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The leaching behavior of incinerator bottom ash as affected by accelerated ageing

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Abstract

Different artificial ageing treatments were applied to fresh incinerator bottom ash with the aim of promoting/accelerating the natural reactions occurring over time on the mineral phases in the material. The weathering treatments included accelerated carbonation, treatment with air and treatment with nitrogen gas. Both fresh and treated bottom ashes were characterized for their mineralogical composition, acid neutralization capacity and leaching behavior of metals. The results were compared to those obtained from similar characterization of bottom ash samples weathered under atmospheric conditions for 4 years.

The findings from the experimental campaign indicated that both the release of metals and the type of solubility-controlling solid phases varied as a function of the duration and type of ageing treatment. The most appreciable influence on mineralogy, acid neutralization behavior and metal leaching was observed for the 48 h carbonation treatment. It was also observed that 4-day aeration of bottom ash was capable of producing similar buffering behavior to that displayed by naturally aged bottom ash.
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Keywords: Bottom ash; Ageing; Weathering; Metal leaching; Acid neutralization behavior

1. Introduction

Utilization and disposal of incineration residues may produce adverse environmental impacts due to the potential release of contaminants, namely heavy metals and salts [1–3]. The extent of release from incineration residues can vary significantly over time, since the leaching behavior of such materials is largely dependent on the mineralogical alterations which naturally occur as a consequence of contact with atmospheric agents. In a medium to long term scenario, the residual pollution potential may not be negligible, and this may lead to uncertainties concerning either the optimal conditions for utilization or the proper management strategies for the passive care period of landfilling.

As a consequence, an appropriate strategy for management of incinerator residues could be to bring forward the natural reactions occurring within the material, with the purpose to attain a more chemically stable solid matrix which is capable of keeping contaminant release rates over time at environmentally acceptable levels.

Several studies [2,4–9] demonstrated that, when exposed to atmospheric conditions, incinerator bottom ash is subjected to a number of weathering reactions which cause alterations in the original mineralogy. The variability in mineralogical composition can be explained by the fact that most of the high-temperature solids contained in incinerator bottom ash, which are formed as a consequence of rapid quenching of the material at the exit of the combustion chamber, are metastable under atmospheric conditions. The modification of the original minerals and formation of new phases largely dictate the leaching behavior of the material. The modified leaching behavior is the result of several interrelated processes, including hydrolysis, hydration, dissolution/precipitation, carbonation, oxidation/reduction, complexation, sorption, formation of solid solutions as well as mineral neo-formation. The chemical and mineralogical changes caused by such processes lead to changes in macroscopic

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properties of the material, including acid neutralization capacity and pH, redox potential, as well as sorption and ion exchange capacity [6,10], with all such parameters greatly affecting metal solubility as well as speciation.

This paper presents the results from an experimental study to evaluate the effects of different artificial ageing treatments applied on incinerator bottom ash with the purpose of promoting/accelerating natural weathering processes.

2. Materials and methods

Fresh bottom ash was sampled daily over a 1-week-period at an Italian solid waste incinerator from a storage tank where the material exiting the bottom ash quenching unit was collected. The sampling procedure was carried out through quartering. The total amount of bottom ash collected during the sampling period was about 100 kg. An additional quartering step was applied to the material in order to derive homogeneous and representative laboratory samples to be used for chemical characterization and ageing experiments. Thereby, bottom ash was characterized for a number of physical properties (namely water content, bulk density and loss on ignition, LOI) and for chemical composition (elemental and anion composition, acid neutralization capacity, ANC, as well as metal leaching). Measurements followed ASTM D2216 for water content, ASTM C29 for bulk density and ASTM C25 for LOI. The acid neutralization capacity was determined through the ANC test as proposed by the Canadian Wastewater Technology Centre [11]. All the measurements were conducted in triplicate with the exception of the ANC test, which was run on a single replicate. The microstructural composition of bottom ash was also investigated by means of powder X-ray diffraction analysis (XRD) using Cu Kα radiation (copper tube operated at 30 kV and 40 mA) at 0.05 2ϑ (count time = 3 s).

