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Application of accelerated carbonation on MSW combustion APC residues for metal immobilization and CO_2 sequestration

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ABSTRACT

The present study focuses on the application of an aqueous phase accelerated carbonation treatment on air pollution control (APC) residues from municipal solid waste combustion, aimed at assessing its influence on the environmental behaviour of the residue under concern, as well as the potential of the process in terms of sequestration of the CO_2. APC residues are considered hazardous waste and must be treated before final disposal in order to achieve the immobilization/mobilization of critical contaminants such as heavy metals as well as mobilization of soluble salts. The treatment applied proved to be effective in reducing the mobility of Pb, Zn, Cr, Cu and Mo, the optimum final pH for the carbonated APC residues being in a range of 10–10.5, whilst a mobilization effect was noticed for Sb and no effect was assessed for chlorides. The effect of carbonation treatment on the contaminant release was further evaluated by means of a sequential extraction procedure, indicating that the distribution of contaminants on water soluble, exchangeable and carbonate fraction was modified after treatment. The CO_2 sequestration potential assessed for the APC residues showed that the carbonation technology could be a technically viable option in order to reduce emissions from WtE plants.

1. Introduction

The Waste to Energy treatment, whilst being a feasible option for recovering energy and reducing the mass of municipal solid waste fractions to be landfilled, is known to entail concentration of inorganic contaminants in the post-combustion by-products such as bottom ash, fly ash and flue gas cleaning residues [1–3]. In particular the APC residues, which are the solid by-products generated by flue gas treatment, are classified as hazardous wastes according to the Commission Decision 2000/532/EC. The main concern related to the disposal or reuse of these residues is represented by the potential release of metals and soluble salts [4], so that a pretreatment is required in order to reduce the associated environmental impact. In the literature, several treatment technologies have been proposed as appropriate treatments for APC residues, including solidification and/or stabilization, physical and chemical separation and thermal processes (see e.g. and references therein). Among these options, accelerated carbonation has been described as an important treatment technology capable of improving the environmental and technical behaviour of different types of combustion residues, consisting in the adsorption of CO_2 by alkaline materials which causes pH decrease and calcite precipitation [6–11]. This process that occurs also spontaneously under ambient conditions, results in a number of chemical/mineralogical transformations which involve also the immobilization of several inorganic contaminants of concern. Furthermore, accelerated carbonation of alkaline residues has been found to represent an effective way to achieve CO_2 sequestration from industrial point sources, an interesting additional benefit in view of the decrease of the emissions of greenhouse gases [12–16]. However, despite the widespread experimental studies carried on so far, several aspects still need to be elucidated, especially pertaining to both the optimal operating conditions to be adopted and the leaching behaviour of some heavy metals and oxyanion-forming elements (e.g. Cr, Mo, As and Sb) for which contradictory results in terms of reduction of mobility have been reported in literature [9,17–19].

The present study focuses on the application of an accelerated carbonation treatment to APC residues, in order to assess the potential of the process in terms of sequestration of the CO_2 emitted from WtE point sources as well as the influence on the environmental behaviour of the residue under concern.

2. Materials and methods

APC ash samples were collected at a WtE plant in Cagliari, Sardinia (Italy), where the flue gas treatment system produces

