Short-term natural weathering of MSWI bottom ash

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Abstract

The release of heavy metals from MSWI bottom ash has been the key concern in the management of this material. The leaching distribution values obtained from 100 freshly quenched bottom ash samples, according to the German DIN 38414-S4 procedure test, showed the release of lead, zinc and copper to be the main hazards associated with bottom ash utilisation as a secondary building material. Currently, natural weathering of MSWI bottom ash, for an estimated period of 1–3 months, is the most economic treatment available to ensure the eventual utilisation of this material. The leaching of natural weathered bottom ash in the short-term (up to 9 months) was studied. The most significant changes in the bottom ash were found to occur in the first 90 days. At pH values greater than 12, lead, zinc and copper were the main heavy metals to be released from the MSWI freshly quenched bottom ash samples studied. Natural weathering for a period of about 90 days reduced the leaching of heavy metals, stabilising the bottom ash pH to minimise the solubility of metal hydroxides, and enabled the residue to be used as secondary building material. The profile of the pH neutralisation curve is similar to that described by carbonates, which would suggest that the reaction is controlled by CO_2. The formation of insoluble oxides as well as carbonates control the immobilisation of certain heavy metals, e.g. lead and zinc. The leaching of aluminium increases during this short natural weathering stage due to elemental metal oxidation. Aluminium solubility is controlled by the precipitation of gibbsite or other aluminium-sulphate neoformations. The latter may contribute to the immobilisation of heavy metals. © 2000 Elsevier Science B.V. All rights reserved.

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1. Introduction

Municipal solid waste incineration produces ash, approximately 11 Mt per year in the EU and 8 Mt per year in the USA that is a reduction by about 90% of the refuse volume and by

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60–70% of its mass [1,2]. The composition of the ash generated and the final management of ash depends on site-specific factors, regulatory requirements and utilisation or disposal objectives. For example, in the USA, the predominant MSWI residue stream is combined ash, i.e. a mixture of bottom ash, grate siftings and air pollution control residue (APC), and currently this residue is landfilled, either mixed with other wastes or monofilled. In some European countries residues are separated into bottom ash and APC residues prior to management. About 50% or more of stockpiled municipal waste incinerator bottom ash is used as secondary building material or for other similar purposes (e.g. Germany, The Netherlands, France and Denmark), in road sub-bases and the construction of embankments, wind and noise barriers and other civil engineering applications [1,3,4].

However, MSWI residue disposal in landfills as well as its reuse as secondary building material, are strictly controlled, e.g. some countries do not landfill the ash unless it is properly treated. The potential leaching and release of some heavy metals from the residues, particularly lead, copper, cadmium and zinc, must be evaluated before any decision is taken about their eventual disposal or use. Many leaching tests have been designed by regulatory agencies to characterise trace element mobility and to simulate a field-leaching scenario so that the amounts of toxic trace elements available for leaching can be estimated. All test methods can be evaluated with different attentions and criteria, the actual release of contaminants do not measure the release rates that one would expect under utilisation and disposal conditions.

Due to the great economic potential and the restrictive emission requirements, several equipment manufacturers are examining the options available for treating MSW combustion residues. Solidification, stabilisation, vitrification, classification by granulometric size particles and ageing or weathering are some of the currently available methods [1,5,6]. Treatment, however, is always dependant on site specific conditions, including regulatory requirements and utilisation or disposal objectives.

The main problem with most ash treatment methods are the high investment and operating costs. In EU countries, mainly in Germany, the weathering of the bottom ash for an estimated period of 1–3 months before their final disposal or their eventual utilisation is the most commonly employed method due to its low investment and operating costs. In this short period, some of the characteristics of the bottom ash undergo their most significant changes. Oxidation, carbonation, neutralisation of pH, dissolution and precipitation are some of the reactions that can occur in the weathering of bottom ash from MSWI [1,7,8]. Chemical stability is achieved by reducing the solubility of many toxic elements, and consequently their release. Other processes, such as sorption and mineral neoformation, are also chemical and physical mechanisms that contribute to reducing release [9–12]. Three major stages in weathering have been identified [7,8], and pH has been recognised to be a very significant parameter in trace element leaching. However, these last two processes are more important in the long-term, and the weathering of MSWI bottom ash continues even after its utilisation as secondary building material.

