The standard deviation for the sum of all elements was less than 1.5%. $2.6 \pm 1.5\%$ per weight of the dry solids was not identified, which can be due to elements such as N and Se that were not analyzed.

Thermal analysis of the 16 treated samples showed that the fly ash consisted of three major volatile phases, viz., pore water, hydrated water, and carbonates (Fig. 2). Evaporation of pore water is an endothermic process and was observed up to $\sim 230^\circ\text{C}$. While hydrated water was liberated within a broad temperature range between ~ 230 and $\sim 640^\circ\text{C}$, the combustion of organics occurred at $\sim 470^\circ\text{C}$. The latter was characterized by small positive peaks in the DTA curves. However, the mass loss due to organics was estimated at less than 1.5% by weight compared with a total mass loss of up to about one-third at 550°C . This outcome shows that the loss on ignition analyzed according to standard methods, e.g., CEN (2000), is inappropriate to determine the content of organics in fly ashes. Fly ash dried at 105°C still

Fig. 2. Thermograms for 16 carbonated fly ash samples (Table 1). Each diagram illustrates both the thermogravimetric analysis and the differential thermal analysis. Positive and negative ΔT peaks qualitatively indicate exothermic and endothermic reactions, respectively.

contained significant amounts of mechanically and chemically bound water, hence dominating the loss on ignition (Fig. 2).

Carbonates were decomposed in a temperature range of ~ 640 – 840°C . To avoid underestimating both the formation of such new phases and the leachability of metals, the concentrations of the components were related to the fixed solids (FSs). In the following, the term FS is defined as the mass remaining after the evaporation of water, the oxidation of organics, and the decomposition of carbonates.

The data matrix consisted of 16 observations on four factors and responses from TG as well as leachate analyses from three different leachings including multiple element analyses. Due to the complexity of the data matrix itself and possible variable interactions, the data evaluation was performed by means of PLS modeling. Input data were expressed on the basis of FSs. Variables not at all affected by the factors were excluded from the modeling. $\log_{10}$ transformations were performed on skewed variable data sets.

The PLS modeling resulted in two principal components comprising 43 and 13% of the data variation. The loading plot (Fig. 3) illustrates the impact of the factors on the response variables. For the following discussion and its conclusions, it is important to keep in mind that the received model covers 56% of the data variation, i.e., there still remains unresolved variability, due to either noise or the impact of factors that were not considered.

The carbonate content depended greatly on the concentration of CO_2 in the gas phase, but also on water addition and time. The content of pore water was dominated by the amount of added water, whereas time and temperature had a somewhat negative effect. Both CO_2 , water additions, and time increased the content of hydrates. The effects of the factors CO_2 and time are quantified in Fig. 4.

The factors CO_2 and time both had a negative effect on the pH of the zero-headspace leachates (pH_0) and the acid addition at pH_{stat} 8.3 (carbonate alkalinity Alk_C) (Figs. 3 and 5). The maximum difference observed in pH_0 was almost 5 units from 13.1 (observation No. 4) to 8.5 (observation No. 16), illustrating that

Fig. 3. Partial least squares loadings illustrated in three plots. Factors are set off with arrows. Notations for response variables: $_0$ means zero-headspace leaching, $_C$ means pH_{stat} leaching at pH 8.3.

carbonation at its extreme (all factors high) resulted in a carbonate system dominated by bicarbonates.

Carbonation also affected the leaching behavior of several metals as investigated by zero-headspace assays (notation $_0$) and pH_{stat} 8.3 assays (notation $_C$) (Fig. 3). Without any pH adjustment ($_0$), two major element groups were identified. For the first,

Fig. 4. Evolution of the content of carbonate, hydrated water, and pore water in $\text{g} \cdot (\text{kg} \cdot \text{FS})^{-1}$ as function of CO_2 concentration and time using the partial least squares model. Constant factor levels: water addition $0.50 \text{ kg} \cdot \text{kg}^{-1}$ and temperature 20°C .

metal availability decreased with an increased concentration of CO_2 and time. For the second group, the same factors had the opposite effect, i.e., they increased the mobility. The first group depicts three typical carbonate formers, viz., Ca, Ba, and Pb forming calcite, witherite, and cerussite. In addition, the two remaining elements of that group, Zn and Cu, can be bound as carbonaceous phases; Zn preferably as smithsonite and Cu as malachite. The grouping demonstrates that carbonation demobilized environmentally relevant metals such as Pb and Zn while mobilizing Cd, Al, Ni, and Mn. Cr was an exception because its availability was almost unaffected by CO_2 ; however, time appeared to have a positive impact while water addition had a negative influence (Figs. 3 and 5).

These findings complement former investigations (Ecke et al. 2002). With respect to the strictest EC limit values for landfill leachate, Ecke and co-workers identified in the cited study Pb, Zn, Cr, and Cd as the potential major pollutants in fly ash from MSWI. Based on chemical equilibrium calculations, it was postulated that carbonation leads to a reduced mobility of Pb and Zn due to the predominance of PbCO_3 ($6 < \text{pH} < 9$) and $\text{Zn}(\text{OH})_2$ ($9 < \text{pH} < 11$), respectively, while the availability of Cr is a function of both carbonation and electron activity. A change in Cd mobility could not be deduced.

