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Waste silica sources as heavy metal stabilizers for municipal solid waste incineration fly ash

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Abstract The present work discusses a new method, based on the use of silica fume, for heavy metal stabilization. The inertization procedure is reported and compared with other technologies, involving the employ of amorphous silica as stabilizing agent for municipal solid waste incinerator fly ash treatment (i.e. colloidal silica and rice husk ash). The obtained final materials are characterized in terms of chemical composition and phase analysis.

The reported method, realized at room temperature, employs all waste or by-product materials. As a consequence it appears to be economically and environmentally sustainable.

1. Introduction

Municipal solid waste incinerator (MSWI) fly ash (FA) is classified as hazardous waste, because of its leaching behavior related to heavy metals, soluble salts, and sometimes to traces of chlorinated organic compounds. However, it contains also mineralogical phases such as anhydrite ($CaSO_4$), quartz (SiO_2), gehlenite ($Ca_2Al(AlSi)O_7$), hematite (Fe_2O_3), and portlandite ($Ca(OH)_2$) (Quina et al., 2011), that can have interesting characteristics for the possible re-use of this ash, for example, as filler materials.

According to the literature, their main potential applications are in construction materials (Zacco et al., 2012), such as cement, concrete, ceramics, glass and glass–ceramics or geotechnical applications (Quina et al., 2008). Very recently it was shown that inertized MSWI fly ash has a very promising new application field: it can be used as a filler of polymer materials, producing an enhancement of its mechanical properties (Besco et al., 2013).

Obviously the employ of this waste as secondary hand-made material necessitates of suitable stabilization/inertization procedures.

In order to utilize the waste and reduce the environmental impact, the treatments for FA can be grouped into three classes (Chandler et al., 1997): (1) separation processes; (2) solidification/stabilization (S/S); (3) thermal methods. For a comprehensive review about these technologies see Reference (Zacco et al., 2014).

The main problem in the use as a filler of MSWI fly ash based-materials, is due to the presence of high amount of soluble salts. Recently water extraction method was considered as a useful pre-treatment of FA for extracting many problematic substances from the ash matrix, which opens the possibility for further recovery and recycling (Liu et al., 2009; Quina et al., 2011). However, this involves leaching of heavy metals and treatment of high amount of wastewater.

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It was recently proposed a new method for MSWI fly ash inertization, based on the use of colloidal silica (Bontempi et al., 2010). It was based on the use of commercial amorphous silica and other fly ashes, like coal and flue gas desulfurization (FGD) fly ashes (Bontempi et al., 2009). The obtained inert was demonstrated to be very promising as a filler for polypropylene (Besco et al., 2013).

This method has the great advantage of stabilizing heavy metals, with the possibility to recover almost pure soluble salts, in a next washing process.

In view of the need to move toward more sustainable and economic convenient inertization procedures, it was very recently proposed an improvement of the technology based on the amorphous silica employ: silica source was extracted from agricultural waste, i.e. rice husk ash (RHA) (Bosio et al., 2013a,b).

In the present paper a new methodology of MSWI fly ash inertization, based on the use of waste amorphous silica source is proposed (Bontempi et al., 2012). Silica source is done by silica fume.

Silica fume is a byproduct of the smelting process in the silicon and ferrosilicon industry. It is also known as micro silica, condensed silica fume, volatilized silica or silica dust. It consists of very fine particles of amorphous silica. Because of its extreme fineness and high silica content, silica fume is a highly effective pozzolanic material (Khan and Siddique, 2011). As a consequence it is employed as a substitute for fine aggregates in cement and concrete, in bricks and ceramic tiles, as well as filler in plastics and paints (Siddique, 2011). Recently silica fume was also employed to produce mesoporous silica (Zhu et al., 2013). It was very recently shown that silica fume can be used to stabilize As in coal fly ash (Rodella et al., 2013).

In the present work the new inertization technology is presented for the first time, and the obtained material is compared with the corresponding obtained by inertization of MSWI fly ash by using colloidal silica and RHA.