The aim of this paper is to determine the short-term natural weathering effects on the leaching behaviour of MSWI bottom ash as a function of time and experimental conditions. The relationship between pH values obtained in the natural weathering of MSWI bottom ash and the leaching behaviour of the main heavy metals is investigated. The inorganic chemical reactions involved, and the possible mechanisms of weathering are studied. In

addition, heavy metals and other parameters from leaching data DIN 38414-S4 procedure test of freshly quenched bottom ash, collected during a 1-year period, are presented.

2. Methods and materials

All bottom ash used in this study came from a single municipal solid waste incinerator, employing energy recovery (waste-to-energy, WTE). The incinerator is located in the metropolitan area of Tarragona (Spain) and began to operate in 1991. With two parallel trains of 9.6 t/h (150,000 t per year), it produces 50,000 MWh per year of electric power and 7000 t per year of scrap iron. The feed stream is mainly household waste, with a small input from commercial vendors. The residue is moved across the combustion chamber by rotating roll. Following combustion, bottom ash (35,000 t per year) is water-quenched, then carried by a drag conveyor. The residue is trommeled to 25 cm for iron and ferrous metals recovery and is finally stored in a bunker before disposal. The flue gases are cooled through heat exchangers with a boiler and sent to a semi-dry scrubber; particles are recovered by a fabric filter producing 4000 t per year of APC residue. Evacuation, handling and management of bottom ash and APC residue are carried out separately.

The bottom ash used in this study also included grate siftings, which makes up about 1–3% of the weight of the bottom stream [1], and heat recovery ash, which also accounts for 1–3% of the weight of bottom ash collected at this facility. A total of 100 samples of 10 kg of freshly quenched bottom ash were taken from the drag conveyor just before being transferred to the storage pile between November 1996 and February 1998. After each sample had been homogenised, a subsample of 3 kg was separated. The bottom ash samples thus obtained were maintained in a hermetically closed polyethylene bottle to avoid carbonation. According to the standard test for the permeability of thermoplastic containers to packaged reagents [13], the polyethylene bottle was filled with MSWI bottom ash, sealed and exposed at room temperature (23°C) for 90 days. After this period the weight gain was lower than 0.1%. So, the air permeability of the polyethylene bottle used in this experimental series was negligible. Afterwards, at laboratory scale, representative subsamples on which leaching test was performed were obtained by the quartering to 1/16 split procedure.

The natural weathering study of bottom ash was performed with some of the subsamples, with leachates having a high concentration in some hazardous heavy metals (e.g. lead and zinc). The natural weathering was carried out with samples of 1.5 kg bottom ash, in a chamber at room temperature for 3 months. Afterwards, a leaching test was carried out and the results were compared with those from freshly quenched bottom ash. At the same time, the natural weathering of bottom ash was also studied as a function of time, using 75 kg of freshly quenched bottom ash, transferred into two polyethylene containers (500 mm × 340 mm × 160 mm) for a period of 8 months and turned over daily to homogenise the sample. Representative subsamples of aged bottom ash were taken periodically and a leaching test was carried out.

The German DIN 38414-S4 procedure (10:1 liquid/solid ratio using deionised water in a wide-necked plastic flack slowly turned for 24 h) [14] was the leaching test used in this study. The aim of this test is to determine and assess the behaviour of some potentially hazardous contaminants in those conditions found in groundwater under an application or disposal

of MSWI bottom ash. Bottom ash moisture was first determined by drying subsamples at 105°C for 24 h. The resulting solutions were filtered and the pH was then measured. Clear filtrates were divided in two samples. One sample was acidified with concentrated HNO_3 and was used for the analysis of heavy metals in the leachate (Pb, Zn, Cu, Mn, Sn, Cr, Ni, V, Al, Fe, Ba, As and Cd). These were determined by inductive coupled argon plasma atomic emission spectrometer (ICP-AES). The second sample was left untreated and subjected to ion chromatography in order to analyse chloride and sulphates.

