



Potential use of municipal solid waste incineration bottom ash as supplementary cementitious materials based on particle size fraction

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Abstract Incinerator bottom ash (IBA) is a heterogeneous by-product mainly composed of mineral phases. Despite its variable composition, it is generally classified as a silica-rich secondary raw material, containing significant amounts of aluminium, calcium, and amorphous phases. These characteristics make IBA a potential candidate for use as

a supplementary cementitious material (SCM) in blended cement formulations. However, due to its heterogeneity, evaluating the behaviour of different particle size fractions is essential to ensure consistent performance. In this study, the pozzolanic activity of various IBA fractions was assessed using three standard reactivity tests: the modified Chapelle test, the modified R^3 test, and the strength activity index (SAI). The results varied depending on the test applied, but a general trend was observed in which coarser fractions exhibited greater pozzolanic activity than finer ones. The fraction larger than 8 mm consistently showed the best performance across all tests. Additionally, the environmental impact of each fraction was analysed through batch leaching tests to determine the potential release of metal(loid)s. Regarding environmental safety, leaching results confirmed that all particle size fractions met the requirements to be classified as non-hazardous at the end of their life cycle. These findings support the use of IBA, particularly its coarser fractions, as a sustainable and safe SCM in the production of blended cement.

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1 Introduction

The cement industry is a significant source of pollution in the construction sector, mainly due to the



production of clinker, the key component of cement, which is both carbon- and energy-intensive. Clinker manufacturing is responsible for approximately 5–8% of global CO₂ emissions and 7–15% of global industrial energy consumption, making it the industrial process with the highest CO₂ emissions [1–3]. Accordingly, to reduce the environmental impact of the cement industry, mitigation strategies focus on improving clinker production efficiency or decreasing clinker use. Key solutions include enhancing process efficiency, switching to alternative fuels and feedstocks, implementing carbon capture and storage, and substituting clinker with alternative cementitious materials [4].

A current and short-term solution to reducing the carbon footprint of cement is the production of blended cement, which involves partially replacing clinker with alternative materials [5]. Mineral additions are commonly used to lower the clinker-to-cement ratio in Portland cement (PC) or reduce PC content in concrete and mortars. Among them, supplementary cementitious materials (SCMs) have emerged as one of the most promising short- and medium-term strategies for decarbonising the cement industry [6]. SCMs are powdered mineral additions with varying compositions, mainly rich in SiO₂, a combination of SiO₂ and Al₂O₃, or SiO₂, Al₂O₃, and CaO [7, 8]. This approach enables the incorporation of secondary raw materials, such as by-products or industrial waste, to partially replace clinker, thus reducing the CO₂ emissions associated with cement production and contributing to circular economy.

Over the past few decades, incineration of municipal solid waste (MSW) has garnered increasing interest in many regions due to its dual benefits: energy recovery and reduced reliance on landfill disposal. The global number of waste-to-energy (WtE) plants has significantly increased, with approximately 30% of the total MSW generated worldwide currently being incinerated [9]. Incineration bottom ash (IBA) is a residue generated during the MSW incineration process in WtE plants, accounting for approximately 85% of the solid material generated from waste combustion [10]. In the European Union (EU), around 18 million tonnes of IBA are produced annually [11], although the valorisation rate of this material as a secondary source is unequal depending on the location and specific WtE plant. Investigating sustainable applications for IBA is therefore essential to reduce

landfill disposal and promote circular economy practices, repurposing this waste material for valuable secondary uses, particularly in the construction sector [12].

IBA is mainly constituted of a mineral fraction (80–85 wt.%), ferrous metals (mainly steel 12–10 wt.%), and non-ferrous metals (2–5 wt.%), with approximately two-thirds of the non-ferrous fraction consisting of aluminium [13]. Despite its high heterogeneity, IBA is regarded as a silica-rich secondary raw material, mainly due to the presence of primary and secondary glass [14], along with substantial amounts of calcium and aluminium [15]. It also includes other oxides, such as Na₂O and K₂O, along with trace amounts of heavy metal(loid)s, which are predominantly found in the finest fractions [16, 17]. This composition makes IBA a promising material for construction applications, with potential uses in various forms such as asphalt concrete [18], alkali-activated concretes [15, 19, 20], autoclaved aerated concrete [21] SCM [22–24], and the production of secondary lightweight aggregates [25–28].