2. Materials and methods

For the new method, the inertization procedure was the following: 18 g of silica fume was mixed with 200 g of a mixture of three different fly ash typologies (fly ash (FA) from municipal solid waste incinerator, flue gas desulfurization residues (FGD) and coal fly ash at relative weight percentages of 65%, 20% and 15%, respectively). 200 ml of water was added followed by mixing for 1 h. The process was performed at room temperature.

As a comparison, the inertization procedures, by using colloidal silica and RHA were done, following the method reported in references (Bontempi et al., 2009, 2010, 2012; Bosio et al., 2013a,b). The corresponding samples are named S1 and S2 respectively. The sample obtained by using silica fume is named S3.

Table 1 summarizes the quantity and typology of all materials employed for inertization, obtaining samples S1, S2 and S3. All employed fly ashes were collected from power plants located in northern Italy.

Leaching tests were performed according to the CEN normative (CEN EN 12457-2), in order to quantify the leachability of heavy metals in water. The contact time of materials and aqueous solution was 2 h. This time was considered sufficient to establish equilibrium in subsequent experiments with MSWI fly ash (Yanga et al., 2009).

After chemical stabilization of heavy metals, the solid inertized material can be washed out to recover almost pure soluble salts (Bontempi et al., 2010).

The MSWI fly ash and the inertized material (S1, S2 and S3) were digested in acid solution by means of CEM Discover Microwave for chemical composition analysis. Samples were prepared using 0.18 g of powder sample in a solution of 2 ml HNO_3 (65 vol.%), 2 ml HF (47 vol.%) and 2 ml HCl (37 vol.%) (Instrument parameters: temperature 210 °C, time 9 min, power 300 W, medium stirring).

Chemical analysis was done by means of Total Reflection X-ray Fluorescence technique (TXRF).

TXRF analysis was made by the Bruker TXRF system S2 Picofox (air cooled, Mo tube, Silicon-Drift Detector), with operating values of 50 kV and 750 μA using an acquisition time of 600 s. TXRF quantitative analysis of the suspended samples was performed by the internal standard procedure. A proper amount of gallium, used as an internal standard element, was added (Borgese et al., 2009).

Leaching tests were made after the samples were naturally dried (one month after the inertization). The same tests were repeated one month later, to verify the materials' stability.

2D X-ray diffraction (XRD^2) measurements were made in reflection mode on a Dymax-RAPID X-ray microdiffractometer, with a cylindrical imaging plate detector, that allows collection from 0 to 160° (2θ) horizontally and from -45 to $+45°$ (2θ) vertically upon using CuKα radiation. A collimator diameter of 300 μm was used. The exposure time was 40 min for each XRD^2 pattern. Conventional XRD patterns were then obtained by integration of 2D images. Two-dimensional XRD measurements were made to verify and avoid possible microstructural effects (see discussion).

3. Results and discussion

Fig. 1 reports the results of leaching tests performed on MSWI fly ash and all samples, one and two months after the inertization, to verify samples' stability. Results of leaching of all detectable elements are reported. The figure scale is logarithmic, to highlight the change in the leaching of all elements, considering differences in several magnitude orders. It appears that most soluble elements are K, Ca, Br and Cl. Na is not reported because it is not detectable by means of TXRF, but

Table 1 Samples' composition.

Sample	MSWI fly ash (FA) g	Colloidal silica g	RHA g	Silica fume g	FGD residues g	Coal fly ash g
S1	130	50	–	–	40	30
S2	130	–	18	–	40	30
S3	130	–	–	18	40	30

