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Assessment of Pb-slag, MSWI bottom ash and boiler and fly ash for using as a fine aggregate in cement mortar

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Abstract

Three types of wastes, metallurgical slag from Pb production (SLG), the sand-sized (0.1-2 mm) fraction of MSWI bottom ash from a grate furnace (SF), and boiler and fly ash from a fluidised bed incinerator (BFA), were characterized and used to replace the fine aggregate during preparation of cement mortar. The chemical and mineralogical behaviour of these wastes along with the reactivities of the wastes with lime and the hydration behaviour of ordinary Portland cement paste with and without these wastes added were evaluated by various chemical and instrumental techniques. The compressive strengths of the cement mortars containing waste as a partial substitution of fine aggregates were also assessed. Finally, leaching studies of the wastes and waste containing cement mortars. Formation of expansive products like ettringite, aluminium hydroxide and H₂ gas due to the reaction of some constituents of BFA and SF with alkali creates some cracks in the paste as well as in the cement mortars, which lower the compressive strength of the cement mortars. However, utilization of all materials in cement-based application significantly improves the leaching behaviour of the majority of the toxic elements compared to the waste as such. © 2007 Elsevier B.V. All rights reserved.

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1. Introduction

Management and treatment of solid wastes generated by different industries as well as of municipal waste has taken more and more importance. These wastes range from rather inert, e.g. glass bottles, excavated soil, building and demolition waste, to hazardous waste with high concentrations of heavy metals and toxic organic compounds.

Thermal treatment of municipal solid wastes generates different types of solid residues [1]. Although some of these contain rather high concentrations of heavy metals and toxic organic compounds, there exist several possibilities for using, e.g. the bottom ash in construction applications [2,3]. Moreover, ferrous and non-ferrous industries are among the most important

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producers of solid wastes that contain heavy metals, such as metallurgical slag, wastes from the air pollution control technology applied in these industries, and various sludges produced from blast furnaces. Land filling or recycling as construction material is the main management option for these wastes [3,4]. Provided the heavy metal concentration and the physico-chemical characteristics permit, recycling is to be preferred, both in view of environmental considerations (less disposal) and of resource management. Solid waste can be recycled as such in granular applications or after solidification/stabilization in monolithic form [5,6]. In the latter option cement and water are added in order to form a monolithic product. This technique was originally applied in view of land filling in order to decrease heavy metal leaching and to limit transport of components into the environment. In view of recycling of the material, civil-technical requirements such as sufficient strength, durability as well as environmental properties such as leaching of toxic elements from product must be met [7].

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Two types of applications of the waste materials considered are: utilization after grinding as a pozzolanic additive in cement and use as a granular material to partially substitute the fine and coarse aggregates of cement mortar and concrete. The useful application of some waste materials in mortar and concrete is nowadays considered a possible option in the waste management policy of several countries such as the USA, Denmark, The Netherlands and Belgium [3]. Indeed, materials such as fly ash from coal combustion in power plants have pozzolanic properties and are already used as a pozzolana for a long time with cement [8]. Ground-granulated blast furnace (GGBFS) has been used for many years as a supplementary cementitious material in Portland cement concrete, either as a mineral admixture or as a component of blended cement [8–10]. Bottom ash from coal combustion in power plants and blast furnace slag from iron production may partly substitute the sand fraction in mortar and concrete [11]. Sludge ash generated from MSW incineration has been reported as a filler material with cement and as a fine aggregate in concrete [12,13]. Prior treatment of the waste is often needed to decrease the concentration or mobility of hazardous components [3,14–16].

Limited uses of slags generated from non-ferrous metal (Pb, Zn, Cu, Ni and Cr) processing industries have so far been reported, whereas slags generated from iron processing industry are widely used in construction. Zn and Pb-slag, e.g. is produced during pyrometallurgical treatment of the sulphide ores and has been used as an aggregate in road construction [17,18] and in concrete [19–21]. The major problem in using Pb or Zn-slags as a secondary material in road construction and in concrete preparation is obviously the presence of high amounts of toxic elements like Pb, Zn and Cu, which may be leached by water. The pollution caused by these toxic elements in the vicinity of non-ferrous slag dumping site is of much concern from an environmental point of view [22].

The MSW incineration process generates various inorganic wastes, the major ones being bottom ash and fly ash. The characteristics of ashes are influenced by the incineration parameters like furnace type and temperature, capacity, waste input [3]. Utilization of different types of ash generated from MSW incineration in the cement and concrete industry, e.g. as a raw material for clinker production (fly ash) [23,24] or as fine and coarse aggregates to replace sand and gravel in cement mortar and concrete preparation (bottom ash) [14,25,26] or as a pozzolanic additive into cement [27–29] have been reported. MSWI bottom ash is widely used as a material for road construction or in road base [18,30–32]. Most of the MSWI bottom ashes used as an aggregate in concrete have a particle range of 2–40 mm.

In this paper, we will focus on the use of two types of MSWI ashes with particle size <2 mm, generated from two different MSW incinerators, the sand-sized (0.1–2 mm) fraction of MSWI bottom ash from a grate furnace (sand fraction, SF) and boiler and fly ash from a fluidised bed incinerator (BFA) as a fine aggregate in preparation of cement mortar and concrete. The metallurgical slag from Pb production (SLG) is also considered in this study as the particle size of SLG is such that it can also be used as a fine aggregate in preparation of cement mortar

and concrete like the other two ashes. A comparative evaluation of physico-chemical, mineralogical and technical characteristics of these wastes as well as their reactivity in cement will be reported. Also the environmental properties of these wastes and the cement-based products will be reported and evaluated. It will be shown that the combined technical and environmental evaluation of the wastes is invaluable for their use in construction.