Previous studies by Chimenos et al. [29] and del Valle et al. [30] showed that fresh IBA exhibited pozzolanic reactions during the weathering process, resulting in the formation of calcium silicates and aluminosilicates. These reactions contribute to the agglomeration and hardening of pavement layers when IBA is used as a road sub-base material. However, the same authors [14, 31] also showed that the particle size distribution of IBA significantly affects the composition of materials, particularly primary glass (cullet fragments from post-consumer containers) and secondary glass (vitreous phases formed in-furnace from melting and re-solidification processes), as well as natural and synthetic ceramics. This fact also influences its major mineralogical phases and the availability of silicon and aluminium for pozzolanic activity [32]. Accordingly, IBA, with its high silica and calcium silico(alumin)ates content, may be of significant interest as SCM for use in blended cement formulations, though its heterogeneity and the variation in material composition across particle size fractions must be considered.

In this regard, this work aims to assess the suitability of IBA as a SCM by exploring its individual particle size fractions. This study is presented as a case study based on material from a single waste-to-energy plant; however, the physicochemical characteristics of



the IBA and the operating conditions of the facility are representative of many other plants with similar technology, suggesting that the findings may be extrapolated, at least in tendency, to such systems. Given the substantial variations in materialography and chemical–mineralogical composition among the IBA fractions, differences in their pozzolanic behaviour are therefore to be expected. Building on this premise, the study examines the chemical and mineralogical characteristics of each fraction and evaluates their pozzolanic potential through different experimental methods, while also determining the mechanical and environmental performance of the binders produced with these fraction-specific materials.

2 Material and methods

2.1 Materials

Fresh IBA was initially collected from a WtE plant located in Mataró (Barcelona; NE Spain), where the feed stream mainly consists of household rubbish, with a minor contribution from commercial sources. The plant produces approximately 31 kT of fresh IBA annually, which undergoes a subsequent conditioning and valorisation process at the facilities of the Pedreres Maresme Rusc, S.L. company. This process includes the recovery of ferrous and non-ferrous metals, as well as the removal of lightweight materials. After conditioning, the IBA is stockpiled outdoors for at least two months to promote heavy metal immobilization through natural weathering. Around 150 kg of weathered IBA, obtained by combining 40–50 sub-samples collected from various stockpiles corresponding to different incineration periods and from different locations within each stockpile, was homogenised and stored in 30 L plastic containers.

The particle size distribution (PSD) of weathered IBA, dried overnight at 105 °C, was determined using a procedure described in standard EN 933–2:2020. The obtained PSD (Fig. 1) closely aligns with those documented by other researchers in other WtE plants employing grate furnace technology [10]. The main fraction of IBA consists of particles smaller than 10 mm, accounting for 72 wt.%. In terms of the other fractions, approximately 33 wt.% consists of particles smaller than 2 mm, while less than 34 wt.% comprises particles larger than 8 mm.

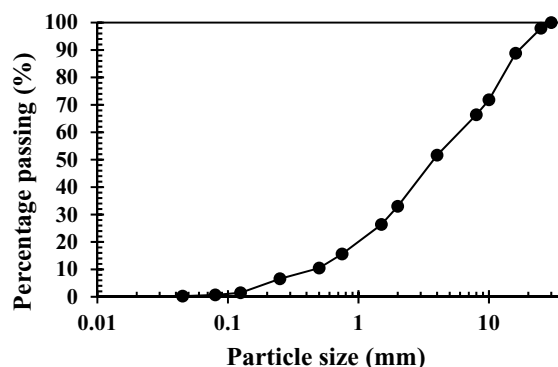


Fig. 1 Particle size distribution of weathered IBA

For consistency with previous studies by del Valle-Zermeño et al. [14], weathered IBA was sieved through mesh openings of 2, 4, 8, and 16 mm to obtain the same granulometric fractions. Based on the previously determined particle size distribution, approximately 32 wt.% of weathered IBA falls within the 0–2 mm fraction, 18 wt.% within the 2–4 mm fraction, 15 wt.% within the 4–8 mm fraction, 21 wt.% within the 8–16 mm fraction, and 14 wt.% within the largest fraction of > 16 mm.