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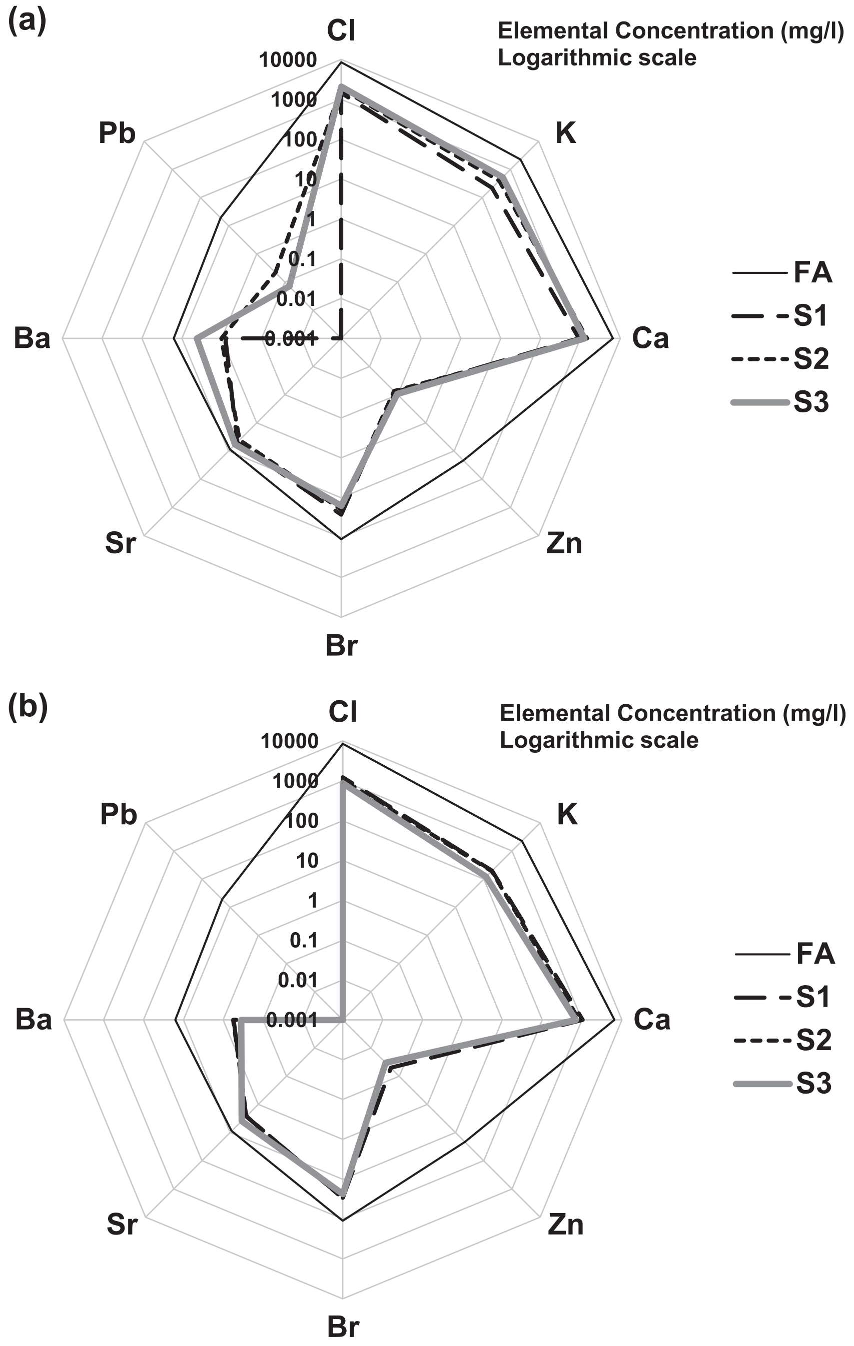

Figure 1 Concentrations of element (mg/l) of the leached solutions performed on MSWI fly ash and S1, S2 and S3 samples, one and two months after the inertization.

it is expected to be present in high concentrations (Struis et al., 2013; Bontempi et al., 2010). Moreover these elements will be recovered in soluble salts, after the inertization process.

Comparing the concentrations of heavy metals of FA and samples S1, S2 and S3, it appears evident that the three inertization processes are effective in Pb and Zn reduction, as already reported for the technologies using colloidal silica and RHA. In particular, these metal concentrations are reduced by at least two orders of magnitude. However, in comparing the data, it appears that sample S1 after one month, shows Pb and Zn concentrations lower than those of corresponding S2 and S3 samples (Pb concentrations are about 0.22 and 0.07 ppm respectively for samples S1 and S2). Fig. 1b reports the results of leaching tests, two months after the inertization process. In this case it is evident that it is very difficult to distinguish results obtained from the three different processes: the concentrations of the leachable elements, in particular the most interesting Pb and Zn, are nearly the same (Pb has values lower than DL). These results mainly highlight that, despite the difference in kinetics, the three inertization processes produce comparable results in terms of metal stabilization. In addition, it also demonstrates that the obtained