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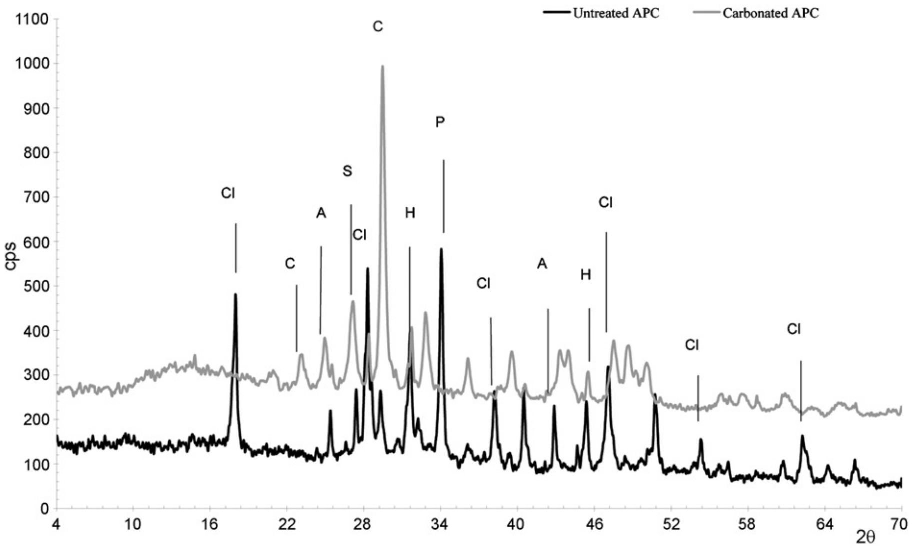

Fig. 1. Diffractometric curves for the untreated APC and carbonated APC$_{8.2}$ ash. Cl = CaClOH, C = $CaCO_3$, A = $CaSO_4$, S = KCl, H = NaCl, P = portlandite.

a residue composed of ashes coming from the heat exchanger (boiler ashes) and the particulate matter collected by the bag filters after addition of dry hydrated lime to neutralize acid components. Around 50 kg of APC ash were sampled by collecting three samples per day over a 48-h period and homogenized in order to produce representative laboratory subsamples to be used for preliminary characterization: metal and anion content, total carbon content (TC) and loss on ignition (LOI). In order to investigate the effect of the accelerated carbonation on the APC ash mineralogy and, consequently, on the environmental behaviour and CO_2 sequestration yields, release and partitioning of metals (Pb, Zn, Cr, Cu, Mo and Sb), APC ash buffering capacity and mineralogy were evaluated. The APC ash mineralogy was studied by means of X-ray diffractometric (XRD), thermogravimetric (TG) and differential thermo analyses (DTA) performed on both untreated and carbonated ash subsamples. Carbonate content was evaluated by calcimetry and TG analysis. The total metal content was determined by hot plate acid digestion, by adding 4 ml of hydrofluoridric acid, 9 ml of nitric acid and 3 ml of hydrochloridric acid to 0.3–0.4 g of dry sample. The macro element concentration was determined in platinum melting pots by alkaline fusion of 0.2–0.4 g of dry sample with 2.0–2.5 g of lithium metaborate. The mix was heated at 1050 °C, followed by dissolution of the molten material in a 10% HNO_3 solution. After both acid digestion and alkaline fusion, metals and macro elements concentration was measured by Inductively Coupled Plasma spectrometry (ICP-OES, Varian 710 ES). Soluble chlorides and sulphates were determined by using the Italian standard test procedure (UNI 8520) which involves dissolution with water and subsequent analyses by ion chromatography (Dionex, ICS-90). LOI was determined by the EN-15169 method. TC was measured using a CHN analyzer (LECO, CHN 1000).

The Cu, Zn, Pb, Cr, Sb and Mo partition in APC ash was investigated by performing the three-step sequential extraction procedure proposed by the Standards, Measurements and Testing Program of the European Union [20]. The first step allows to extract the exchangeable, water and acid-soluble (e.g. carbonates) metal fraction; the second step identifies the metal bound to the reducible fraction (iron/manganese oxy-hydroxides) and finally, the third step involves the metal bound to the oxidizable fraction (organic matter and sulphides). An additional fourth step, consisting of a strong acid attack (the same method used to evaluate the total metal concentrations), was added to allow mass balance calculations. Diffractometric analyses were performed using a RIGAKU device, with Cu Kα radiation (copper tube operated at 30 kV and 30 mA). TG analyses were performed using a TG/DTA analyzer in air atmosphere (Stanton Redford, model STA 780) equipped with a Pt/Rd crucible (V = 135 mm^3, D = 6 mm) at a heating rate of 15 °C/min. Carbonate content was measured by placing 0.5 g of APC ash subsample in a Dietrich-Fruehling apparatus, and adding 10 ml of HCl 1:1 (v/v). The buffering capacity of both untreated and carbonated APC ash was evaluated by means of the Acid Neutralization Capacity (ANC) test, by mixing several subsamples of dry material with distilled water containing pre-selected amounts of nitric acid (1 or 10 M) at a L/S = 10 and stirring the suspension for 48 h (UNI CEN/TS 15364). The effect of carbonation on metals (Pb, Zn, Cr, Cu, Mo and Sb) mobility was assessed by performing the EN 12457-2 test on the untreated and carbonated APC ash, as well as through the above mentioned ANC test. The metals concentration in the eluates was measured with ICP-OES spectrometry. Chlorides and sulphates concentration were measured by ion chromatography.