To investigate the potential application of matured IBA as SCM, 250 g of each fraction was initially crushed in a jaw crusher and then milled until the entire sample reached a particle size smaller than 90 µm. Milling was conducted using a RETSCH PM 100 planetary ball mill, equipped with 500 ml zirconia vessels and 10 mm zirconia balls, operating at a rotational speed of 300 rpm for a maximum of 60 min, resulting in a homogeneous powder. This procedure was designed to prevent unintended mechanochemical activation of specific silico(alumin)ates phases present in certain particle size fractions, which can occur during high-energy milling processes.

2.2 Physicochemical characterisation of IBA

For each powdered size fraction of weathered IBA, dynamic light scattering was used to determine the particle size distribution, with measurements conducted using a laser diffraction particle-sizing analyser (LS 13 320, Beckman Coulter). The specific surface area (SSA) was determined using nitrogen gas as an inert medium, following the

Brunauer–Emmett–Teller (BET) method on a TriStar 3000 V6.04 instrument.

The chemical composition of the major and minor elements was determined through X-ray fluorescence spectroscopy (XRF) using a Bruker S2 PUMA Light Element Energy Dispersive X-ray Fluorescence Spectroscopy (ED-XRF).

The main crystalline phases were measured through X-ray diffraction (XRD) with a Bragg–Brentano PANalytical X'Pert PRO MPD alpha1 powder diffractometer. The CuK_α wavelength ($\lambda = 1.5418 \text{ \AA}$) was selected using a Ni filter. Samples were scanned in the 2θ range of 4.5° to 100° 2θ , with a step size of 0.026° , a 4° antiscatter slit, and a 0.04 rad Soller slit. Five repeated scans were conducted, each lasting 24 min. The amorphous content (W_{amorph}) was estimated through the degree of crystallinity (DOC) function, as described in Eqs. (1) and (2), by fitting the background area assigned to amorphous phases and the peak area assigned to crystalline phases, yielding a semiquantitative estimate of W_{amorph}

$$\text{DOC} = \frac{\text{Crystalline Area}}{\text{Crystalline Area} + \text{Amorphous Area}} \quad (1)$$

$$W_{\text{amorph}} = (1 - \text{DOC}) \cdot 100 \quad (2)$$

2.3 Pozzolanic activity measurements

The pozzolanic activity of each powdered particle size fraction of weathered IBA was conducted through three reliable reactivity tests: (i) the modified Chapelle test, (ii) the modified R^3 test, and (iii) the strength activity index (SAI) method. Measuring the pozzolanic activity enables evaluating the chemical reactivity of each powdered fraction to form compounds with cement-like properties.

The modified Chapelle test aims to quantify the capability of the aluminosilicate sources to react with Ca units [33, 34]. In the test, an aqueous slurry was prepared by combining 1 g of each powdered fraction of weathered IBA, 2 g of CaO, and 250 mL of deionised water in a spherical flask at 90°C . The slurry was then stirred continuously for 16 h at a constant temperature, with a reflux condenser in place to prevent evaporation. Subsequently, 250 mL of a 0.7 M sucrose solution was added to the slurry and mixed for 15 min. The liquid fraction of the slurry was then

filtered and titrated with HCl 0.1 M. The amount of Ca(OH)_2 consumed per gram of each powdered fraction was determined through Eq. (3),

$$\frac{\text{mgCa(OH)}_2}{\text{g powdered IBA}} = 2 \cdot \frac{V_1 - V_2}{V_1} \cdot \frac{74}{56} \cdot 1000 \quad (3)$$

where V_1 represents the volume of HCl required for the blank titration, and V_2 is the volume of HCl used for the titration of the slurry containing each powdered fraction of weathered IBA.