2. Experimental

2.1. Materials

The metallurgical slag is a residue from a lead blast furnace. After separation of the slag from the bullion, the residue is cooled down and broken into finer pieces. The fraction used in this study is the 0–4 mm fraction (SLG) and the generated amount is about 35,000 t/year. The SLG is a very hard black colour granular material.

The MSWI bottom ash from which the sand fraction (SF) used in this study was derived underwent an on-site treatment after quenching [16]. This wet treatment – consisting of various sieving and washing units – separated the light organic fraction and the fraction larger than 50 mm from the main stream, and sent these back into the incinerator. The main stream went through a wet sieving process to produce three fractions: \emptyset 0–2, 2–6 and 6–50 mm. The smallest fraction (\emptyset 0–2 mm) was further separated into a sand fraction (\emptyset 0.1–2 mm) and a sludge fraction (\emptyset 0–0.1 mm). The total amount of MSWI bottom ash generated from the incinerator is 100,000 t/year and the weight percentages of the sludge, sand and granulate fractions (\emptyset 2–6 and 6–50 mm) of this amount were 10%, 33%, 11% and 24%, respectively. The SF consisted mostly of hard granular materials along with some fibres.

The third waste material is a residue from a fluidised bed incinerator that treats both sludge (capacity 190,000 t/year) and high calorific waste (capacity 270,000 t/year). The incinerator is equipped with a steam boiler, an electrofilter; scrubbers and a baghouse filter. The boiler ash from the steam boiler and the fly ash from the electrofilter are collected together and used in this research (BFA).

2.2. Characterization of the materials

SiO₂, SO₄^{2–}, PO₄^{3–} and Cl[–] contents in the wastes were determined by standard gravimetric methods [33]. Other major and minor element concentrations in the samples were determined by inductively coupled plasma mass spectrometry (ICP-MS; Thermo Elemental, X Series). To prepare the solution for ICP-MS analysis, approximately 0.1 g of sample was taken in a Teflon beaker and digested successively by 5 ml of concentrated HNO₃, HClO₄ and HF acids on a hot plate. The final volume of the solution obtained after dissolution of the residue in 10 ml of concentrated HNO₃ was made up to 100 ml with deionised water. For analysis by ICP-MS, the solutions were again diluted 50 times. From the concentration obtained (µg/l), the weight percentage of the most abundant oxides of the elements determined in the wastes was deduced. A previously reported method was used to determine the amount of water-soluble material present in the wastes [34].

X-ray diffraction patterns of the wastes were recorded to identify the main crystalline components. Measurements were done with a Philips PW 1830 diffractometer, equipped with a graphite monochromator and a gas proportional detector, using Cu K α radiation. Scans were recorded from 5° to 70° 2 θ with a step size of 0.02° and a counting time of 5 s. During the measurements, the back filled sample holders were spun to obtain a reliable data. Mineral matter was identified using the Diffract Plus software employing the ICDP PDF-2 database. The grain size distributions of the materials were determined according to the sieve analysis method for fine aggregates (C132, ASTM).

2.3. Reactivities of the materials (with cement)

2.3.1. Reactivity with lime

To determine the reactivities of the wastes with lime, an accelerated method was used [35]: approximately 0.3 g of the material was accurately weighed and mixed with 50 ml of 0.01 M CaO solution and reacted in a glove bag at room temperature under a N₂ atmosphere for 1, 3 and 7 days. The remaining Ca²⁺ content in the filtrate of the reacted CaO solution was determined by EDTA titration [33] and lime reactivity was expressed in percent of lime consumed by the materials. Metakaolin, a natural calcined pozzolana (NCP), was used to compare the lime reactivities of the wastes. Metakaolin was chosen as a reference due to its high pozzolanic activity, which can be compared with silica fume [8,36].

2.3.2. Hydration study

Mixtures containing 10% powdered wastes and 90% ordinary Portland cement (CEM I 52.5 N) were prepared to study the hydration behaviour of the waste with cement. The mixtures were prepared in an electrical mixer to ensure the homogeneity. Cement pastes were prepared from these mixtures by adding distilled water in a water-to-solid ratio of 0.5:1 (ml/g) and manually mixing for about 5 min in a plastic container. The airtight containers of the paste were kept at ambient temperature in a desiccator to avoid carbonation and allowed to hydrate up to 28 days. Portions of the hydrated pastes were taken from the container after 1, 3, 7 and 28 days and dried in two steps to stop the hydration of the cementitious mixtures: the samples were vacuum dried for 5 h in a vacuum oven at 50 °C, powdered and again vacuum dried for another 6 h. However, slight destruction of some of the hydrates by vacuum drying cannot be excluded. The final hydration products were stored in airtight containers and in a desiccator to avoid carbonation. The products were then characterized by XRD, TG/DSC and SEM techniques.

The thermal curves (thermo-gravimetry/differential scanning calorimetry) of the products were taken in a simultaneous thermo-gravimetric and scanning calorimetric apparatus (Netzsch, STA 409 PC). A total of 30–40 mg of sample was taken in an alumina crucible and heated in a nitrogen atmosphere up to 1000 °C, maintaining 10 °C/min. heating rate and using α -Al₂O₃ as the reference material. The XRD patterns of the hydration

products were recorded using Cu K α radiation (Miniflex, MJ 14848 B01, Rigaku, Japan). The scanning electron micrographs of some of the hydrated pastes were recorded by using a SEM apparatus (Philips XL30 FEG-EDXA). EDXA measurements of some selected samples were also done using the same instrument.