The applied accelerated carbonation treatment consisted of a single step aqueous-route process carried out at ambient conditions (20 °C and atmospheric pressure). The treatment was performed into an open vessel, by blowing CO_2 through the continuously mixed APC ash slurry (L/S = 2.5). The slurry was homogenized by both gas bubbling and a magnetic stirrer. With the aim of studying the evolution of the carbonation process, in terms of both pH decrease and effects on metals mobility, the pH value was continuously monitored and the input of CO_2 was stopped when pH reached prefixed values in the range between 13.17 (pH of untreated APC ash) and 8.22 (final pH attained during carbonation); APC ash slurry samples were collected at the end of each carbonation step in order to perform the leaching tests. In the following, APC$_{10.7}$, for instance, indicates APC ash carbonated until pH 10.7 was reached. All the samples were immediately dried at 105 °C and stored in closed bottles to prevent ageing reactions. The time required to reach the final pH of 8.22 was about 3 h.

3. Results and discussion

The results of the APC ash preliminary characterization are reported in Table 1. Calcium and chloride are the main constituents, but the content of metals such as Zn, Pb, Cu and Sb is also remarkable, in agreement with other studies reported in the literature [10,11]. The XRD analysis (Fig. 1) displayed portlandite, $Ca(OH)_2$, as the main mineral phase, high amounts of NaCl, CaClOH, and

Table 1
Chemical composition (mg/kg dry weight), water content, LOI and TC (% by weight) for the untreated APC ash residues. SD = standard deviation ($n = 3$).

Major elements	Concentration	Minor elements	Concentration
Na	20,233 ± 1256	Zn	4,372 ± 497
K	41,178 ± 2238	Cu	441 ± 26
Ca	308,069 ± 9144	Ni	86.87 ± 6
Mg	29.243	Cd	112 ± 4
Fe	1,946 ± 92	Pb	2,176 ± 101
Mn	170 ± 16	Sb	434 ± 18
Al	6625 ± 61	Mo	8.28
Si	30,002 ± 756	Cr	74.04 ± 11

Chemical-physical properties	Concentration	Anion	Concentration
LOI (%)	29.11 ± 0.21	Sulphate	157 ± 89
Water content (%)	2.52 ± 0.47	Chloride	104,118 ± 1352
TC (%)	2.22 ± 0.02		

Table 2
Comparison between the release in EN 12457-2 test on untreated, carbonated APC ash (both $APC_{10.7}$ and $APC_{8.2}$) and the release limits as derived from the Italian landfill acceptance criteria [21].

Element	Concentration (mg/kg)			Limit concentration (mg/kg)		
	Untr. APC	$APC_{10.7}$	$APC_{8.2}$	HW	*n*-HW	IW
Pb	228.00	0.03	1.59	50	10	0.5
Zn	18.13	0.05	3.05	200	50	4.0
Cr	9.36	0.09	3.58	70	10	0.5
Cu	0.65	0.01	0.26	100	50	2.0
Mo	4.60	0.08	0.51	30	10	0.5
Sb	0.01	0.17	5.81	5.0	0.7	0.06
Cl	104,118	112,400	98,491	25,000	15,000	800

Na_2SO_4 crystalline phases and a little amount of $CaCO_3$, indicating a partial degree of natural carbonation of the fresh material. The XRD performed on the carbonated APC ash showed the disappearance of the $Ca(OH)_2$ peak, whilst the calcite peak was more evident, consistently with the applied carbonation process. The TC content increased after the complete carbonation treatment from 2.22% to 6.93%, due to the carbonates produced during the process.