3. Results and discussion

3.1. Leaching of freshly quenched bottom ash

Bottom ash is a heterogeneous mixture of slag, glass, synthetic ceramics, minerals, para-magnetic and diamagnetic metals and unburned organic matter, the distribution of which depends on granulometric size [15]. The chemical analysis of bottom ash from many facilities in the world indicates that the finest fraction (less than 2 mm) contributes a significant proportion of the heavy metals and soluble inorganic salts [1,15]. The chemical composition of bottom ash is similar to basaltic and other geological materials, and only minor and trace elements (e.g. Pb, Cu and Cd) are enriched in the bottom ash [1,2]. These amounts of trace metals and soluble salts vary daily, depending on the input of the household refuse; the sampling of freshly quenched stockpiled bottom ash is needed to assess the behaviour of these residues. Many leaching tests have been developed to minimise the deviation on sampling in order to evaluate the hazardous behaviour over a long period of time more accurately.

The pH values obtained in all the leachates from freshly quenched bottom ash ranged between 12.4 and 12.6. At these alkaline pH values, the solution is close to solubility equilibrium of portlandite, $Ca(OH)_2$. However, the calcium analysed in some samples were in the range 700 ± 100 mg/l, which is greater than the expected solubility in this pH range (250–1000 mg/l of calcium). This means that the L/S ratio, used in leaching test procedure, was oversaturated with respect to calcium hydroxide. By decreasing the L/S ratios, the leachate becomes increasingly oversaturated with respect to calcium hydroxide (Fig. 1). The presence of $Ca(OH)_2$ in the bottom ash is due to the calcination at combustion temperatures in furnaces of natural or synthetic calcite ($CaCO_3$), gypsum ($CaSO_4\cdot2H_2O$) or anhydrite ($CaSO_4$), generating CaO and CO_2 or SO_2 in the reaction. Later, most of the generated CaO is hydrolysed as a result of quenching, and forms the corresponding hydroxide. Calcite, gypsum and anhydrite in bottom ash have been identified by XRD by many authors [2,7], corroborating that these minerals were not completely calcined. However, at a pH greater than 12 the solubility of these minerals is negligible.

Fig. 2 shows the distribution values of lead, zinc and copper, respectively, obtained from 100 samples of freshly quenched bottom ash leaching tests. These results are significant in that they illustrate the heterogeneity of bottom ash residue. The rest of the heavy metal analysed in all leached samples has a concentration lower than the minimum concentration used in the determinations (0.02 mg/l for Cd and As, and 0.1 mg/l for the rest).

The chloride analysed in the leachate showed a broad distribution (Fig. 2). These chlorides, most of them concentrated in the finest fraction of the bottom ash, and soluble in
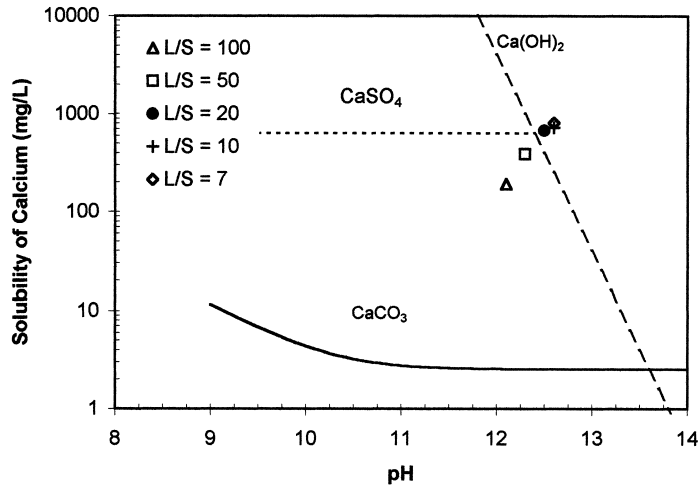
Fig. 1. Calcium solubility and freshly quenched bottom ash pH as a function of leaching test L/S ratio.
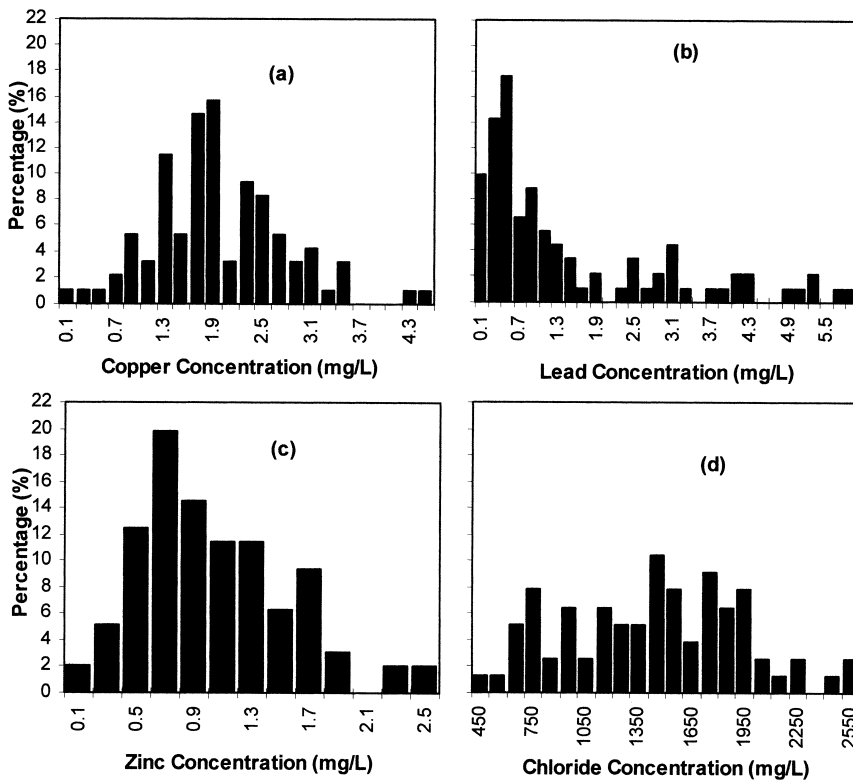