2.3.3. Potential alkali-silica reactivity and the effect of organic fraction

Potential alkali-silica reactivity was evaluated according to the ASTM standard test method for fine aggregates, C289-94. In this experiment, the granulometric fraction ranging from 0 to $300 \,\mu\text{m}$ was used instead of the granulometric fraction ranging from 150 to $300 \,\mu\text{m}$ as specified in ASTM C 289-94 method. The method consists of attacking the aggregate with a standard 1N NaOH solution at about $80\,^\circ\text{C}$ for 24 h, measuring the concentration of dissolved silica and alkalinity by using a colorimetric and an acid–base titration method. The plot of the amount of dissolved silica against the total reduction in alkalinity according to the ASTM C289-94 method indicates the nature of fine aggregate (innocuous or deleterious) to use in cement mortar preparation.

The effect of organic impurities of the wastes during the application as fine aggregate in cement mortar and concrete was evaluated by the ASTM C 40-92 method. In this method, about 450 g of waste is treated with 3% NaOH solution for 24 h and then the colour of the supernatant liquid is compared with the colour of a freshly prepared reference standard colour solution (0.25 g K₂Cr₂O₇ in 100 ml concentrated H₂SO₄). If the colour of the supernatant liquid is darker than that of the reference standard colour solution, the fine aggregate under test is considered to possibly contain deleterious organic impurities.

2.3.4. Compressive strength

The compressive strength of cement mortar cubes containing waste as a partial substitution of fine aggregates were determined by a compressive strength determination machine with 105 kN loading capacity. Mortar samples were prepared in a Hobart planetary mixer using a 1:2 ratio of cement (CEM I 52.5 N) and fine aggregate, a mixture of 25% SLG, SF and BFA, respectively, with 75% natural fine aggregates and a control, 100% natural fine aggregate. Consistencies of control and waste containing mortar mixtures were kept constant instead of a constant W/C value, as it was not possible to get a workable BFA and SF containing mortar composition at a W/C value of 0.5. In this condition, the W/C values of control, SLG, SF and BFA containing mortar compositions were 0.50, 0.48, 0.58 and 0.60, respectively. The mortars were cast into moulds $(4 \text{ cm} \times 4 \text{ cm} \times 16 \text{ cm})$ and vibrated to reduce the volume of entrapped air. The mortar bars were demoulded after 24 h and cured in a humidity chamber (>95% room humidity) until the determination of 3, 7 and 28 days compressive strength. Before determination of compressive strength, mortar bars were cut into $4 \text{ cm} \times 4 \text{ cm} \times 4 \text{ cm}$ mortar cubes. The average values of the compressive strength determined from three specimens were reported.

2.4. Leaching behaviour of wastes and cement mortars

The leaching behaviour of the wastes was evaluated by using European standard test, EN 12457-2. Powdered waste materials were agitated with distilled deionised water for 24 h maintaining a 10:1 liquid-to-solid ratio. The leachate (about 100 ml) was filtered through a 0.45 μ m membrane filter and separated into two equal volume fractions. One fraction was acidified with 1 ml of 65% HNO₃ acid and kept for cation analysis by ICP-MS. The other fraction was directly used for analysis of oxyanions of As, Sb, Se, Mo and V by ICP-MS.

The leachability of elements from the cement mortars was evaluated by the European standard test EN 12457-2 test as well as by the Dutch diffusion-leaching test, NEN 7345. The EN 12457-2 test was done on powdered cement mortar samples according to the procedure described in the previous paragraph. For the NEN 7345 test, $4.0 \text{ cm} \times 4.0 \text{ cm} \times 4.0 \text{ cm}$ mortar cubes cut from mortar bars after hydration for 28 days were immersed in distilled water (liquid to monolith volume = 5) adjusted to pH4.0 with HNO₃, which was intermittently renewed after 6, 24, 54, 96, 216, 384, 864 and 1536 h. After determination of the conductivity and the pH, a portion of the leachate obtained after each renewal was collected for the determination of the oxyanions of As, Sb, Se, Mo and V content. The remaining portion of the leachate was acidified to pH < 2 and used for the determination of the cation content. For all leaching tests of cement containing samples, control specimens (cement mortar without wastes) were also tested.

3. Results and discussion

3.1. Chemical, mineralogical and physical properties of the wastes

The chemical composition of the wastes is shown in Table 1. All the materials contain high amounts of common constituents of ordinary Portland cement (OPC) like Al₂O₃, CaO, Fe₂O₃, SiO_2 and SO_4^{2-} along with considerable amounts of alkali oxides (Na₂O + K_2 O), MgO and PO₄³⁻. SLG also contains high amount of CuO, MnO₂, PbO, SnO₂ and ZnO. All the wastes contain high amounts of other toxic elements. The toxic element concentrations in SLG are, as expected, higher than those in the other two types of wastes. SF and BFA comprise of considerable amounts of soluble material (3.45% and 4.09%, respectively). The high loss on ignition (LOI) value of SF indicates the presence of high amounts of carbonaceous materials as well as some inorganic salts like calcite, chloride, sulphates in SF. On the other hand, the increase in weight during the heating of SLG indicates the oxidation of some compounds present in these wastes. The presence of high amount of SiO₂ in BFA (about 61%) compared to SF (about 42%) is due to the use of silica sand as a fluidised material in the furnace during combustion of wastes.