The ageing treatments were carried out in a lab-scale airtight column reactor containing fresh bottom ash (as obtained from the homogenization procedure) where a continuous water-saturated gas flow was blown using an external pumping apparatus. The reactor was a 10 cm Φ, 30 cm h cylindrical perspex column in which approximately 100 g of bottom ash were placed in three individual layers separated by plastic grids. Bottom ash was subjected to different ageing treatments with the purpose of simulating different utilization or disposal conditions, including carbonation, oxidation by atmospheric oxygen and establishment of anoxic conditions. The effect of carbonation resulting from the uptake of atmospheric CO_2 by the initially alkaline bottom ash was simulated using a pure CO_2 flow. Air was used to test the influence of oxidizing conditions resulting from the contact between the material and atmospheric oxygen at the initial stages of landfilling. The influence of the establishment of anoxic conditions as a result of biologically-mediated degradation of residual organic carbon was investigated by blowing an N_2 flux into the reactor. Residence time values of 6, 24 and

48 h were selected for the different treatments; in the case of the air treatment, the contact time was prolonged to 4 days.

The artificially aged bottom ash samples were characterized for acid neutralization behavior and metal leaching as a function of pH through the ANC test, as well as for mineralogical composition by means of XRD analysis.

A sample of 4-year-old bottom ash taken from the same incineration plant and weathered under natural conditions in covered vessels was used for reference purposes. ANC, metal leaching and the presence of crystalline species were analyzed on the naturally weathered material as well, and the findings were compared with the results obtained for both fresh and artificially aged bottom ash.

The metal concentrations as a function of pH as obtained from the ANC test were inputted into the MINTEQA2 geochemical speciation code [12] to allow for estimation of the composition of the leachates in equilibrium with potential solubility-controlling minerals. The standard MINTEQA2 database was integrated by adding the solubility constant for ettringite (log K_{sp} = −56.7 [13]) and modifying that for gypsum (log K_{sp} = +4.62 [5]). Application of the speciation code followed a three-step procedure: (1) MINTEQA2 was run using the measured concentrations and pHs as input data and suppressing precipitation for all solid phases; (2) potential solubility-controlling minerals where chosen among those displaying saturation indices (SI) in the range $-1 \leq SI \leq +1$ on the basis of likelihood of formation in bottom ash; and (3) the program was run enabling precipitation of the selected minerals as infinite solids. Application of MINTEQA2 in the case of fresh, artificially aged and naturally weathered bottom ash allowed to infer about possible modifications in the mechanisms of metal leaching from bottom ash as a result of ageing.

3. Results and discussion

From the preliminary characterization, the following values were measured for the main physical properties of fresh bottom ash: water content = 13.65%, bulk density = 1511 kg/m^3, LOI = 3.93%. The chemical composition of the material is reported in Table 1, which indicates that the major trace elements were Cu (1080 mg/kg), Ni (330 mg/kg), Zn (200 mg/kg) and Cr (170 mg/kg). Cu, Ni and Cr, according to their lithophilic behavior, are among the trace metals present in waste combustion bottom ash. Zn, which is a volatile element, can still be found in bottom ash in appreciable concentrations [1,3,14].

The mineralogical composition of fresh and aged bottom ash is reported in Fig. 1. For the freshly quenched material, the main crystalline phases were anhydrite ($CaSO_4$) and corundum (Al_2O_3), with small amounts of ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26H_2O$) being evidenced by a low-intensity peak at 9.05° 2ϑ. The presence of ettringite in quenched incinerator bottom ash has been shown to result from the dissolution of Ca- and Al-containing minerals and

Table 1
Chemical composition of fresh bottom ash

Element	Concentration (mg/kg dry wt.)
Al	8393
Ca	22985
Cd	<8
Cr	168
Cu	1084
Fe	4659
K	3264
Mg	3177
Mn	72
Ni	332
Pb	<80
Si	278230
Zn	201
Chloride	<0.01[a]
Sulphate	0.09[a]

[a] Expressed as percentage dry weight.

subsequent reaction between such phases according to the equation [2,5]:

$$6Ca^{2+} + 2Al^{3+} + 3SO_4^{2-} + 38H_2O$$
$$\rightleftarrows 12H^+ + Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26H_2O$$
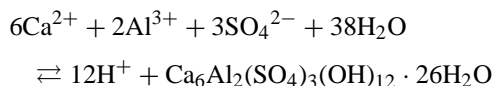
Although portlandite ($Ca(OH)_2$) is likely to form in bottom ash after quenching as a result of hydrolysis of calcium oxide (CaO) [15,16], no significant amounts of crystalline $Ca(OH)_2$ could be identified through XRD analysis. It will be shown later on in the paper that no evidence of the presence of $Ca(OH)_2$, even in the amorphous form, could be gained from characterization of the material.