Fig. 2. Distribution of leached concentrations of copper, lead, zinc and chloride obtained from 100 freshly quenched bottom ash samples.

water at room temperature, originate from the neutralisation reaction between hydrochloric acid and alkaline and alkaline-earth oxides. The hydrochloric acid is generated in municipal waste combustion, mainly from the PVC and other halogen compounds, so its concentration depends on the amount of these materials in municipal raw waste. A similar neutralisation reaction took place among the sulphur oxides and alkaline and alkaline-earth-alkaline oxides to form the respective sulphates. The sulphur oxides are generated in the combustion of the natural and synthetic sulphates and the oxidising reaction of the sulphur present in most of the organic substances. Although the range of sulphate concentrations obtained in all the leachates from freshly quenched bottom ash (100–550 mg/l) is lower than that of chloride, the leached concentration of this anion also depends on the amount of these materials in municipal raw waste.

Each EU member state has its own environmental legislation and regulatory agencies operating different requirements for MSWI bottom ash disposal in landfills or for its utilisation, based on different leaching procedures. Thus, according to the local environmental requirements for this MSWI facility [16], 76% of all samples of freshly quenched bottom ash studied in this paper cannot be reused as secondary building material, and must be treated before their final management.

3.2. Natural weathering of freshly quenched bottom ash

Natural weathering has been shown to be the most economic method of treatment, in that it leads to chemical stability, mainly by reducing the release of heavy metals present in the residue. Most of these heavy metals are oxides formed in the combustion process within the furnaces, or metal materials that can be oxidised by the oxidising agents (i.e. atmospheric oxygen). The release of these heavy metals is mainly caused by the redissolution of their respective hydroxides, as the bottom ash pH is controlled by the solubility of $Ca(OH)_2$. Thus, the main weathering reactions must lead to a decrease in pH, so that, the solubility of hydroxides may also decrease, as is depicted in Fig. 3 for some of the main heavy metals. Meima and Comans [7,8,12] suggest that three major stages occur during the weathering of bottom ash, and the pH is controlled at each stage by the solubility of calcium hydroxide (pH 12.2), the formation of ettringite, gibbsite and gypsum (pH 10–10.5) and precipitation of calcite (pH 8.5), respectively.

Natural weathering in the short-term is enough to decrease bottom ash pH and to form the heavy metal insoluble oxides, that contribute to a reduction in leaching [1]. Table 1 records the effects of weathering in a 3-month period on three freshly quenched bottom ash samples. The initial leachates of these samples showed high concentrations of certain hazardous heavy metals, and it can be observed that, after weathering, the leachate concentration was significantly lower. The chloride concentration, remained virtually unaltered after natural weathering. Any differences in the values could largely be attributed to the heterogeneity of the samples. However, aluminium and sulphate concentrations after natural weathering were higher than initial concentrations in all samples.