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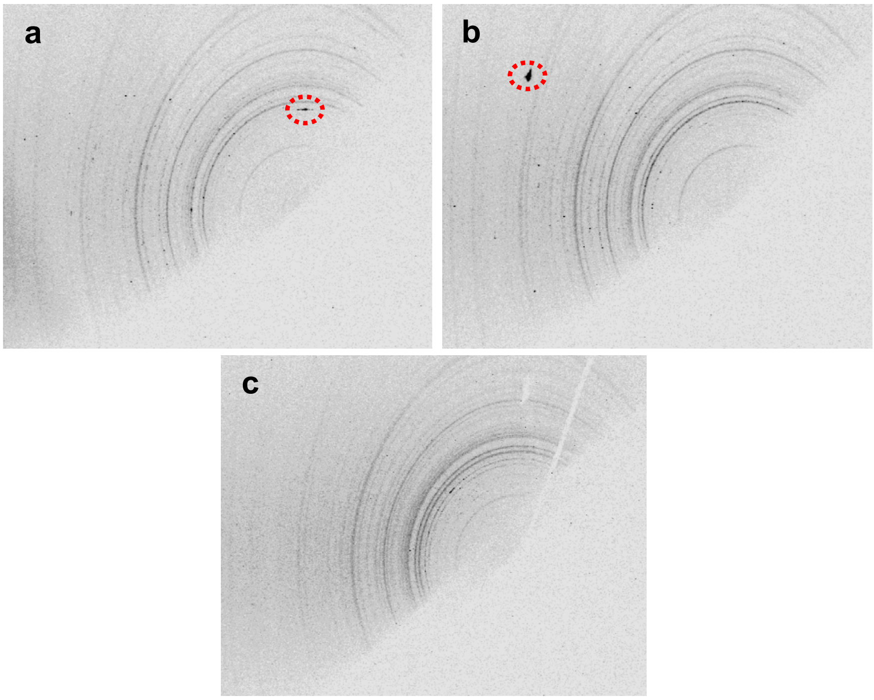

Figure 2 2D diffraction images obtained on the FA samples, measuring in two different points (a and b) and 2D diffraction image collected with sample spinning (c). Red circles highlight spots (in 2a at about 26.4° 2θ and in 2b at about 65° 2θ) that can be attributed to crystalline silicon dioxide (quartz).

inertized materials are stable, and chemical stability improves in time.

To study the differences in obtained final inertized materials, all samples were analyzed by means of XRD. Indeed, to understand the stabilization mechanism it is essential to verify the mineral phase that can be associated to each element.

In addition samples were also digested and analyzed by TXRF to verify the eventual differences in composition.

To verify the presence of crystalline phases with large grains, 2D diffraction images were collected in laboratory. 2D diffraction (XRD^2), using a two-dimensional detector, allows to increase the amount of scattered X-ray observed, with respect to a point detector.

To the best of our knowledge there are no papers in the literature reporting 2D X-ray diffraction images collected of MSWI fly ash.

Fig. 2 reports the 2D diffraction images obtained on the FA samples, measuring in two different points.

Comparing Fig. 2a and b, it appears that these two images not only show continuous Debye rings, but also some spots in the XRD^2 patterns. It is evident that sample is inhomogeneous, due to the presence of large grains, producing different contributions in the scattered intensities along a single Debye ring. For example, in Fig. 2a a spot is highlighted at about 26.4°. In Fig. 2b a spot at about 65° is also indicated. They can be attributed to crystalline silicon dioxide (quartz).

Continuous rings are characteristic of phases with a fine microstructure, while spotty rings are typical of coarse grained materials.

As a consequence of samples' microstructure, 1D conventional XRD patterns may present diffraction peak intensities of some phases not correlated to phase content in the investigated mixture. The works devoted to quantitative analysis of MSWI fly ash by means of XRD (Rietveld method) often do not take into account these microstructural effects.

To overcome this XRD^2 images on all samples were collected with the sample spinning. For FA, Fig. 2c reports the 2D diffraction image collected in this way. It is evident that this microstructural effect is avoided.