The ettringite detected in fresh bottom ash was found to disappear as a result of both natural and artificial ageing (see Fig. 1), which is an indication of ettringite being converted into more stable Ca- and SO_4-containing minerals [17]. Unlike ettringite, anhydrite and corundum were still detectable in the aged materials, although with decreased peak intensities if compared to fresh bottom ash. In the case of corundum, the reduction in peak intensity with ageing may support the

hypothesis that Al is converted to amorphous (hydr)oxide (including AlOOH and $Al(OH)_3$) or aluminosilicate forms as a consequence of mineralogical alterations induced by ageing [2,6,10].

When considering both the naturally weathered and the artificially carbonated bottom ash, an additional peak was recognized at $29.4° 2\vartheta$, which was interpreted as the main peak for calcite ($CaCO_3$). Reasonably, calcite formed as a result of CO_2 uptake by the initially alkaline bottom ash. In addition, precipitation of calcite can be related to the observed depletion in ettringite and anhydrite from the fresh material, in that, according to the mass-action law, precipitation of calcite requires the dissolution of other less stable Ca-containing minerals.

An interesting feature which was derived from the XRD patterns was related to the degree of crystallinity. An indication for that was obtained by calculating the so-called crystallinity index, as defined by the ratio between the peaks area (related to the crystalline species overall) and the total area under the XRD curve (related to both the crystalline and amorphous content). This was found to increase for the naturally aged bottom ash, leading to the conclusion that natural weathering resulted in the formation of a more crystalline structure if compared to the fresh and artificially aged material. However, the resolution of the XRD analysis did not allow for identification of any neoformation mineral in 4-year-old bottom ash.

As far as the acid neutralization behavior of the material was concerned, a first comparison was made between the "own" pHs of the material, i.e. the pH values established in the ANC eluate with no acid added. In the case of fresh bottom ash, the own pH was 10.68. This indicates that, as suggested by previous studies [5,16,18], the leachate was strongly undersaturated in portlandite, which would control the equilibrium leachate pH at as a high value as 12.5. This result coupled with XRD analysis suggests that no significant amounts of either crystalline or amorphous $Ca(OH)_2$ were present in bottom ash. If one refers to a pure system containing the minerals gibbsite ($Al(OH)_3$), gypsum ($CaSO_4 \cdot 2H_2O$) and ettringite,
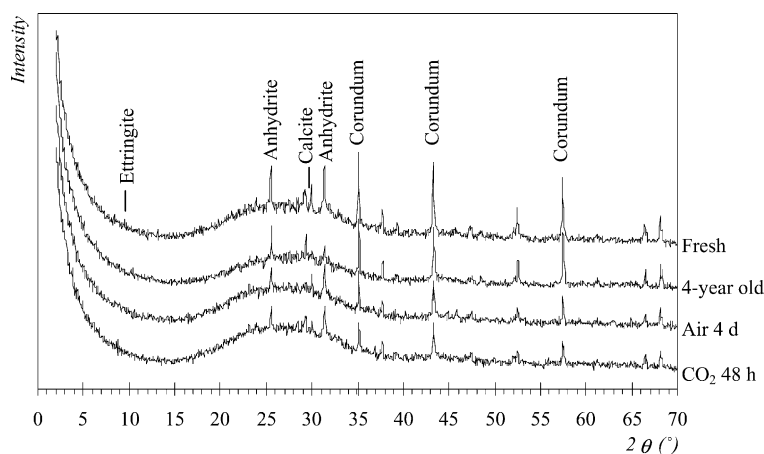

Fig. 1. XRD patterns of fresh, naturally aged and artificially weathered bottom ash.

prediction of equilibrium pH through geochemical modeling yields a value of 10.33, which is much closer to the measured value. This states that the pH of fresh bottom ash is largely dictated by the major constituents Ca, Al and SO_4^{2-}.