The acid titration curves obtained for both the untreated APC ash and the same residue after the last step of the carbonation treatment ($APC_{8.2}$) are depicted in Fig. 2. It can be observed that the buffering capacity of the APC ash was altered after the treatment. The shape of the curve related to the untreated APC ash, evidences a plateau at pH around 12, corresponding to the presence of calcium hydroxides buffering systems [14]. Due to the decrease in the free oxides and hydroxides content and the subsequent precipitation of calcite induced by carbonation, the ANC curve after the treatment showed a significant buffering capacity between neutral and alkaline values, as pointed out by the wide plateau at pH around 7–8.

As far as the effect of carbonation on the mobility of metals was concerned, Fig. 3 reports the release of Pb, Zn, Cu, Cr, Mo and Sb as a function of the pH reached at different stages of carbonation. The leaching from untreated APC ash, as resulted from ANC test, is also reported.

As for the observed leaching trends, it can be assessed that the leachability from both untreated and carbonated APC residues was largely controlled by the solution pH. Leaching of Pb, Zn and Cu was characterized by a V-shaped release curve, typical of amphoteric metals. A certain degree of pH dependency was observed also for Cr leaching (increased mobility at higher pH values) and Sb (increased mobility at lower pH values). As far as Mo leaching was concerned, the data did not seem to follow a clear trend, thus any dependence from pH cannot be underlined. When comparing the leaching behaviour of untreated and carbonated APC ash, it can be stated that the release from the carbonated material was generally lower than that from the untreated APC ash, with the only exception of Sb. Indeed, the leaching trends at the different pH indicated a higher mobility of Pb, Zn, Cu and Cr from untreated APC ash as compared with the carbonated one. The positive effect of the carbonation can be noticed especially when comparing the release from the untreated APC ash with that from the carbonated one till a pH of 10.5–10.7. As a result of the carbonation treatment, the amount of Pb released was reduced by 4 orders of magnitude, the release of Zn and Cu of 2 orders of magnitude, whilst the mobility of Cr and Mo was reduced of one order of magnitude. Conversely, at the same level of carbonation, the negative effect on Sb mobility corresponded to an increase of two orders of magnitude. Based on the demobilization effect assessed for Pb, Zn, Cu, Cr and Mo, the optimum final pH for the carbonation treatment was estimated to be close to 10.5. These results are consistent with the study of Wang et al. [11], who found the best effect of carbonation, in terms of reduction of metal mobilization, at pH of 9.5–10.5. As observed in Table 2, if the overall performance of the treatment is evaluated in view of final disposal, i.e. by comparing the EN 12457 eluates concentrations with the Italian limits for disposal in landfills for inert, non hazardous and hazardous waste [21], it can be concluded that accelerated carbonation was effective; in fact, as far as only metals and oxyanions are considered, untreated APC ash is classified as hazardous waste, whilst carbonated $APC_{10.5}$ would be allowed for disposal in landfills for non-hazardous waste. However, it has to be emphasized that leaching of chloride, which is also a critical species for APC ash in view of landfilling, was not affected by carbonation treatment (see Table 2), according to results obtained by other authors [10,11] but in disagreement with the study reported in [13], where a reduced leaching of chlorides and sulphates was obtained following carbonation. On the basis of the results obtained in the present study, with the aim of classifying the waste as non-hazardous with respect to final disposal, an additional treatment aiming at removing chlorides should hence be provided.