The ageing of two samples of freshly quenched bottom ash during the same time period, with high initial lead, copper and zinc leached concentration, was performed in hermetically closed containers. Table 2 shows that the pH value, moisture percentage and heavy metal concentrations, did not differ substantially from initial values. Note that in these experi-
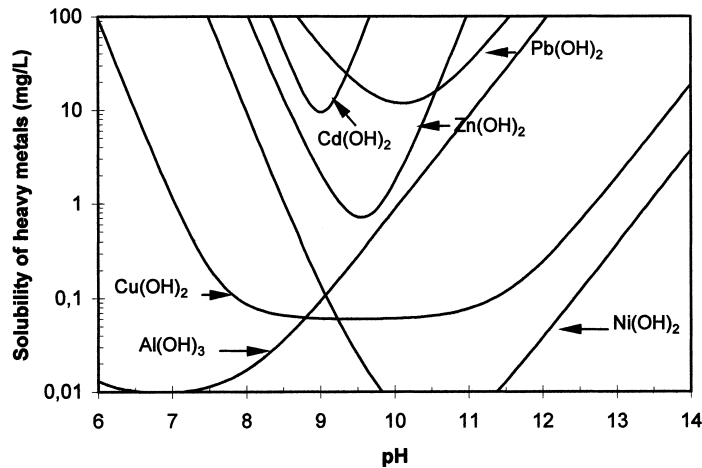
Fig. 3. Solubility of the most common heavy metal hydroxides as a function of pH.

ments the sulphate concentration fell and the aluminium concentration registered only a slight increase. The differences obtained between both weathering experiments can only be attributed to the presence of atmospheric reagents such as oxygen and carbon dioxide.

Fig. 4 illustrates the variation in bottom ash pH and moisture as a function of time during natural weathering. From this figure, three stages may be considered. During the first stage, pH value is greater than 12; as discussed previously and as is described by Meima and Comans [7,8,12]. At this stage the pH is controlled by the solubility of portlandite. The onset of the carbonation of portlandite and the oxidation of metals can also be attributed to this range of pH. These metals are mainly aluminium and iron, both of which are particularly abundant in the bottom ash, and their cations participate in the neoformation of compounds

Table 1
Analysis of natural weathering during 3 months under cover in a chamber at room temperature of 1.5 kg freshly quenched bottom ash samples[a]

Parameter	A_i	A_w	B_i	B_w	C_i	C_w
Moisture (%)	22.6	0.1	25.4	0.1	30.6	0.1
pH	12.6	9.6	12.5	9.8	12.5	9.8
As (mg/l)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Cd (mg/l)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Cu (mg/l)	2.51	0.95	4.45	0.80	1.2	0.71
Cr (mg/l)	<0.1	<0.1	0.1	<0.1	<0.1	<0.1
Pb (mg/l)	13.7	<0.1	23.4	0.3	4.99	<0.1
Zn (mg/l)	1.47	<0.1	2.14	<0.1	0.76	<0.1
Al (mg/l)	<0.5	19.6	<0.5	24.7	<0.5	22.5
Cl^- (mg/l)	2027	1992	1141	1074	1597	1488
SO_4^{2-} (mg/l)	366	1424	335	1218	242	1442

[a] Values obtained from the DIN 38414-S4 procedure leaching test. X_i: sample of freshly quenched bottom ash analysed before natural weathering; X_w: sample analysed after 3 months of natural weathering.