The modified R^3 test determines the reactivity of SCMs by measuring the heat released during their cementitious reaction with Ca(OH)_2 , offering a more accurate simulation than the modified Chapelle test [35, 36]. The R^3 test was conducted by mixing each powdered fraction of weathered IBA with Ca(OH)_2 in a 1:3 mass ratio, then mixing this solid blend with a 0.5 M reagent-grade KOH solution. Approximately 40 g paste was prepared by blending the solid mixture with the KOH solution at a liquid-to-solid ratio of 0.9, using a high-shear mixer for 4 min. Then, 7 g of paste was poured into a plastic ampoule and placed inside the TAM Air isothermal calorimeter (TA Instruments), which had been preconditioned at $50 \pm 0.05^\circ\text{C}$ [37]. As the calorimeter operates at a higher temperature than the material and ampoule, it takes about 45 min for the signal to stabilise, and this period is excluded from the data. The heat flow from each sample was recorded for 240 h (10 days), after which the samples were taken out of the calorimeter. The pozzolanic activity was quantified based on the heat released over the 10 days period from each powdered fraction of weathered IBA.

In this study, the procedure for determining the SAI was adapted from ASTM C311 standard. Control mortars were prepared following the EN 196–1:2018 standard by mixing 1350 g of normalised sand, 450 g of CEM-I 42.5 R cement, and 225 g of water ($w/c=0.5$). Blended mortars were formulated by replacing 25 wt.% of the cement with a powdered fraction of weathered IBA. Mortars were cast into $40 \times 40 \times 160 \text{ mm}$ prism moulds, using the same water-to-cement ratio ($w/c=0.5$) as the control mortar, where the binder refers to the total amount of cement and IBA. Maintaining a constant water-to-cement ratio is essential to ensure that any variations in compressive strength are solely due to the pozzolanic reaction over time. After 24 h, the specimens



were demoulded and then were cured in a climatic chamber at 20 °C and 95% relative humidity for the remaining time until testing for compressive strength at 28 days. Three specimens were tested for each cement-powdered IBA mortar mixture. The results were expressed according to Eq. (4):

$$SAI = \frac{R}{R_c} \cdot 100 \quad (4)$$

where R is the compressive strength of the blended mortars (MPa), and R_c is the compressive strength of the control mortar (MPa).

Compressive strength tests after 28 days of curing were conducted using an Incotecnica MULTI-R1 universal machine, with a loading rate of $240 \text{ kg}\cdot\text{s}^{-1}$ applied until fracture. Six measurements were taken for each blended and control mortars.

2.4 Environmental assessment

As weathered IBA is a by-product of municipal solid waste incineration, its potential environmental impact was assessed using a batch leaching test to evaluate the potential metal(loid)s release. Leaching concentration of heavy metal(loid)s from each powdered fraction of weathered IBA and blended mortars were analysed according to European standard EN 12457, which classifies waste at the end of its life cycle to assist in landfill management decisions [38]. For the granular leaching test, powdered raw materials (EN 12457-2 for particles lower than 4 mm) and crushed fragments of blended mortars obtained post-compressive strength testing (EN 12457-4 for particles lower than 10 mm) were used. These materials were continuously agitated at 5 rpm in deionised water (with an L/S ratio of $10 \text{ L}\cdot\text{kg}^{-1}$) for 24 h at room temperature. The resulting leachates were filtered using a $0.45\text{-}\mu\text{m}$ nitrocellulose membrane. Each sample was analysed in duplicate, with two aliquots per replicate, using inductively coupled plasma mass spectrometry (ICP-MS) on a PerkinElmer ELAN instrument to determine concentrations of As, Ba, Cd, Cr, Cu, Mo, Ni, Pb, Sb, Se, V, and Zn. The chloride (Cl^-) and sulphate (SO_4^{2-}) anions were measured using an ionic chromatographer 861 Advanced Compact IC Metrohm equipped with Metrosep columns.

The EN-12,457 leaching test was applied to both the powdered IBA size fractions and the blended

mortars formulated with these fractions as SCMs. In all cases, the landfill-acceptance thresholds were used exclusively for comparative purposes, providing a widely standardised reference framework to interpret the leaching behaviour of the raw materials and the corresponding mortars prior to their potential use in construction. These limits are not intended to imply suitability for construction applications.

3 Results and discussion

3.1 Physicochemical characterisation of IBA

The grinding process of each weathered IBA fraction to a particle size below $90 \mu\text{m}$ aimed to achieve a size comparable to that of the PC used as a binder. This ensured the formation of homogeneous mixtures, enabling the evaluation of pozzolanic activity of the different granulometric size fractions of IBA, based on their composition and the reactivity of silica- and alumina-based mineral phases present in each fraction, which react with portlandite in the formation of binder phases (CSH and/or CASH). Therefore, a low-energy grinding process with short time intervals was employed to prevent and/or minimise an increase in pozzolanic activity caused by the formation of additional amorphous phases resulting from mechanochemical activation.