The XRD patterns of the wastes are shown in Fig. 1. The minerals identified from the XRD patterns are presented in Table 2.

SLG contains minerals like akermanite, hardystonite, kirschsteinite from the melilite and olivine groups along with sphalerite and wuestite. Some other minerals like anorthite,

Table 1		
Chemical	compositions of was	ste materials

	Amounts (% on dry basis) in			
	SLG	SF	BFA	
Oxides				
Al ₂ O ₃	5.41	9.57	8.25	
BaO	0.841	na	na	
CaO	18.9	16.4	10.0	
CuO	0.421	0.445	0.393	
Fe ₂ O ₃	33.95	13.5	4.99	
K ₂ O	0.308	1.01	0.958	
MgO	1.41	2.23	1.34	
MnO ₂	0.656	na	na	
Na ₂ O	0.872	2.08	1.36	
PbO	2.47	0.379	0.121	
SiO ₂	25.7	41.3	60.6	
SnO ₂	0.503	na	na	
ZnO	5.01	0.735	0.373	
Cl-	nd	1.50	1.79	
PO_4^{3-}	1.19	2.57	3.36	
SO_4^{2-}	1.17	1.24	4.92	
Others	1.19	7.08	1.56	
	(As, Sb)	$({\rm H_2O}^*,{\rm CO_3}^{2-},{\rm C})$	(CO_3^{2-}, C)	
LOD	2.50	10.2	0.82	
LOI (% on dry basis)	(+)	12.1	0.89	
	3.95			
Elements (mg/kg)				
As	4200	na	312	
Cd	5.81	14.06	30.15	
Cr	1001	292	67.6	
Мо	279	29.5	31.0	
Ni	416	217	129	
Sb	3077	na	1208	
Se	292	na	26.4	
V	805	341	252	
Soluble matter	_	3.45	4.09	
(% on wet basis)				

LOI: loss on ignition; LOD: loss on drying; *: combined water in hydrated phases; na: not analysed; nd: not detected.

belite (larnite), quartz and possibly sjogrenite may also be present in minor amounts. While determining the LOI for SLG, the mass increased due to the oxidation of wuestite (FeO) to Fe_2O_3 . The mineral composition of the SLG sample is similar to that of the same type of slag reported by other authors [18,37,38]. In contrast to Zn-containing minerals, Pb-containing minerals are not detected by XRD analysis, probably due to the difference in behaviour of these two elements during formation of slag. Unlike Zn, Pb does not dissolve in the silicate melt during slag formation and therefore it does not crystallize as a separate silicate minerals like Zn. Pb may be present in different forms [38]: as a metallic inclusion trapped in the amorphous glass, in crystalline forms as a pure metal or in intermetallic state or as a sulphide compounds, of which the amounts of these minerals are possibly below the detection limit of XRD.

Major minerals present in SF include calcite and quartz along with gehlenite and magnetite. The SF also contains anhydrite, anorthite, belite (larnite), halite and wuestite with some other possible metal/minerals like elemental aluminium, anorthoclase, ettringite and hematite. The mineralogical composition of the



Fig. 1. XRD patterns of SLG, SF and BFA (Al: aluminium, Am: almandine, Ah: anhydrite, An: anorthite, Ao: anorthoclase, C: calcite, Co: corundum, Q: quartz, H: hematite, G: gehlenite, Gy: gypsum, Ap: apatite, La: larnite, Ha: halite, M: magnetite, W: wuestite, E: ettringite, Hd: hardystonite/akermanite, K: kirschsteinite, S: sphalerite).

sand-sized fraction of MSWI bottom ash is comparable to the ones of different size fractions of MSWI ash reported in literature [18,39,40].

The major mineral found in BFA is quartz along with anhydrite, anorthite and hematite. BFA also contains considerable amounts of aluminium, belite (larnite), calcite, fluorappatite and gehlenite. Almandine, corundum and gypsum are possibly present in BFA. The peak intensities of Al in the XRD pat-

Table 2	
Minerals identified by XRD in the wastes	

Mineral name		SLG	SF	BFA
Almandine	(Fe,Mn) ₃ Al ₂ (SiO ₄) ₃			(X)
Aluminium	Al		(X)	Х
Anhydrite	CaSO ₄		Х	XX
Anorthite	CaAl ₂ Si ₂ O ₈	(X)	Х	XX
Anorthoclase	(Na,K)AlSi ₃ O ₈		(X)	
Larnite (belite)	Beta-Ca ₂ SiO ₄	(X)	Х	Х
Calcite	CaCO ₃		XXX	Х
Corundum	Al ₂ O ₃			(X)
Ettringite	$Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26H_2O$		(X)	
Fluorapatite	Ca(PO ₄) ₃ (F,Cl)			Х
Gehlenite	Ca ₂ Al ₂ SiO ₇	(X)	XX	Х
Gypsum	CaSO ₄ ·2H ₂ O			(X)
Halite	NaCl		(X)	
Hardystonite– akermanite	$Ca_2(Mg,Zn)Si_2O_7$	XXX		
Hematite	Fe ₂ O ₃		(X)	XX
Kirschsteinite	CaFeSiO ₄	XXX		
Magnetite	Fe ₃ O ₄		XX	
Quartz	SiO ₂	(X)	XXXX	XXXX
Sjogrenite	Mg ₆ Fe ₂ CO ₃ (OH) ₁₆ ·4H ₂ O	(X)		
Sphalerite	$(Zn_r, Cd_{1-r})S$	XX		
Vuestite	FeO	Х	Х	

XXXX: highly abundant; XXX: abundant; XX: moderately abundant; X: low abundance and (X): possible.

tern of BFA are considerably higher than the same present in the XRD pattern in SF. The violent turbulence and the short retention time of the waste in the fluidised bed incinerator possibly inhibit the oxidation of aluminium present in the waste [41]. The mineralogical composition of BFA is rather similar to the one of the sand-sized fraction of MSWI bottom ash (SF) and of other types of MSWI ashes as reported by other authors [18,39,40,42].