The two investigations are in good agreement with regards to Pb and Zn. As an effect of carbonation, the leachability of Pb and Zn decreased by two orders of magnitude and yielded for both ~ 1

Fig. 5. Leachate pH and leachability $[\text{mg} \cdot (\text{kg} \cdot \text{FS})^{-1}]$ of Al, Cd, Cr, Pb, and Zn determined by zero-headspace leachings. Responses illustrated as function of CO_2 concentration and time using the partial least squares model. Constant factor levels: water addition $0.50 \text{ kg} \cdot \text{kg}^{-1}$ and temperature 20°C .

$\text{mg} \cdot (\text{kg} \cdot \text{FS})^{-1}$ at a minimum (Fig. 5). Carbonation increased the mobility of Cd (Fig. 5) possibly due to the transition from $\text{Cd}(\text{OH})_2$ to CdCl_2 (Ecke et al. 2002). From an environmental point of view, this observation is critical and calls for extra attention in future investigations. According to Ecke et al. (2002), the mobility pattern of Cr can be attributed to pe rather than other factors such as CO_2 or pH. At $\text{pe} > 4$, highly mobile hexavalent Cr(VI) dominates the fly ash leachate system. As a function of time, the fly ash samples might have been treated toward more oxidizing conditions due to the impact of atmospheric O_2 , i.e., the mobilization of Cr was favored. On the other hand, mixing fly ash with water might have facilitated the chemical reaction with other elements and, thus, the reduction of Cr(VI) to species in the trivalent oxidation state, which is characterized by a much lower mobility, as in $\text{Cr}(\text{OH})_3$.

Ecke et al. (2002) found that for fresh fly ash leached at $\text{pH}_{\text{stat}} 8.3$, the leachability of Pb decreased by three orders of magnitude compared with zero-headspace leachings (pH_0). Other metals are barely affected and remain at low levels, e.g., Al, Cd, Cu, Ni, and Zn. Except for Al, the carbonation led to an even more reduced availability at $\text{pH}_{\text{stat}} 8.3$ (C in Fig. 3), especially for all crucial metals, i.e., Pb, Zn, Cd, and Cr. Among the latter, the addition of water appeared to have a positive effect on the availability of Zn and Cd; however, the increase in mobility of Zn and Cd diminished with time.

Some observations were also likely based on or had interference from secondary effects. Significant impacts might be due to the formation of calcium silicate hydrates (C-S-H) or calcium aluminosilicate hydrates (C-A-S-H) (Ecke et al. 2002). Through bonding and adsorption, the crystalline matrix retains the metals. However, carbonation can cause the decomposition of silicates as observed during the weathering of natural silicates (Appelo and Postma 1999), which could explain the anomalous behavior of Al (Fig. 3). An effect that enhances the sequestering of a variety of cations (Pb, Cd, Cu, Zn, etc.) is probably the adsorption onto CaCO_3 leading to coprecipitation (Morse and Bender 1990; Stumm 1992; Gutjahr et al. 1996; Rimstidt et al. 1998; Schwartz and Ploethner 2000). This is a possible explanation for the reduced availability of Cu. However, the mobility pattern of Cd must be attributed to other processes because carbonation mobilized Cd when leached at pH_0 .

With respect to the two principal components (Fig. 3), temperature had the lowest factor importance. However, temperature correlated with time, indicating that the higher the temperature, the more carbonation was favored.

All variables belonging to the $\text{pH}_{\text{stat}} 4.5$ leachings were omitted from the PLS modeling because data variations were dominated by noise and could not be related to the factors. Thus, carbonation did not considerably change the leaching characteristics of fly ash at low pH. This implies that metal mobility at pH 4.5 was still high (Ecke et al. 2002), thereby emphasizing the need for measures to counteract acidification in MSWI fly ash landfills. In this investigation, carbonation had the potential to lower the carbonate alkalinity from 7.4 to below $0.05 \text{ eq} \cdot (\text{kg} \cdot \text{FS})^{-1}$ (detection limit). Assuming that fly ash is pretreated by carbonation, and thereby leads to a depletion of carbonate alkalinity, abundant calcite will take over to buffer proton activity. According to the PLS model at high factor levels, the maximum calcite content was $\sim 250 \text{ g} \cdot (\text{kg} \cdot \text{FS})^{-1}$. When the supply of calcite is dissolved, fly ash then becomes vulnerable to proton attack and significant metal mobilization can be expected.