Table 2

Analysis of weathering during 3 months of 1.5 kg freshly quenched bottom ash samples introduced into hermetically closed containers[a]

Parameter	D_i	D_w	E_i	E_w
Moisture (%)	33.4	30.1	29.0	22.1
pH	12.6	12.3	12.5	12.4
As (mg/l)	<0.1	<0.1	<0.1	<0.1
Cd (mg/l)	<0.1	<0.1	<0.1	<0.1
Cu (mg/l)	3.19	2.55	3.48	2.68
Cr (mg/l)	<0.1	<0.1	<0.1	<0.1
Pb (mg/l)	2.65	1.91	4.27	2.31
Zn (mg/l)	1.22	1.05	1.11	1.30
Al (mg/l)	<0.5	1.28	<0.5	0.58
Cl^- (mg/l)	1922	1818	1870	1800
SO_4^{2-} (mg/l)	211	86	280	58

[a] Values obtained from the DIN 38414-S4 procedure leaching test. X_i: sample of freshly quenched bottom ash analysed before weathering; X_w: sample analysed after 3 months of weathering.

such as gypsum, gibbsite, ettringite and secondary minerals such as Fe/Al-(hydr)oxides and aluminosilicates [12]. The sorption of carbon dioxide is favoured by the alkalinity according to the acid–base reaction. Consequently, in the stage in which pH is lower than 12, the pH is controlled mainly by the solubility of carbonate compounds as calcite [9,17]. Fig. 4 shows that the curve obtained for the pH variation is very similar to that obtained in the neutralisation of a carbonate compound ($pK_{a2} = 10.3$ and $pK_{a1} = 6.3$), where carbonate coexists with bicarbonate below the second pK_a. However, for pH values close to the second acid constant the pH may also be controlled by the solubility of other species, i.e. ettringite [7–9]. At the lower pH values, carbon dioxide adsorption continues and the pH is again controlled by the solubility of calcite. Thus, carbonation seems to be the main reaction
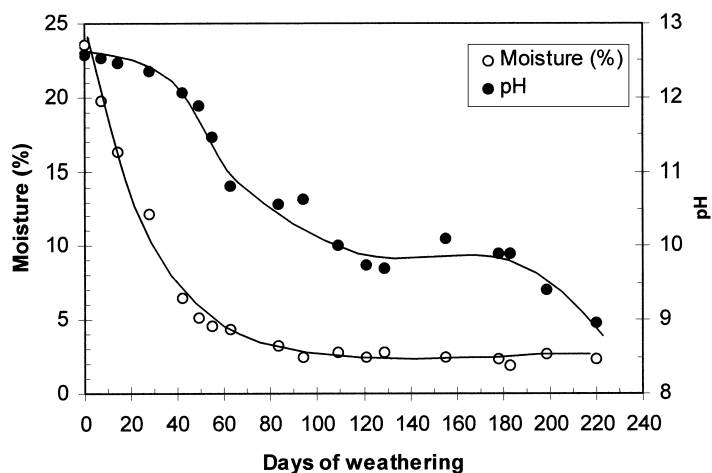

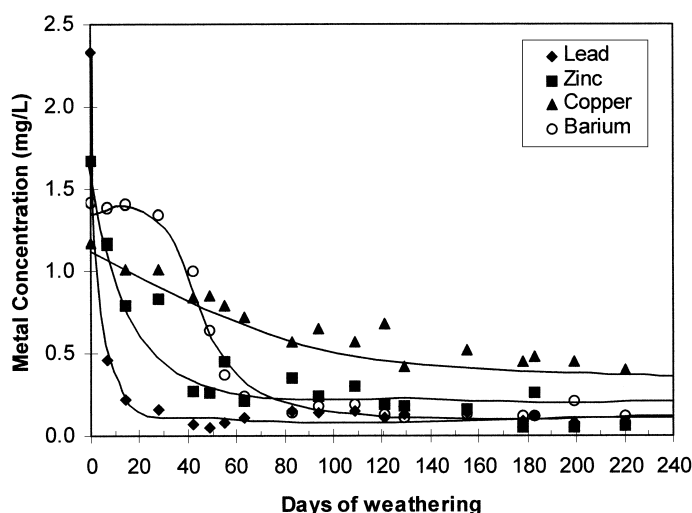Fig. 4. Moisture and leached pH from natural weathered bottom ash samples with time.

Fig. 5. Leached concentrations of lead, zinc and copper from natural weathered bottom ash samples with time.

controlling pH. As a direct result of this, and considering the partial pressure of carbon dioxide to be constant, the diffusion of this gas to the bottom ash surface is the parameter that controls the neutralisation rate during natural weathering. Gas diffusion takes place through the particle boundary layer, where the carbon dioxide previously dissolves. Thus, the degree of moisture present in the bottom ash must be a significant parameter. As Fig. 4 shows, it decreases and then levels off at about 3% of total bottom ash weight. If the degree of moisture could be kept constant during the weathering process, a possible increase in the neutralisation rate would be observed.