For the naturally weathered material, the own pH (8.58) can be reasonably well explained by the presence of calcite ($CaCO_3$) in equilibrium with atmospheric CO_2, which would theoretically control the solution pH at a value of 8.25. If the acid neutralization behavior of naturally weathered bottom ash is considered (see Fig. 2a), the presence of calcite is indicated by the pH plateau on the pH versus acid-added curve in a pH range between 8.16 and 8.58.

The effect of the accelerated ageing treatments on the acid neutralization capacity of the material is depicted in Fig. 2b–d. In general, it was found that a residence time of 6 h was not able to produce any significant effect on acid neutralization behavior, irrespective of the type of treatment applied.

It was found (see Fig. 2b) that the carbonation treatment altered the acid neutralization behavior of bottom ash significantly. After 48 h accelerated carbonation, a second pH plateau at approximately 5.5 units was identified on the ANC curve, which was responsible for a significant increase in the acid neutralization capacity of the material under acidic conditions, resulting in an ANC to pH 5 of 0.88 meq/g, as opposed to 0.46 and 0.48 meq/g for fresh and naturally weathered bottom ash, respectively.

The N_2-based ageing treatment (see Fig. 2c) produced a general reduction in pH in the alkaline range if compared to both the fresh and naturally aged material, the magnitude of such an effect increasing with increasing the treatment time. Reasonably, the establishment of anoxic conditions led to a release of protons which contributed to a decrease in pH.

The effect of 2-day and 4-day forced aeration (see Fig. 2 d) on acid neutralization behavior was very similar to that of natural weathering, although the latter was able to produce a larger pH plateau in the alkaline pH range. The conclusion that can be drawn from this feature is that the ANC of naturally weathered bottom ash can be easily reproduced by the application of a forced aeration treatment of limited duration.

The study of leaching of major elements and especially Ca is particularly important as it has been shown in this work and elsewhere [5,16,18] that Ca minerals are capable of controlling the leachate pH and exhibiting sorption properties for a number of trace metals. The release of Ca from fresh, artificially carbonated (48 h) and naturally weathered bottom ash is shown in Fig. 3a along with possible solubility-controlling phases as identified by MINTEQA2 calculations. Under alkaline conditions, Ca leaching appeared to be controlled by ettringite for the fresh material (at pH > 9) and by calcite for both naturally and artificially aged bottom ash, which is in good agreement with the findings from XRD and ANC analysis. As noted before, this suggests that the ettringite formed
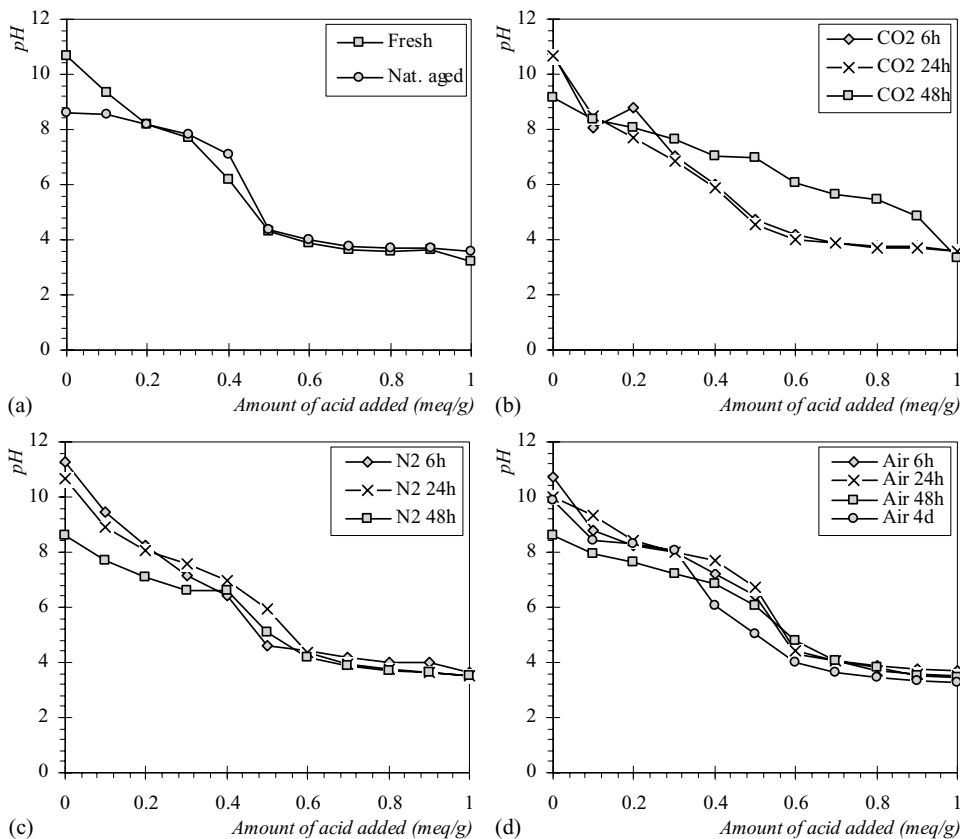