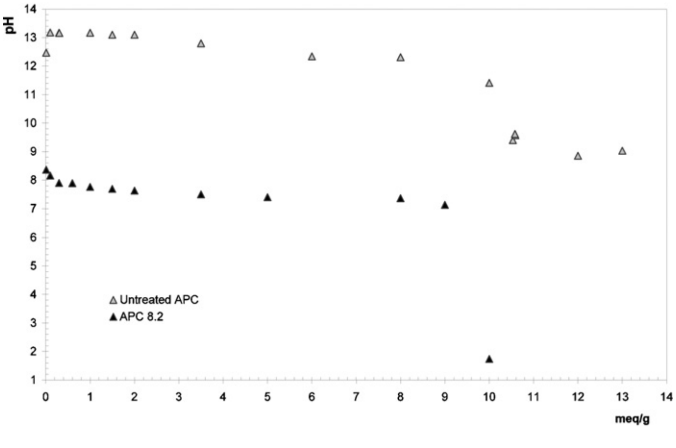

Fig. 2. ANC curves for the untreated APC and carbonated $APC_{8.2}$ ash.

The positive effect exerted by carbonation on the Pb, Zn, Cu, Cr and Mo leaching could be related with the chemical and mineralogical transformations induced by carbonation [6,13,17]: (i) carbonation generates a pH decrease to values at which several metals exhibit their minimum solubility; (ii) both carbonation and pH decrease cause the conversion and precipitation of metal oxides and/or hydroxides to metal carbonates, that are less soluble; (iii) an additional retention mechanism is based on the affinity of some metals with calcite, thus to possible sorption onto $CaCO_3$ and/or coprecipitation into newly formed minerals. The higher release observed for Sb after carbonation could be explained taking into account its oxyanionic nature: often Sb in high temperature solids is incorporated in ettringite-like minerals and/or Ca antimoniate,