The grain size distributions of the materials as determined by ASTM sieve analysis for fine aggregates are shown in Fig. 2. The amount of coarser particles in SLG is higher than the ASTM specified amount. For SLG, only 20% of the particles is less than 0.63 mm size. BFA contains the largest amount of fine particles (about 75% less than 0.63 mm) followed by SF (about 60% less than 0.63 mm).



Fig. 2. Sieve analysis of waste materials (ASTM high and ASTM low: high and low boundaries as defined by ASTM C33 method for fine aggregates).



Fig. 3. Lime reactivities of wastes along with natural calcined pozzolana (NCP).

3.2. Reactivities of the wastes with lime and cement

The XRD patterns in Fig. 1 indicate the presence of hydraulic minerals like belite and gehlenite in the wastes. Belite is an important mineral phase of Portland cement clinker. Gehlenite reacts with lime and forms various products like calcium silicate hydrate, and calcium alumino silicate hydrate [43]. The alkali metal oxides, which have high concentrations in the wastes compared to the cement may stimulate the formation of expansive products due to alkali-silica reactions during cement hydration [44]. Metallic aluminium found in BFA and SF may generate hydrogen gas during application as an aggregate in concrete [44]. Anhydrite and gypsum in SF and BFA may form ettringite at a later stage of hydration due to the delayed ettringite formation [44]. ZnO and PbO, present in SLG, may form some hydrated compounds, which may retard the hydration reaction [45–47]. Therefore the reactivity and the hydration behaviour of powdered waste-cement mixtures are evaluated in order to understand the possible effect of these wastes when applied as a fine aggregate.

Fig. 3 shows the lime reactivities of the wastes along with one of natural calcined pozzolana (NCP). Compared to NCP, all the materials exhibit poor lime reactivities. It appears that about 25% of the Ca^{2+} ion is consumed by the waste after 7 days. The poor reactivities of BFA and SF are due to the presence of high amounts of quartz and calcite, which are nonhydraulic in nature. The poor lime reactivity of SLG indicates that the majority of the minerals present in SLG are also hydraulically inert in nature.

Fig. 4 shows the differential scanning calorimetric (DSC) patterns of various hydrated cement pastes obtained from mixtures of ordinary Portland cement (OPC) with or without wastes after 1, 7 and 28 days of hydration. The endotherms observed at different temperature regions are due to dehydration of various common hydration products: calcium silicate hydrate gel (CaO–SiO₂–H₂O or C–S–H gel) and ettringite (3CaO·Al₂O₃·3CaSO₄·32H₂O) at 100–150 °C, monosulphoaluminate (3CaO·Al₂O₃·CaSO₄·12H₂O) at 150–220 °C and calcium hydroxide (CH) at 420–550 °C.

All the pastes show the formation of CSH, ettringite and CH after 1-day hydration. On prolonging the hydration time to 7 and 28 days, a new endothermic peak appears in the region of 150-220 °C due to the formation of monosulphoaluminate (an



Fig. 4. Differential scanning calorimetric patterns of different hydrated pastes (1, OPC; 2, SF; 3, SLG and 4, BFA; AFm, monosulphoaluminate).

AFm phase). The intensity of the endothermic minimum due to CH observed in the waste containing cement pastes in the 420-550 °C region is slightly lower than the one observed in OPC paste. This is partly due to the low content of cement in the waste-cement pastes and partly due to the low reactivities of the wastes consuming small amounts of CH during hydration. The 28 days hydrated paste of BFA containing cement paste shows a weak endothermic minimum at about 265 °C, which can be related to the formation of Al(OH)₃, due to the presence of metallic aluminium in BFA. The SEM results of the 28 days hydrated sample of BFA-cement pastes (Fig. 5) show the formation of needle shaped crystalline and some amorphous products along cracks. The EDXA analyses of some selected portions of 28 days hydrated cement paste containing BFA (figure not shown here) indicate the presence of ettringite, calcium aluminate, aluminium hydroxide as well as K and Mg containing complex calcium alumino-silicates. Visual observation of the BFA containing hydrated paste obtained after 7 d shows



Fig. 5. Scanning electron micrographs of 28 days hydrated pastes of cement–BFA pastes (white arrows in the figures show the cracks and black arrows indicate the EDXA analysis region).

cracks and spalling on the surface. Therefore it is suggested that the metallic aluminium contained in the BFA reacts with alkalis during hydration of cement and forms products like aluminium hydroxide and H₂ gas [14,42,44]. The large amount of sulphate present in BFA also promotes the formation of ettringite (secondary ettringite formation) due to the reaction of sulphate with the product of reaction of aluminium with alkalis [44,48]. The reaction of metallic aluminium with cement can be expressed by the following chemical equations, where at high pH, metallic aluminium forms hydrogen gas, aluminium hydroxide and aluminate [44]:

 $\mathrm{Al}\,+\,2\mathrm{OH}^-\,+\,\mathrm{H}_2\mathrm{O}\,\rightarrow\,\left[\mathrm{AlO}(\mathrm{OH})_2\right]^-\,+\,\mathrm{H}_2\uparrow\,\,p\mathrm{H}\,>\,7$