Fig. 3 reports the integrated intensities obtained from 2D-XRD patterns collected on all samples. All the patterns were corrected by background subtraction.

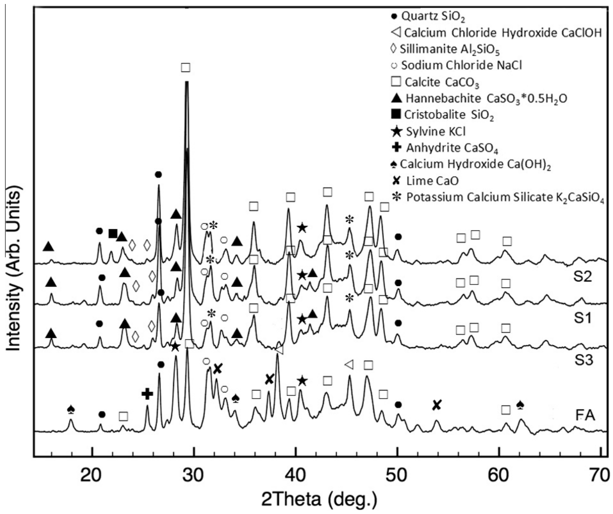

Figure 3 Integrated intensities obtained from 2D-XRD patterns collected on all samples. All the patterns were corrected by background subtraction.

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The mineralogy of FA involves an assemblage of crystalline and amorphous phases. This is the consequence of several processes including vaporization, melting, crystallization, vitrification, condensation and precipitation occurring during the combustion and the air pollution control treatment (Eighmy et al., 1995).

MSWI fly ash shows the presence of calcium hydroxide, quartz, anhydrite, calcite, lime, sylvite, calcium chloride hydroxide, and sodium chloride.

XRD patterns of S1, S2 and S3 are very similar. They show the presence of several crystalline phases: hannebachite, quartz, calcite, sodium chloride, and sylvite. There are also some peaks that may be attributed to silicates.

As already reported (Bontempi et al., 2010), the calcium hydroxide and calcium chloride hydroxide phases, that can be found in MSWI fly ash, cannot be found in corresponding inertized materials, due to their reaction with carbon dioxide (the carbonation reaction), to produce calcium carbonate. Indeed, the peak of calcite is more intense in samples S1, S2 and S3 with respect to the MSWI fly ash, demonstrating the increase of $CaCO_3$ in inertized samples.

A deeper observation of the XRD patterns allows to find the presence of a different crystalline phase for sample S2: the peak at about 21.8° can be attributed to the cristobalite.

Cristobalite is naturally present in the RHA, due to the combustion of starting rice husk (Bosio et al., 2013b). Indeed, as shown in the literature, the crystalline silicon dioxide formation depends on the burning temperature of starting rice husk (Xu et al., 2012). Amorphous silicon is very reactive during sample inertization. On the contrary, Cristobalite is a non reactive phase found also in the final inertized material.

Table 2 reports the chemical composition of MSWI fly ash and all inertized samples (S1, S2 and S3), obtained by means of TXRF analysis after the digestion procedure. Carbon, hydrogen, oxygen and sodium were not analyzed, because they cannot be detected by the technique. Cl was not calculated because of the employ of HCl in the extraction procedure. Si is not quantified, because of its presence in the sample carriers. The major elements in the MSWI fly ash are Al, Ca, K, Fe, Ti, Zn and S. High concentrations of P, Pb Cu, Cd, and Mn were also detected. The reported values are in accord with chemical composition of other MSWI fly ash, reported in the literature (Chou et al., 2009). However, the Zn and Pb concentrations are lower with respect to the reported ones. This may be justified by local variability of MSWI fly ash, but another reason may be due to the possible volatilization of Pb and Zn, during the digestion procedure. Indeed, as recently reported (Struis et al., 2013), the employed MSWI fly ash contains these two metals also in form of chlorides. Chlorine-compounds with heavy metals have relatively low boiling points and high vapor pressure. That is the reason why these metal chlorides are more volatile (Wang et al., 1999). However, the goal of this analysis is to compare the results obtained from samples inertized in three different methods.