Table 1 presents the particle size distribution parameters (d_{10} , d_{50} , and d_{90}) for each fraction ground to sizes below $90 \mu\text{m}$ (IBA_M). The distributions are highly similar across all fractions, with d_{10} , d_{50} , and d_{90} values remaining within the same order of magnitude. Minor variations can be attributed to differences in the hardness and/or brittleness of the constituent materials in each fraction. Given this uniformity in particle size distribution, any potential pozzolanic effect can be ascribed solely to the mineralogical composition of each granulometric fraction rather than to differences in particle size.

The specific surface area (SSA) of each powdered IBA fraction is also reported in Table 1. While the differences are not highly significant, a clear gradation can be observed as particle size increases, and the specific surface area decreases. This variation is primarily due to differences in material composition and mineralogy. Coarse fractions ($> 16 \text{ mm}$, $8\text{--}16 \text{ mm}$ and $4\text{--}8 \text{ mm}$) contain higher amounts of glass cullet

Table 1 Particle size data (d_{10} , d_{50} and d_{90}) and specific surface area (SSA) of each powdered fraction of weathered IBA

	Particle size fractions (mm)					
	IBA_0-2_M	IBA_2-4_M	IBA_4-8_M	IBA_8-16_M	IBA_> 16_M	IBA_Entire_M
d_{10} (μm)	1.27	1.24	1.59	1.19	1.04	1.10
d_{50} (μm)	10.70	7.80	7.43	6.56	4.86	8.66
d_{90} (μm)	28.65	30.17	35.60	33.74	29.93	36.82
SSA ($\text{m}^2\cdot\text{g}^{-1}$)	11.34	10.16	6.68	4.28	4.89	6.36

d_x : Particle size at which x% of the total volume of particles are smaller

and fired ceramics [14, 31], materials with inherently low porosity, leading to reduced surface area. In contrast, the finer fractions (0–2 mm and 2–4 mm) have higher specific surface areas due to their greater content of soluble salts and neoformed mineral phases.

The major and minor elemental compositions of each powdered fraction of weathered IBA, described as elemental oxides and trace elements, are summarized in Table 2. As expected, the most abundant

oxides were SiO_2 , CaO , and Al_2O_3 , which are also fundamental components in cementitious materials [39]. It should be noted that the content of these three major elements in the different size fractions of weathered IBA closely aligns with values reported by the same authors in studies conducted at other WtE plants within the same geographical region and utilising similar grate technology [14, 32]. The elemental composition varied across size fractions. Coarse

Table 2 Major-, minor- and trace-elements for each size fraction of weathered IBA

	wt. %					
	IBA_0-2_M	IBA_2-4_M	IBA_4-8_M	IBA_8-16_M	IBA_> 16_M	IBA_Entire_M
SiO_2	30.10	37.01	48.38	53.16	45.89	42.71
CaO	20.92	19.12	17.08	13.79	17.42	17.21
Al_2O_3	9.80	8.47	9.11	8.18	12.57	10.18
Fe_2O_3	11.06	9.63	8.92	5.54	7.49	7.23
Na_2O	2.42	3.81	5.48	6.66	3.09	3.47
MgO	2.93	2.93	2.31	2.05	2.66	2.43
K_2O	1.48	1.35	1.31	1.25	1.78	1.55
P_2O_5	1.77	1.58	1.27	0.63	0.48	0.81
TiO_2	0.96	0.87	0.77	0.62	0.84	0.77
SO_3	1.83	1.15	0.79	0.31	0.47	0.67
Cl	0.98	0.60	0.31	0.33	0.16	0.40
Zr	0.05	0.18	0.08	0.11	0.22	0.19
Zn	0.43	0.35	0.23	0.13	0.16	0.19
Cu	0.26	0.20	0.16	0.09	0.08	0.10
Mn	0.14	0.12	0.11	0.10	0.09	0.09
Pb	0.09	0.12	0.04	0.03	0.06	0.08
Sr	0.06	0.06	0.08	0.04	0.08	0.07
Cr	0.10	0.09	0.10	0.09	0.05	0.06
Sc	0.07	0.06	0.05	0.04	0.05	0.05
Ba	0.00	0.04	0.05	0.02	0.06	0.03
Sn	0.04	0.03	0.02	0.02	0.02	0.02
V	0.01	0.01	0.01	0.01	0.01	0.01
Ni	0.03	0.02	0.02	0.01	0.01	0.01
LOI*	14.37	12.16	3.29	6.91	5.89	11.70