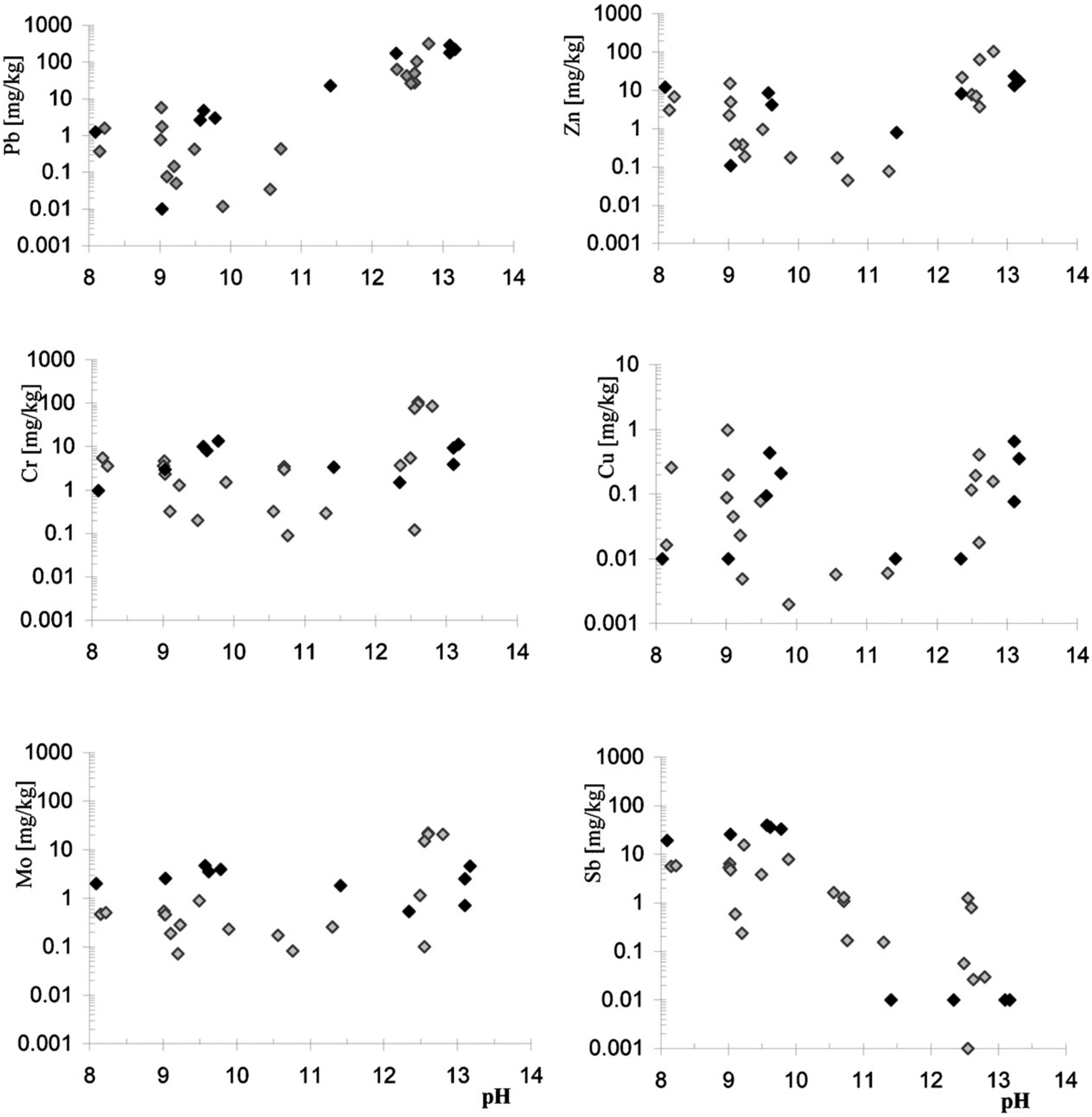

Fig. 3. Metal release vs pH in EN 12457-2 leaching test on treated APC ash at different stages of carbonation (◊) and metal release vs pH in ANC test on untreated APC ash (♦).

which dissolve upon carbonation [10,17]; however, it has to be underlined that the mineralogical characterization on the fresh material did not evidence presence of such mineral phases, at least above the XRD detection limit.

The results of the metal partitioning assessed for both the untreated and carbonated $APC_{10.7}$ ash are reported in Fig. 4.

As far as the untreated APC ash was concerned, all the elements of interest were extracted mainly under oxidizing conditions (step 3: metals bound to organic matter and sulphides). Copper was also present in the reducible fraction (step 2; 35%) and a relevant amount of Pb was found to be bound to the soluble, exchangeable and carbonate fraction (step 1; 24%).

In the case of Sb, a negligible amount was found to be bound to the soluble, exchangeable and carbonate fraction (0.5%), exhibiting a low mobility under natural/acid conditions. As shown in Fig. 4, the carbonation treatment did not affect the elements distribution in the residual fraction, whilst the influence on the other fractions was of different extent depending on the element under concern. Lead and Cr amounts bound to the soluble, exchangeable and carbonate fraction decreased with benefit for the oxidizable fraction, whilst it increased for Zn, Mo and Sb, to detriment of the oxidizable fraction. The evolution of the soluble, exchangeable and carbonate fraction during carbonation is of particular interest, as the metals mobility is mostly related to this fraction. However, the mobility assessed through the EN 12457-2 leaching test takes into account only the soluble fraction, so that data are not directly comparable. The Pb and Cr reduced mobility assessed by performing the EN 12457-2 test could be explained also by the SE data, hypothesizing an increased affinity of these metals to organic matter and sulphides after the carbonation treatment. Ferrari et al. [22] reported that the organic matter in APC ash is composed mainly by elemental carbon and not-extractable organic carbon which are water insoluble. As far as Zn and Cu were concerned, given the reduced mobility assessed after the treatment, the increase of the soluble, exchangeable and carbonate fraction should be interpreted in terms of increase of the carbonate one, which is less soluble