Fig. 5 shows the variation in the leached concentration of heavy metals with an initial concentration usually greater than the limits established for the reuse of bottom ash as secondary building material. It can be observed that the leached concentrations of lead and zinc fall dramatically after a very short period of natural weathering; in contrast, the copper concentrations fall only slowly during the period studied. According to the literature [7,18–20], copper has a strong affinity with organic material to form organometallic complexes. So, the release of copper may be due to the presence in the leachate of dissolved organic carbon (DOC). Thus, the slight decrease shown in Fig. 5 may be due to the partial oxidation of organic material by atmospheric oxygen and the further formation of insoluble copper oxide at this pH. Lead concentration, on the other hand, decreases more quickly than the pH values for the same period (Fig. 4). The dissolved lead concentration obtained in all the samples studied shows an undersaturation with respect to lead hydroxide, anglesite ($PbSO_4$) and cerrusite ($PbCO_3$). Similar findings have been described by other authors [18]. The lower lead solubility may be a result of sorption processes and/or formation of more stable mineral phases [7,10,18]. The high concentration of chloride and sulphate ions as well as the carbonate ions formed during the weathering process may also contribute to a reduction in the release of lead by the formation of its insoluble compounds. This can also
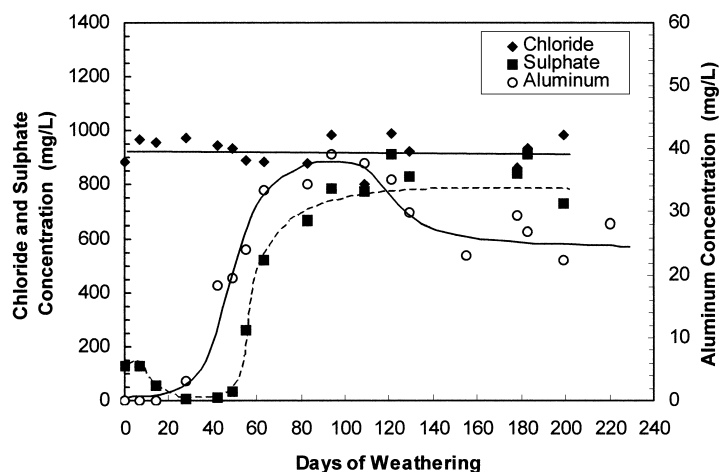
Fig. 6. Leached concentrations of aluminium, sulphate and chloride from natural weathered bottom ash samples with time.

be observed in the dramatic fall in the leached concentration of barium (Fig. 5), the activity of which is strongly affected by the sulphate concentration.

Fig. 6 plots the leached concentrations of aluminium, chloride and sulphate against the time of natural weathering. The chloride concentration was constant throughout the natural weathering of bottom ash, the sulphate concentration (see also Table 1) increased during the first 3 months and then remained constant, while the aluminium concentration peaked early on and then levelled off. The concentration values obtained are higher than those for the solubility of amorphous aluminium hydroxide but similar to those of gibbsite, which seems to control the solubility of aluminium. It is also possible that the neoformation of aluminosilicates or ettringite [7–10,12] may help in this process. Aluminium accounted for more than 90% of the total amount of diamagnetic metals, and seems to be randomly distributed in all size fractions [15]. At strongly alkaline pH, the aluminium may be oxidised by reacting with water (moisture) and oxygen. On the other hand, the gypsum available in the bottom ash seems to establish different equilibria with aluminium hydroxide to form calcium aluminates, ettringite or other non-stoichiometric compounds that include aluminium and sulphates. This could explain the high activity of both ions in the leachate obtained from weathered bottom ash samples.