*LOI: Loss on ignition at 1000 °C



fractions (> 16 mm, 8–16 mm, and 4–8 mm) exhibited the highest SiO_2 levels, attributed to the presence of primary and secondary glass, along with synthetic ceramic materials found in these fractions [14, 40]. The finest fractions (0–2 mm and 2–4 mm) exhibited a high CaO content, mainly due to the significant presence of calcium carbonates, which form through the weathering of portlandite [32]. The elevated CaO levels can be mostly attributed to cementitious materials derived from PC and synthetic ceramics originating from small-scale domestic construction. These materials undergo fragmentation into combustion furnaces as a result of mechanical attrition and thermal shock [39]. As for Al_2O_3 , its content generally increases as particle size decreases, with the exception of the > 16 mm fraction. This anomaly is attributed to the Eddy current separator, which recovers non-ferrous metals only from the larger particle size fractions. Additionally, the > 16 mm fraction contains a significant amount of fired ceramics, contributing to its Al_2O_3 content [14, 41]. Minor and trace elements, mainly metal(loid)s and chlorides, are predominantly concentrated in the finer fractions of the weathered IBA, with the notable exception of Zr. The higher Zr content in coarser fractions is primarily attributed to the grinding process, as the wear and abrasion of grinding balls occur when handling harder materials, such as fired ceramics and glass, which are more

prevalent in larger fractions. Additionally, more time is required to achieve the target particle size (< 90 μm).

Some studies have demonstrated a positive linear correlation between the SiO_2 and Al_2O_3 content of SCMs and the performance of blended cement [42, 43]. According to the chemical requirements established by ASTM standard C618 [44], for a material to be classified as pozzolanic, the combined content of SiO_2 , Al_2O_3 , and Fe_2O_3 must exceed 70 wt.% in pozzolans. Based on this criterion, none of the powdered fractions of the IBA surpass the 70 wt.%. The fractions with the highest sums are coarse fraction, namely IBA_4-8_M, IBA_8-16_M, and IBA_>16_M with values of 66.41, 66.88, and 65.95 wt.%, respectively.

The XRD patterns of each powdered particle fraction (IBA_M), shown in Fig. 2, further demonstrate that IBA is a heterogeneous material with low crystallinity, primarily composed of amorphous phases, as observed in the halo centred at around 20–35 $2\theta^\circ$. Table 3 presents the main crystalline phases identified in each particle size fraction of IBA, both from sieving and after milling to below 90 μm . The crystalline phases consistently present across all fractions, whether sieved or milled, include akermanite, feldspars, hematite, magnetite, and quartz. Certain mineral phases, such as calcite, were detected in all sieved

Fig. 2 XRD patterns of powdered particle size fractions of IBA: 0–2 mm; 2–4 mm; 4–8 mm; 8–16 mm; > 16 mm and entire fraction