Fig. 2. ANC curves for (a) fresh and 4-year-old, (b) carbonated, (c) N_2-treated and (d) air-treated bottom ash.
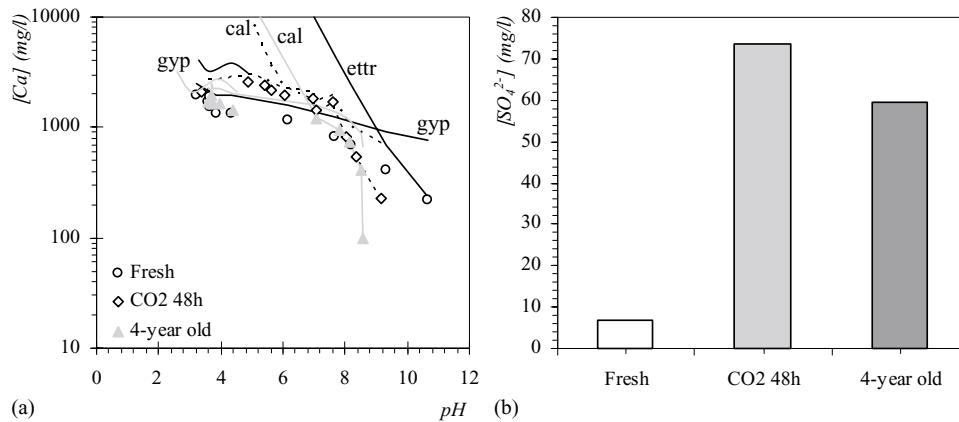
Fig. 3. Ca leaching and solubility-controlling phases (a) and sulfate release (b) from fresh (—),- carbonated (- - -) and naturally weathered (▬) bottom ash (cal = calcite; ettr = ettringite; gyp = gypsum).

after quenching is dissolved as a result of precipitation of calcite. This phenomenon can also explain the increase in sulfate concentration in the ANC leachates with no acid added for the carbonated and naturally weathered material if compared to fresh bottom ash, as depicted in Fig. 3b. In general, it appears that both natural and artificial ageing enhanced the release of sulfates from the material, so that, as far as salts concentration is concerned, the quality of the first leachate at the disposal site is anticipated to change if weathered bottom ash is landfilled.

Solubility control by calcite was also responsible for a lower Ca leaching at alkaline pHs if compared to that observed from the fresh material. In the acidic pH range Ca solubility appeared to be controlled by gypsum, irrespective of both the age and the type of material. However, due to common ion effects and increased ionic strength, Ca concentrations in the leachates from CO_2-treated bottom ash were significantly higher than for the other samples.

Fig. 4 depicts the leaching of Zn and Cu as a function of pH and the possible solubility-controlling phases suggested by geochemical modeling. The leaching of Zn and Cu was strongly pH-dependent for all the bottom ash samples investi-

gated, with the lowest leachate concentrations being detected at alkaline pH values. No Zn mineral was found to match the experimental leaching curves, so that all the leachates were strongly undersaturated with respect to both zincite (ZnO) and Zn hydroxide ($Zn(OH)_2$). Either surface precipitation or surface complexation mechanisms of Zn onto bottom ash phases including Fe and Al (hydr)oxides may possibly explain the observed leaching behavior.