 $[AlO(OH)_2]^- + H_2O \rightarrow Al(OH)_3 + OH^- pH = 9-10$

$$Al(OH)_3 + OH^- \rightarrow [Al(OH)_4]^- pH > 10$$

At high pH, $[Al(OH)_4]^-$ may combine with SO_4^{2-} and Ca^{2+} to form ettringite. Formation of H₂ gas, Al(OH)₃ and ettringite exert expansive forces on the monolithic structure, which ultimately can create cracks during hydration. Reducing the Al content would therefore reduce these problems in the case of BFA. Similar reactions are also possible in SF as the XRD pat-



Fig. 6. XRD patterns of hydrated pastes obtained after 1 and 28 days hydration (1, alite + belite; 2, CH; 3, $CaCO_3 + CSH$; 4, CSH).

tern of SF also shows weak peaks of Al along with the intense peaks of anhydrite.

The XRD patterns of the hydrated pastes obtained after 1 and 28 days of hydration are shown in Fig. 6. All the figures are diffuse in nature due to presence of a high amount of amorphous materials. All the hydrated pastes show peaks at $2\theta = 18.1^{\circ}$, 34.14°, and 47.32°, due to the presence of CH. The intensities of the peaks due to CH in SF and BFA containing pastes are slightly lower than those for the hydrated OPC and SLG containing pastes after 1 day of hydration. This indicates that SF and BFA consume a small amount of CH during hydration of cement. The hydrated products obtained from all cement pastes also exhibit a peak at $2\theta = 29.5^{\circ}$, either due to calcite or CSH. The approximately same intensities of the peaks of CH, alite and belite $(2\theta = 32.4^\circ, 32.6^\circ \text{ and } 34.3^\circ)$ in the XRD pattern of SLG containing hydrated pastes with the peaks present in the XRD patterns of hydrated OPC paste obtained after 1 day of hydration indicate that ZnO and PbO in the SLG do not affect the cement hydration rate. Morrison et al. [20] report that the hydration of OPC and Zn-slag containing cement paste occurs to the same extent but in Zn-slag the onset of the hydration is retarded, due to the formation of a gelatinous layer of metal ions such as Pb and Zn around the cement grains. When the water penetrates to the cement grain, the layer is completely disrupted and hydration can take place unhindered [20]. Possibly this type of layer formation and disruption occurs in the SLG containing cement pastes within the first few hours of hydration, which is then not observed in the XRD patterns of 1 day hydrated SLG containing paste.

Fig. 7 shows the nature of the alkali-silica reactivities of three wastes determined according to ASTM C 289–94 test method. From the figure, it can be concluded that the alkali-silica reaction will possibly not occur during application of these materials as fine aggregates. The soluble silica contents



Fig. 7. Dissolved silica vs. reduction in alkalinity curve according to ASTM C 289 test (solid line shows the division between innocuous and deleterious aggregate; A, aggregates considered innocuous and B, aggregates considered deleterious).

in the NaOH extracts as specified by ASTM method are lower than 15 mmol/l although BFA and SF consumed high amount of alkalis (>425 mmol/l). The consumption of alkalis by BFA and SF is possibly due to the presence of aluminium in these materials.

Both BFA and SF contain small amounts of organic materials, which may have effect during application of these materials as fine aggregates in cement mortar. Arickx et al. [49] report that the organic matter present in SF mainly consists of fulvic and humic acids along with a hydrophilic portion. BFA also contains a hydrophilic portion, which exhibited FTIR bands mainly due to aliphatic compounds (figure not shown here). The colour of the supernatant obtained from SF after treatment with NaOH solution according to the ASTM C 40-92 method is darker than that of the reference standard colour solution, which indicates that SF contains deleterious organic impurities if SF is used as a fine aggregate in cement mortar or concrete. On the other hand, organic matter present in BFA shows a very light colour indicating the innocuous nature of this material during application as a fine aggregate in concrete.

The compressive strengths of the mortar cubes with different cementitious compositions at different time periods up to 28 days are shown in Fig. 8. The BFA and SF containing mortar cubes show a much lower compressive strengths at all ages, and cannot be used as a fine aggregate, which is attributed to the presence of metallic aluminium and high sulphate contents in these materials. Visual observation also shows the craters and spalling on the surface of the BFA and SF containing mortars, which indicates the effect of metallic aluminium during hydration. The BFA and SF containing mortar bars also showed swelling during the first few hours of curing, indicating the formation of hydrogen gas [50]. Presence of harmful organic impurities in SF may also affect the compressive strength of SF containing mortars. The presence of metallic aluminium in BFA and SF thus needs to be addressed before considering these materials as a granular additive in the preparation of concrete. Treatment of MSWI bottom ash by NaOH solution considerably improves the properties of bottom ash by converting metallic



Fig. 8. Compressive strengths of the mortar cubes at different time intervals (Cont: mortar cubes with natural aggregates; SLG, BFA and SF: mortars prepared by 25% SLG, BFA and SF containing natural aggregate, respectively).

aluminium into aluminate [14]. Wet grinding of MSWI bottom ash for a few days also converts metallic aluminium into other forms [29]. On the other hand, the mortar cubes containing natural fine aggregates with 25% SLG exhibit approximately the same strengths as natural fine aggregates containing mortars at all ages. This suggests the possibility of utilizing SLG as a fine aggregate in the cement mortar and concrete. However, the sample preparation and experimental set-up can have an impact on results and these results are therefore only valid for the sample preparation and experimental set-up as described in the paper.