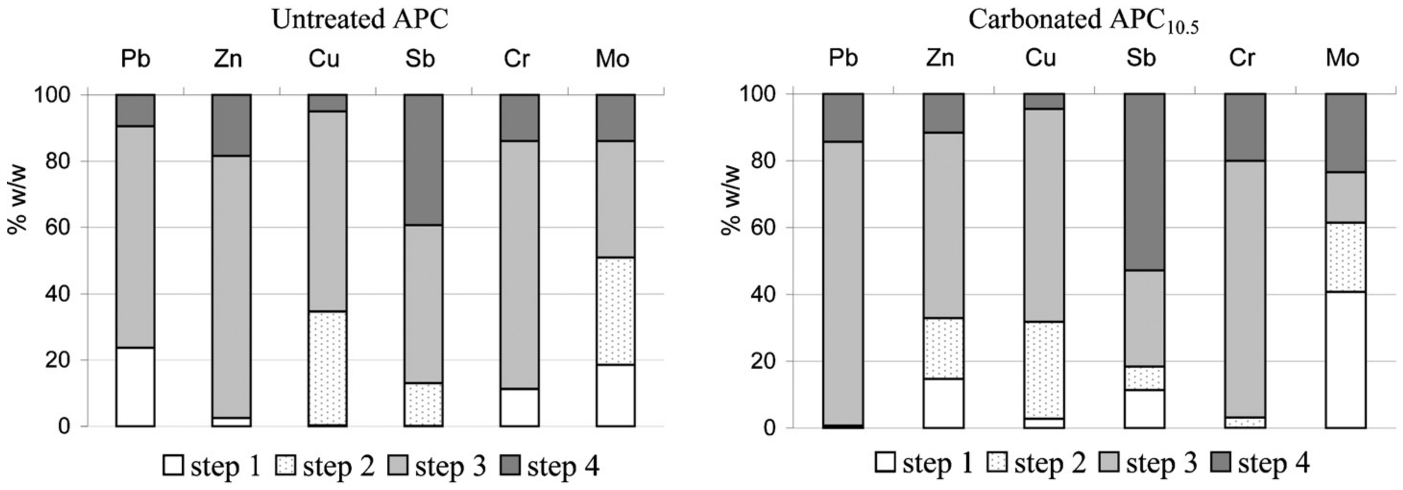

Fig. 4. Elements partitioning assessed by the sequential extraction procedure of untreated and carbonated $APC_{10.7}$ ash.

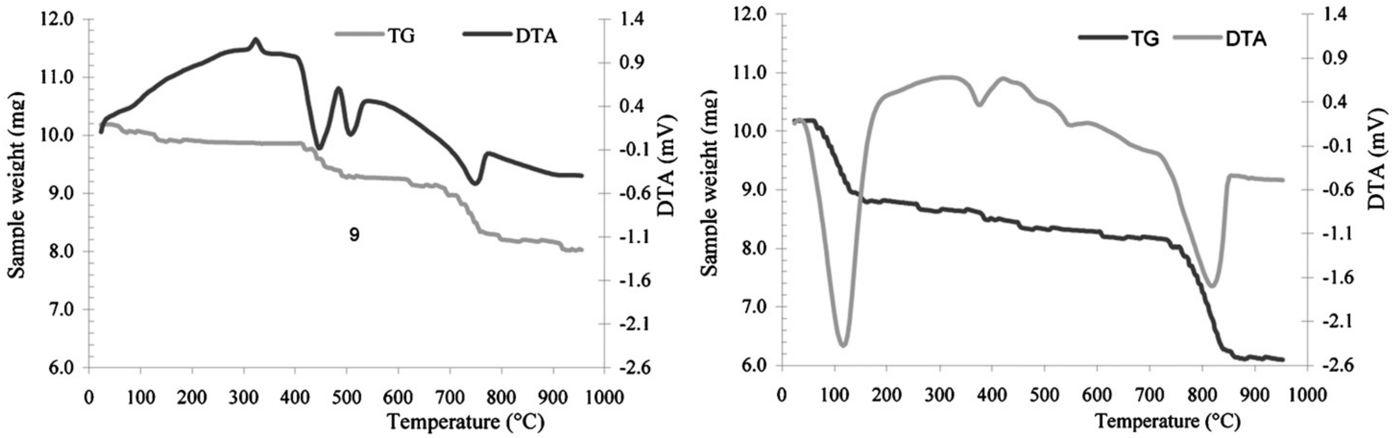

Fig. 5. TG/DTA results on untreated (a) and carbonated (b) APC ash.