In the case of Cu, fresh bottom ash leachates were strongly oversaturated at high pHs and strongly undersaturated at low pHs with respect to tenorite (CuO) and Cu hydroxide ($Cu(OH)_2$), which is a common feature for bottom ash leachates in case only inorganic complexation reactions are taken into account [6,19]. Oversaturation in the alkaline pH range is likely to be related to the complexation of Cu by dissolved organic matter [19,20], in that Cu is know to exhibit very strong affinity for organic ligands. For the carbonated material, MINTEQA2 modeling identified malachite ($Cu_2(OH)_2CO_3$) as a possible solubility-controlling solid; tenorite was also shown to be stable between pH 6 and 8. However, previous studies [7] indicated that there is no strong evidence for Cu carbonate minerals precipitation, and that
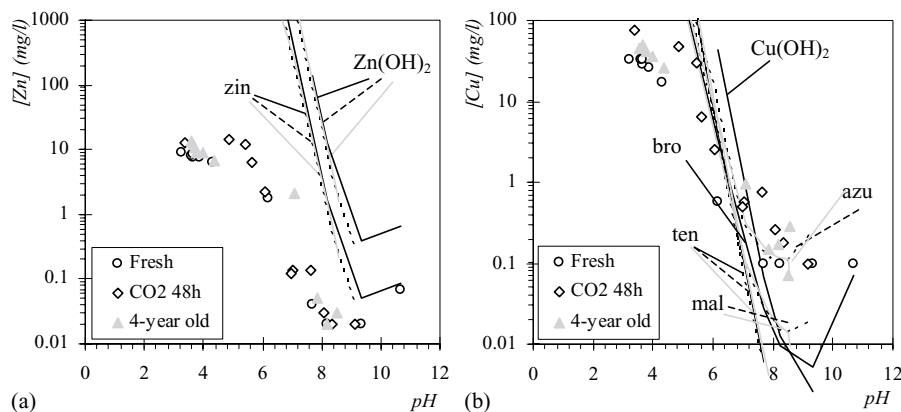

Fig. 4. Zn and Cu release and solubility-controlling phases for fresh (—), carbonated (- - -) and naturally weathered (▬) bottom ash (zin = zincite; azu = azurite; bro = brochantite; mal = malachite; ten = tenorite).
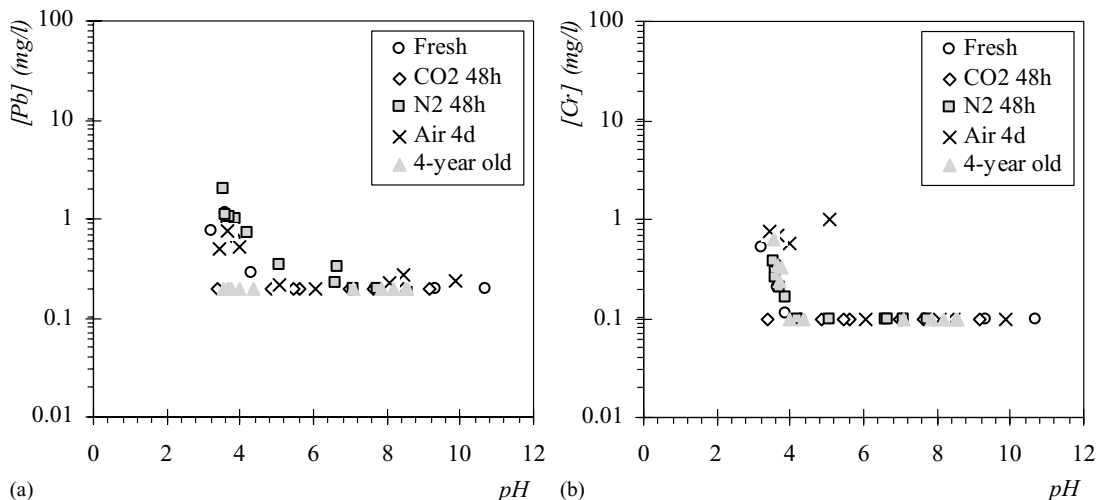
Fig. 5. Pb and Cr release from fresh and aged bottom ash (detection limits: 0.2 mg Pb/l and 0.1 mg Cr/l).

probably Cu leaching is rather related to co-precipitation and surface complexation phenomena with Fe and/or Al minerals, particularly for aged bottom ash for which the number of sorption sites has been shown to be far greater than for the fresh material [19].