To estimate decalcification in an open system, a first approximation is given by Appelo and Postma (1999):

Table 2. Precipitation and Its Chemical Composition at Rickleå near Umeå, Sweden

Variable	Unit	Value
Amount	mm·yr ⁻¹	633
pH	—	4.66
NO ₃ -N	ppm	0.26
NH ₄ -N	ppm	0.19
SO ₄ -S	ppm	0.43
Ca	ppm	0.16
Cl	ppm	0.38
Mg	ppm	0.04
Na	ppm	0.24
K	ppm	0.15

Note: Data from Kindbom et al. (1998).

$$m_{\text{Ca}^{2+}} = \sqrt[3]{\frac{10^{-5.8} \times P_{\text{CO}_2}}{4}} \quad (1)$$

where the molality of Ca^{2+} ($m_{\text{Ca}^{2+}}$) in the leachate is a function of the CO_2 partial pressure (P_{CO_2}) [for assumptions see Appelo and Postma (1999)]. This approach was refined by applying *PHREEQC-2* to calculate decalcification at a fly ash landfill close to the MSW incinerator in Umeå. The model takes into account (1) the chemical composition of the rainwater in Umeå (Table 2), (2) the redox equilibrium of the rainwater with atmospheric O_2 , (3) that 33% of evapotranspiration leads to an increase in the concentration of rainwater components, and (4) an in situ temperature of 5°C (Maurice and Lagerkvist 1997). When the CO_2 partial pressure in the modeled landfill leachate was increased from $p\text{CO}_2 = 3.5$ (atmosphere) to $p\text{CO}_2 = 0.3$ (50% by volume) (Fig. 6), Ca^{2+} increased from 30 to 369 ppm while pH decreased from 8.28 to 6.22. With an annual precipitation of 633 mm, an in situ dry fly ash density of 1.5 t·m⁻³, and a calcite content of 250 g·(kg·FS)⁻¹, decalcification of fly ash yields 0.13 mm·yr⁻¹ when CO_2 is at atmospheric conditions and 1.56 mm·yr⁻¹ when CO_2 is 50% by volume. The first value is the minimum expected decalcification rate. The second value gives an estimate once fly ash is codisposed with putrescible refuse, hence generating an excess of landfill gas through anaerobic respiration. This kind of codisposal should be considered carefully or refrained from. If the formation of CO_2 in situ exceeds the amount needed to neutralize

Fig. 6. Ca^{2+} concentration and pH in landfill leachate from carbonated fly ash as function of $p\text{CO}_2$. The *PHREEQC-2* calculations take account to (1) chemical composition of precipitation (Table 2), (2) redox equilibrium of rainwater with atmospheric O_2 , (3) 33% evapotranspiration with respective increase in component concentration, and (4) in situ temperature of 5°C.

the carbonate alkalinity of fly ash, a CO_2 level of 50% by volume causes a tenfold increase of the decalcification rate of fly ash, and thereby promotes metal mobilization.

Other possible factors that influence decalcification need to be thoroughly assessed, including the location and design of fly ash landfills. For example, some soils such as cat-clays (Brady and Weil 1999) could generate groundwaters with an acidic pH, creating a risk of penetrating and subsequently leaching the fly ash.

Considering the limitations concerning landfill practices, carbonation is a promising stabilization method for most metal pollutants in MSWI fly ash. It might be conveniently performed as a pretreatment method, i.e., before the deposition of the fly ash. As a source of CO_2 , landfill gas should be tested. Landfill gas is abstracted at many landfill sites, is a reasonable resource, and is characterized by a CO_2 content of ~50% by volume, while the rest is mainly energy-rich CH_4 . Before CH_4 is used, it could be purified through absorption in fixed bed filters of wetted fly ash. About 40 g of CO_2 are retained per kg fixed solids (Fig. 4) which might not reverse but might mitigate the greenhouse effect.

On the basis of this bench-scale investigation, the authors recommend verification and improvement (or falsification) at pilot scale. With the exception of Cd, the prospects are good that the strictest EC standards for landfill leachate can be met. Regarding Cd, secondary sequestration processes such as silicate formation need to be assessed. If unsuccessful, preventive rather than remedial measures might be necessary, e.g., encouraging the separate collection and handling of NiCd batteries.

Conclusions

Carbonation of fly ash from municipal solid waste incineration was investigated at a laboratory scale. It had a remedial effect and is recommended for assessment at the pilot scale as a robust and reliable pretreatment technique to meet landfill standards.

The availability of critical elements such as Pb and Zn was reduced by two orders of magnitude. The partial pressure of CO_2 had the largest influence toward reduced mobility followed by time, water addition, and temperature.

Chromium was demobilized mainly due to the addition of water while being remobilized with time probably because of oxidizing conditions caused by atmospheric oxygen during treatment. The latter calls for reducing environments during both treatment and landfilling.

Future investigations should pay extra attention to Cd because of its increased mobility during the treatment. To compensate for this effect, other sequestering processes such as silicate formation could be useful.

Extreme fly ash alkalinity was successfully remedied through carbonation.

During landfilling, long-term fly ash stability relies on the abundance of calcite. For a typical landfill in Sweden, decalcification of carbonated fly ash was estimated at 0.13 mm·yr⁻¹ if controlled by atmospheric CO_2 . Higher partial pressure of CO_2 , as is probably caused by codisposal with putrescible refuse, increases the decalcification rate.

When determining the organic content of MSWI fly ash, the loss on ignition is erroneous by up to one order of magnitude because of the impact of both mechanically and chemically bound waters.

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