than (hydr)oxides present in untreated APC residues [11]. As for Mo, a significant increase of the amount bound to the soluble, exchangeable and carbonate fraction was assessed after the carbonation, consistent with the data presented by Wang et al. [11], whilst the amount bound to both reducible and oxidizable fractions decreased. As for Sb, the SE showed an increase of element bound to the soluble, exchangeable and carbonate fraction, probably due to sorbing onto neo forming $CaCO_3$ [23].

The CO_2 sequestration potential of APC ash was evaluated by both calcimetry and TG analyses. As it is well known, during the TG analysis the weight loss that occurs in the range of 650–850 °C could be directly linked to the carbonate decomposition. As reported in Fig. 5, for the untreated APC ash, the DTA curves showed three steps of weight loss (Fig. 5a): a first one between 300 and 400 °C, associated to organic matter oxidation; the second one between 400 and 500 °C caused by $Ca(OH)_2$ decomposition; a third one, at 750 °C, related to $CaCO_3$ and NaCl decomposition.

In the carbonated APC ash (Fig. 5b), water hydration loss and gypsum dehydration were evident in both TG and DTA curves at a temperature close to 100 °C; the oxidation of organic matter occurred in a range between 200 and 450 °C, and finally at 850 °C the loss of weight was due to $CaCO_3$ decomposition. The carbonate content was evaluated by the percent loss weight at 600–850 °C, before and after loss weight, with respect to the initial sample weight. The amount of CO_2 sequestrated by the residues, on the basis of TG analysis, was estimated by calculating the difference, after and before treatment, of the weight loss in the range of 700–900 °C, with respect to the initial weight of APC ash, taking into account the weight increase upon carbonation. Calcimetry results agreed with the data obtained during TG analysis, with an estimated CO_2 uptake of about 200 g/kg. This result is consistent with several other experiences [10,12,14], where a maximum CO_2 uptake of as much as 250 g/kg is reported, but using high pressures and temperature conditions, differently form the mild conditions adopted during this experimental work.

4. Conclusions

The study presents the assessed effects of an aqueous phase accelerated carbonation treatment performed on MSWI combustion APC ash at different final pHs values. The treatment was able to modify some important properties of APC ash, such the mineralogy, the metals partitioning and mobility. As far as the metals mobility was concerned, carbonation proved to be effective in reducing the release of Pb, Zn, Cr, Cu and Mo, with an optimal final pH, identified on the basis of the demobilization effect, close to 10.5. Nevertheless, along the whole studied pH range ($13.17 < pH < 8.22$), a mobilization effect was noticed for Sb. On the basis of the experimental findings, it could be assessed that the accelerated carbonation treatment exerted a positive effect on the mobility of several elements, but the combination with other process (e.g. sorbent adding) might be necessary to immobilize Sb. Moreover, it has to be underlined that the carbonation treatment does not modify the chloride mobility, thus the carbonated residue should still be classified as hazardous waste. To this regard, an additional washing treatment could be appropriated to remove salts from the material, especially if performed after a carbonation step.

As far as the CO_2 sequestration capacity of APC ash is concerned, the CO_2 uptake evaluated during the accelerated carbonation treatment showed that the residue could be suitable for CO_2 storage, even if the quantity of APC ash produced by incineration plants is relatively low if compared with the WtE plants CO_2 emissions [9].

Accelerated carbonation treatment can be considered as potentially viable option in order to improve the environmental behaviour of APC ash and reduce CO_2 emissions from Waste to Energy plants.

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