Fig. 5 compares the release of Pb and Cr from the fresh, artificially and naturally aged material. In all cases, the leaching patterns were relatively pH-independent. This leads to exclude solubility control by a pure mineral phase, rather indicating the occurrence of sorption mechanisms [21]. For Pb, CO_2-treated as well as naturally weathered bottom ash displayed leachate concentrations below the analytical detection limit (0.2 mg/l) over the entire pH range investigated. In Fig. 5, such a detection limit was used to plot concentrations which were actually below that value. Higher concentrations (up to 2 mg/l) were measured under acidic conditions in the leachates from fresh as well as air- and N_2-treated bottom ash. It may be hypothesized that the low Pb leaching for carbonated and naturally aged bottom ash could be related to the strong affinity of Pb towards surface complexation onto Fe and Al (hydr)oxides (as shown by Meima and Comans [10]). However, it was not possible to unambiguously assess, on the one hand, whether accelerated carbonation and natural weathering resulted in increased amounts of such (hydr)oxides and, on the other hand, whether sorption phenomena may still play a role at low pH values (pH < 4).

The leaching behavior of Cr was significantly affected by the air-treatment, which produced increased Cr concentrations in solution at pH < 6 if compared with the other bottom ash samples. Reasonably, this resulted from the establishment of oxidizing conditions and consequently from the higher solubility of oxidized Cr species.

Another aspect that should not be disregarded when studying trace element leaching from aged bottom ash is the identification of the relevant pH domain at the disposal site. This is the result of acidification of the material produced by external stresses including acidic rain, uptake of atmospheric

CO_2, biochemical processes, as well as oxidation/reduction reactions. When judging about metal release from fresh or pre-treated bottom ash, comparison of the leaching behavior should be made on the same timescale, i.e. considering the same "H+ equivalents load". It can be assumed that the initial landfill leachate corresponds to cumulative L/S ratios of up to 0.2 l/kg; on the hypothesis of an acidic rain at pH 5, this will result in 2×10^{-6} meq. H+ contacting each gram of the material. Under such conditions, the leachate pH will roughly correspond to the own pH of the material. For this reason, on the basis of the results from the ANC test, it is particularly evident from Fig. 4 that the initial leachate from fresh bottom ash will contain higher Zn concentrations if compared to the pre-treated material. From this feature the conclusion can be drawn that it is advisable to promote leaching of the material during the initial stages of landfilling, i.e. when leachate emission control relies upon active care systems. When the mode of landfill operation resembles a "dry entombment", the contact between the landfilled material and the atmospheric agents is hindered. Such a management strategy causes a significant delay in the weathering process and keeps the pollution potential of the material unchanged, so that the high-strength leachate will be generated at the long term as soon as failure of the passive control systems occurs. For the artificially carbonated material, as the own pH corresponds to the solubility minimum of most trace metals, it appears that isolating the material from the contact with atmospheric agents is not required to reduce the leachate pollution potential.

It should also be mentioned that ageing of the material, particularly the carbonation treatment, is responsible for increasing the buffering capacity of the material, which in turn dictates the response of the material itself to external stresses. In the pH range of interest, a reduction in pH will promote mobilization of most trace metals. With a view to this, it appears that the alteration of chemical and mineralogical properties of bottom ash resulting from ageing

should be promoted through either treatment of the material prior to disposal or proper landfill management during disposal.

4. Conclusions

The present study indicated that weathering of bottom ash is capable of modifying the mineralogical and chemical properties of the materials in terms of types of mineral phases, pH, acid neutralization capacity as well as leaching behavior.

When dealing with landfilling of incinerator residues, attainment of the final storage quality of the material, i.e. the condition when contaminants concentrations in the leachate are reduced to an environmentally acceptable level, should be sought. In this respect, the results from the experimental campaign indicated that isolating bottom ash from the environment at the disposal site by preventing any contact with atmospheric agents (water, CO_2, oxygen) would not be advisable, as this would keep the pollution potential unchanged. Allowing weathering reactions to proceed would result in improved stability of the material with respect to the leaching behavior.

Furthermore, the results from the study showed that applying accelerated ageing as a pretreatment stage of limited duration prior to either utilization or final disposal may result in improved mineralogical, chemical and leaching characteristics of bottom ash.

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