S1, S2, and S3 samples contain a large amount of Al, S, K, Ca, Ti, Fe and Zn. The composition of the three samples, considering experimental errors, is quite comparable. The S content is increased with respect to FA, because of the addition of FGD residues. The concentrations of leachable heavy metals, i.e. Zn and Pb are reduced, with respect to the dilution factor.

S2 contains larger amount of Mn, with respect to S1 and S3, because of its presence in original RHA. Indeed, in accord with our data, it was reported that manganese is one of the most common metals present in RHA (Della et al., 2002).

In conclusion it was shown that the new reported inertization procedure of MSWI fly ash, based on the use of silica fume, produces results comparable with those already found for the starting technology, employing commercial colloidal silica. The technology based on the use of RHA allows to obtain a final inertized material with few differences: the final product can contain higher metal quantities, as for example

Table 2 Chemical composition of MSWI fly ash, RHA, Silica fume and all inertized samples (S1, S2 and S3), obtained by means of TXRF analysis after the digestion procedure.

Element	MSWI fly ash ppm	RHA ppm	Silica fume ppm	S1 ppm	S2 ppm	S3 ppm
Al	24,300 ± 12,800	ND	ND	19,000 ± 4000	18,500 ± 1500	19,000 ± 3000
P	3500 ± 300	2150 ± 180	500 ± 150	2100 ± 300	2150 ± 150	2050 ± 340
S	6200 ± 1100	2200 ± 600	3300 ± 1300	12,300 ± 2600	9500 ± 500	11,000 ± 2000
K	16,700 ± 2200	15,400 ± 800	13,000 ± 1000	2000 ± 300	11,500 ± 1000	10,500 ± 800
Ca	205,000 ± 21,000	8100 ± 900	1700 ± 150	150000 ± 18,000	131200 ± 15,000	132,000 ± 15,000
Ti	9300 ± 800	40 ± 10	ND	5300 ± 400	5500 ± 400	5500 ± 350
V	40 ± 15	ND	ND	55 ± 16	65 ± 10	65 ± 15
Cr	300 ± 30	ND	ND	170 ± 30	170 ± 15	150 ± 10
Mn	470 ± 30	1200 ± 80	265 ± 15	280 ± 20	415 ± 25	300 ± 25
Fe	12,300 ± 1100	630 ± 60	610 ± 60	9400 ± 1000	8800 ± 800	8600 ± 600
Ni	140 ± 10	5 ± 1	ND	150 ± 15	120 ± 10	100 ± 10
Cu	650 ± 50	15 ± 2	22 ± 4	430 ± 25	400 ± 25	350 ± 25
Zn	5900 ± 400	140 ± 10	100 ± 10	2800 ± 160	2900 ± 200	2800 ± 200
As	ND	14 ± 2	20 ± 5	ND	ND	ND
Se	ND	ND	7 ± 1	8 ± 2	10 ± 1	10 ± 2
Rb	30 ± 5	6 ± 1	37 ± 3	20 ± 5	18 ± 2	20 ± 4
Sr	370 ± 40	20 ± 3	ND	210 ± 50	190 ± 25	180 ± 30
Cd	720 ± 180	ND	ND	550 ± 140	400 ± 80	460 ± 70
Sb	ND	ND	400 ± 50	ND	ND	ND
Pb	900 ± 70	1.9 ± 0.2	14 ± 2	380 ± 50	420 ± 30	390 ± 50

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Mn, that was found in concentration higher than 400 ppm, due to the original metal presence in RHA; in addition cristobalite is also present in the final mixture, due to the thermal crystallization of silica contained in rice husk.

4. Conclusions

This paper reports, for the first time, a new inertization technology, for MSWI fly ash, based on the use of silica fume as stabilizing agent. This method involves also the employ of FGD and coal fly ashes. As a consequence, only waste (or by-product) materials are employed in the process. The treatment is performed at room temperature and soluble salts, not containing heavy metals, can be recovered after inertization. This work shows that the obtained inertized material is very similar to corresponding samples obtained by using colloidal silica or RHA as amorphous silica source.

All these characteristics make the method environmentally sustainable.

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