3.3. Leaching behaviour of waste and cement mortars

Leaching tests of the wastes and the cement mortars with and without containing wastes are conducted to evaluate the environmental suitability of these materials for application as constituents in cement mortar and concrete.

Table 3 shows the concentrations of selected elements in the leachates obtained from three wastes after application of the European standard EN 12457-2 test along with the Flemish NEN 7343 leaching limit values for recycling as a granular construction material (German leaching values for Se and V). Although the NEN 7343 column test and the EN 12457-2 extraction test have different operating parameters, liquid to solid ratio in both tests is 10, thus making it possible to compare the results on a qualitative basis. The German DIN 38414-54 extraction test is same as the European 12457-2 test. The concentrations of Pb, Se and Sb in the leachate of SLG, of As, Cd, Cr, Mo and Se in the leachate of BFA and of Cd, Cr, Cu, Mo and Sb in the leachate of SF are higher than the specified limit values of those elements. Leaching of the majority of the toxic elements from SLG yields comparatively lower amounts than leaching from BFA and SF, although the concentrations of these elements in SLG are much higher than in the other two wastes. This indicates that the mineralogical composition influences the leaching of elements from these wastes. Compared to Zn, the amount of Pb leached from SLG is very high although the Zn concentration in the SLG is almost

Table 3 Elements leached from the powdered wastes obtained according to EN 12457/2 test

Table 4

Table 5

Total concentration and the concentration of elements in the leachates of powdered cement mortars obtained according to EN 12457/2 test

Elements	Limit value ^a	Leached amounts (mg/kg) from			
		SLG	BFA	SF	
Al		7.0	6.9	743	
Ca		102	3334	3242	
Fe		0.39	1.8	0.32	
K		17.7	2689	986	
Mg		4.8	4.7	2.1	
Na		29.1	2767	2936	
As	0.80	0.11	16.4	na	
Cd	0.03	0.02	0.17	0.07	
Cr	0.50	0.43	1.8	18.5	
Cu	0.50	0.44	0.14	16.10	
Мо	1.5	1.0	2.6	1.8	
Ni	0.75	0.08	0.35	0.37	
Pb	1.3	49.1	0.26	0.32	
Sb	2.0	2.5	0.60	3.4	
Se	0.10	3.5	0.31	na	
V	32	0.33	0.12	0.15	
Zn	2.8	1.7	0.83	0.39	
рН	7–12	10.6	10.9	10.4	

na: not analysed. Italicized data indicate the limit value for recycling of waste as construction material in Germany according to DIN 38414-54 test (Z_2 category). Data that exceed the limit values are in bold.

^a Limit values for recycling of waste as construction material in Flanders region, Belgium according to the Flemish column test NEN 7343.

two times higher than the Pb concentration in SLG. The XRD pattern of SLG shows the presence of considerable amounts of Zn-containing crystalline silicate mineral (hardystonite) and sulphide mineral (sphalerite), which are very stable under the applied leaching condition. On the other hand, Pb may be partly present in metallic or intermetallic state, which may be dissolved during leaching. Barna et al. also reported a similar observation during leaching of Zn and Pb from a Pb-slag [51].

Table 4 shows the total content of the elements in the cement mortars and the amount leached from the powdered mortars subjected to the EN 12457-2 test. From a comparison of the element concentrations in the leachate as presented in Tables 3 and 4, it can be concluded that the leaching of As, Cd, Cr, Mo, Ni, Sb, Se and V from the wastes present in mortar is lower than from the untreated wastes. The Cu leaching from SF is improved when the SF is used in mortar preparation. On the other hand, leaching of Pb and Zn from the waste containing mortars and Cu leaching from SLG and BFA containing mortars are in general higher than from the waste. The concentrations of Cu, Pb and Zn in the leachate of SLG containing mortar and the concentrations of Cu and Pb in the leachate of SF containing mortar exceed the specified limit values of those elements. The lower leaching of the elements from the mortar are due to various chemical processes that generally occur in cement hydration like precipitations of calcium metallate (As, Mo, V) and metal hydroxides (Cd, Ni), as well as incorporation in hydration products (As, Cr, Sb, V). The high concentrations of Cu, Pb and Zn in mortar leachates can be attributed to the dissolution of hydroxides of these amphoteric metals at high pH (>12).

Element	Concentrations of element (mg/kg) in the mortar (<i>A</i>) and mortar leachates (<i>B</i>) of							
	Control		SLG		BFA		SF	
	Ā	В	Ā	В	A	В	Ā	В
Al	15387	3.0	14307	24.1	16783	3.5	16066	7.9
Ca	112406	6638	146406	7780	133225	6581	139830	6385
Fe	13108	0.16	44475	196	14486	7.3	20623	1.8
Mg	1940	0.19	2466	4.4	2434	0.48	2705	0.07
K	3554	1395	3937	1091	5217	1418	4827	1476
Na	3001	1568	3360	1069	5975	1588	7336	2101
As	4.1	nd	96.2	nd	1.6	nd	0.26	nd
Ва	423	36.2	1442	60.0	679	55.6	625	57.3
Cd	0.38	nd	0.58	nd	2.2	nd	0.65	nd
Cr	16.7	0.37	108	0.34	24.3	0.24	35.9	0.16
Cu	39.2	0.04	528	2.92	469	0.40	356	0.56
Mo	5.4	0.10	26.3	0.25	3.7	0.24	6.6	0.21
Ni	30.3	0.45	84.4	0.08	35.6	0.02	45.3	0.03
Sb	7.52	0.002	111	0.38	25.3	0.05	18.3	0.01
Se	3.2	0.02	3.9	0.06	2.2	nd	1.4	nd
Pb	38.4	nd	2935	53.3	219	0.81	1150	1.47
V	98.7	nd	70.0	nd	73.9	nd	77.5	nd
Zn	569	0.11	6191	7.27	877	0.98	1182	0.29
pН		12.3		12.6		12.5		12.5

nd: not detected; data that exceed the limit values (indicated in Table 3) are in bold.