In order to corroborate this, an additional experiment was performed. Metallic aluminium, calcium sulphate and water were mixed and the relations between the compounds were found to be the same as those obtained in the freshly quenched bottom ash samples. The initial pH was obtained by adding the corresponding amount of sodium hydroxide. The system thus obtained was aged in the same way the bottom ash samples described above. The initial aluminium oxidation is a highly exothermic reaction that increases the temperature of the system. This was also observed in the initial natural weathering of freshly quenched bottom ash, where a temperature of 45°C was recorded in the residue in the first stage of the experiment performed (the room temperature was in all cases about 22°C). This suggests
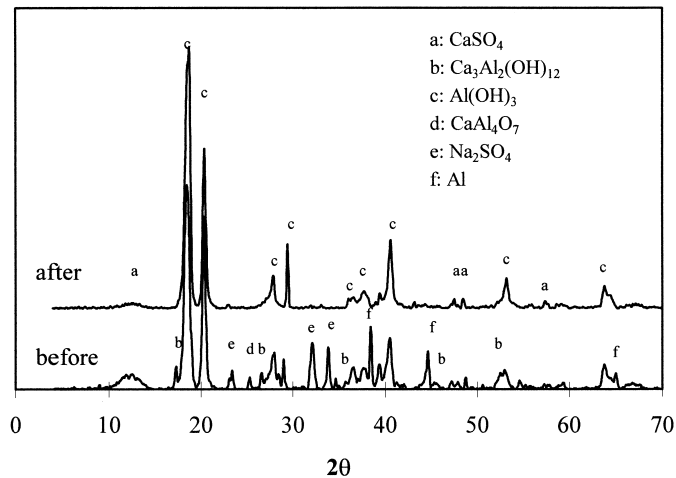
Fig. 7. X-ray diffractogram of the system exposed to 3 months of weathering, formed by elemental aluminium, calcium sulphate and sodium hydroxide before and after carrying out the leaching procedure test.

that the oxidation of metals (e.g. aluminium, iron and copper) or organic materials, jointly with other exothermic reactions such as neutralisation, are the first reactions to occur. Fig. 7 shows, after the system has been exposed to 3 months of weathering of the described system, the X-ray diffractograms obtained before and after carrying out the leaching procedure test DIN 38414-S4. In the first X-ray diffractogram, aluminium and calcium sulphate non reacted may be identified, jointly with other new phases formed in the system, as amorphous aluminium hydroxide, calcium aluminate and sodium sulphate. However, after the leaching test was performed, only aluminium hydroxide and calcium sulphate 0were identified as majority phases. The pH of the leachate was 10.9 and the aluminium and sulphate leached concentrations were 160 and 3500 mg/l, respectively. Although the values reached are higher than those analysed after the leaching of weathered bottom ash, this may be explained by the presence, in the bottom ash, of other compounds such as calcium oxide and silica as well as other metals that might modify these concentrations by reacting to form ettringite or aluminium-silicates.

4. Conclusions

At pH values greater than 12, lead, zinc and copper were the main heavy metals to be released from the MSWI freshly quenched bottom ash samples studied. Natural weathering for a period of about 90 days reduced the leaching of heavy metals, stabilising the bottom ash pH to minimise the solubility of metal hydroxides, and enabled the residue to be used as secondary building material.

In the short-term, the carbonation of calcium hydr(oxide) is the main reaction controlling the pH. In the absence of CO_2 the variation in pH is not significant. This means that reactions involving OH^-, such as the neoformation of aluminium hydroxide, gibbsite, ettringite or aluminosilicates, do not have any influence on the bottom ash pH.

The natural weathering of MSWI bottom ash for an estimated 2–3 months before its eventual disposal or utilisation is enough to diminish the release of heavy metals contained in residue. The concentrations of lead and zinc that are leached fall dramatically in a very short period of time. These concentrations seem to be independent of the pH of the bottom ash and are undersaturated if we compare them to lead hydroxide, cerrusite, hydrocerrusite or anglesite and zincite, respectively. It seems that the release of lead might be involved in sorption processes, such as the neoformation of secondary minerals as aluminates or aluminosilicates. This mechanism of neoformation in the natural weathering of bottom ash in the short-term is strongly influenced by the presence of air.

In the short period of exposure to natural weathering, the kinetic mechanism of aluminium oxidation and subsequent neoformation of secondary materials seem to be more relevant in the presence of oxygen (air). These results suggest that the ventilation of bottom ash before its utilisation or disposal in landfills is a major factor in leaching for a reduction in the release of heavy metals in the short-term.

Acknowledgements

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