The NEN 7345 leaching test provides the long term leaching information from waste recycled in monolithic preparations of e.g. cement mortar. Table 5 shows the total leached amount of some elements after 64-day immersion in leachate according to the NEN 7345 test along with the specified limit for minor elements. The concentrations of most of the minor elements leached from waste containing mortars are higher than those leached from control mortar. However, the cumulative concentrations of all of these elements in the leachates are lower than the limit values (Table 5). Therefore there is no environmental

Cumulative leaching (mg/m^2) amount of elements from the cement mortars after 64-day immersion in leachate obtained according to NEN7345 leaching test

Element	Limit value ^a	Control	SLG	BFA	SF
Al		327	442	1114	694
Ca		16162	16835	10899	11601
Fe		19.8	21.7	31.8	27.4
Κ		13380	16587	18708	17185
Mg		5.27	4.02	6.55	4.81
Na		4909	6276	11070	11747
As	27	nd	nd	nd	nd
Ba		16.9	21.1	7.4	7.3
Cd	1.1	nd	nd	nd	nd
Cr	55	3.3	2.4	11.8	3.1
Cu	25	0.27	0.60	1.1	2.4
Мо		0.68	0.71	5.1	1.2
Pb	60	0.06	9.3	0.30	0.67
V		6.7	5.1	5.6	7.2
Zn	90	0.58	0.79	1.2	2.3

^a Limit values (mg/m²) for recycling of solid waste in monolithic construction materials in Flanders region, Belgium.



Fig. 9. (A) pH and (B) conductivity (μ S/cm) of the leachates generated from cement mortars according to NEN 7345 test.

restriction to use these materials in the investigated mixes of cement mortars and concrete. The leaching of the majority of the elements from BFA and SF containing mortars is higher than the leaching from control and SLG containing mortars, probably due to the difference in microstructure of the mortars. In the NEN 7345 test, the release of elements mainly occurs due to diffusional transport of ions through the liquid in the monolith, which is dependent on the microstructure of the monolith. The application of different waste materials in cement mortar and concrete production generally changes their microstructure due to the difference in chemical reactivity, which also affects the leaching behaviour of an element from the mortar and concrete. The presence of metallic aluminium and high amounts of sulphate compounds in BFA and SF as observed by XRD analysis may generate cracks and macropores in the mortars (see also Fig. 5 for BFA) [44,52,53], which may accelerate the percolation of leachates through the mortars and therefore accelerate the leaching of elements from the mortars. Ba and Pb leaching from SLG containing mortars are higher than from the other types of mortar due to the presence of higher amounts of Ba and Pb in the SLG (Table 1). The leaching amount of Na, K from the mortars are very high although the total contents of these elements are lower than of the other major elements like Al, Ca, Fe (Table 4), which suggests that major portions of K and Na are weakly bound in the cement matrix. The dissolution of hydroxides and hydrates in acidic medium increases the pH (>12) and conductivities of the leachates (Fig. 9). Higher pH and conductivities of the leachates generated from waste containing mortars compared to leachates generated from control indicate the presence of high amounts of ionic species in these leachates. Further increase in conductivities of the

leachates generated from BFA and SF containing mortar are due to dissolution of soluble salts like chlorides from BFA and SF.

4. Conclusions

A comparative technical and environmental evaluation of three different wastes generated from three different processes is carried out for use of these materials as a fine aggregate in preparations of cement mortar and concrete. Results can be summarized as follows:

- (1) Slag generated from Pb-smelting industry (SLG) can be used as a partial (25%) substitution of fine aggregate in the preparation of cement mortar and concrete. SLG hydration with cement does not produce any product that is harmful to the properties of cement mortar. The presence of high amounts of ZnO and PbO in SLG does not affect the rate of cement hydration. The compressive strength development of mortars containing 25% SLG and 75% natural fine aggregates is similar to the mortar cubes containing 100% natural fine aggregates.
- (2) Ashes generated from municipal waste incineration (BFA and SF) cannot be used as such as fine aggregates in cement mortar and concrete preparations as the 28-day compressive strengths of the mortar containing these two wastes are very low compared to control mortar. The presence of deleterious components such as metallic Al, high amounts of sulphate and soluble salts, e.g. chlorides in BFA and SF and organic matters in SF needs to be addressed before considering these materials as a granular additive in the preparation of concrete. The presence of metallic aluminium in these wastes not only generates H₂ gas but also influences the formation of expansive products like ettringite, which drastically reduces the compressive strengths of the mortars.
- (3) The cement-based application of these wastes significantly improves the leaching behaviour of toxic elements and meets the leaching criteria although these materials alone do not meet the subjected leaching criteria. Mineralogical compositions of the wastes also influence the leaching of trace elements.
- (4) From the combined evaluation of technical and environmental properties, it can be concluded for the investigated waste materials that the removal of deleterious components from BFA and SF is the most important issue to solve in order to use these materials as a fine aggregate. The environmental performance of the materials meets the required standards.

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