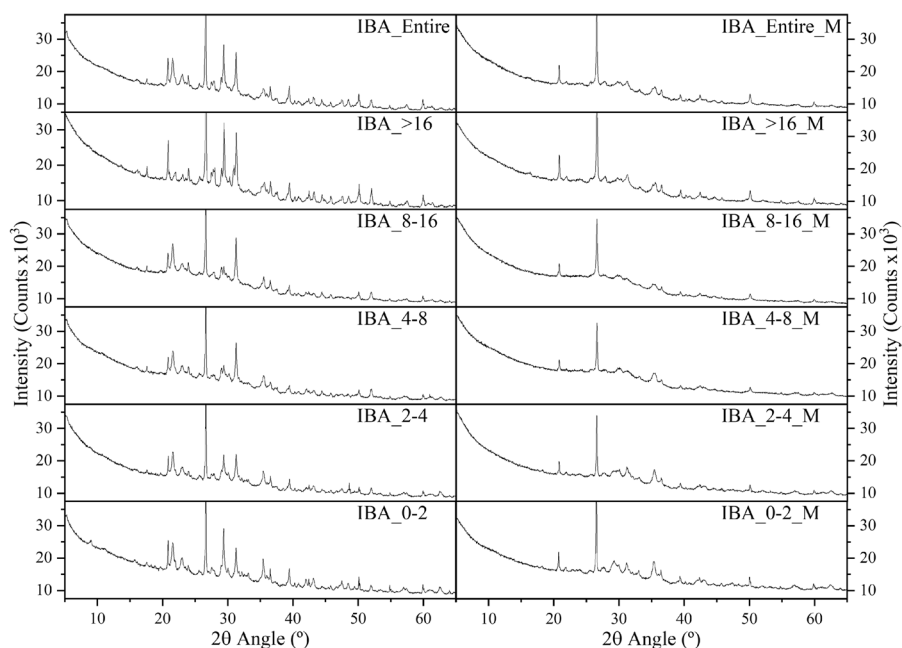


Table 3 Major crystalline mineral phases identified in each particle size fractions of IBA

Mineral phase	IBA_0-2	IBA_0-2_M	IBA_2-4	IBA_2-4_M	IBA_4-8	IBA_4-8_M	IBA_8-16	IBA_8-16_M	IBA_>16	IBA_>16_M	IBA_Entire	IBA_Entire_M
Akermanite	x	x	x	x	x	x	x	x	x	x	x	x
Alumina							x	x	x	x	x	x
Calcite	x	x	x	x	x	x	x	x	x	x	x	x
Cristobalite	x	x	x	x	x	x	x	x	x	x	x	x
Dolomite	x	x	x	x	x	x	x	x	x	x	x	x
Etringite	x	x	x	x	x	x	x	x	x	x	x	x
Feldspars*	x	x	x	x	x	x	x	x	x	x	x	x
Halite	x	x	x	x	x	x	x	x	x	x	x	x
Hematite	x	x	x	x	x	x	x	x	x	x	x	x
Hydrocalumite	x	x	x	x	x	x	x	x	x	x	x	x
Magnetite	x	x	x	x	x	x	x	x	x	x	x	x
Mullite							x	x	x	x	x	x
Quartz	x	x	x	x	x	x	x	x	x	x	x	x
Amorphous content (%)	30	46	31	45	43	59	48	64	35	44	36	53

* Feldspars include but may not be limited to: anorthite, anorthoclase, and albite

Akermanite: $\text{Ca}_2(\text{Mg}_{0.75}\text{Al}_{0.25})_2(\text{Si}_{1.75}\text{Al}_{0.25}\text{O}_7)$; Alumina: Al_2O_3 ; Calcite: CaCO_3 ; Cristobalite: SiO_2 ; Dolomite: $\text{CaMg}(\text{CO}_3)_2$; Etringite: $\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12} \cdot 26\text{H}_2\text{O}$; Feldspars: $\text{CaAl}_2\text{Si}_2\text{O}_8$, $\text{NaAlSi}_3\text{O}_8$, $(\text{Na,K})\text{AlSi}_3\text{O}_8$; Halite: NaCl ; Hematite: Fe_2O_3 ; Hydrocalumite: $\text{Ca}_8\text{Al}_4(\text{OH})_{24}(\text{CO}_3)_3\text{Cl}_2(\text{H}_2\text{O})_{11.6} \cdot 8\text{H}_2\text{O}$; Magnetite: Fe_3O_4 ; Mullite: $\text{Al}_{0.4}\text{Si}_{1.2}\text{O}_{6.6}$; Quartz: SiO_2

fractions but could not be identified in some milled fractions (e.g., IBA_4-8_M and IBA_8-16_M). Additionally, silico-aluminate phases, rich in Si, Al, and Ca, are the most abundant, particularly in the coarser fractions, which is consistent with the results obtained from the XRF elemental characterisation.

The pozzolanic activity of a sample, along with the availability of silica and alumina for the formation of cementitious gels, such as CSH or CASH, increases with the content of amorphous silico-aluminate phases. Therefore, in addition to the chemical and mineralogical composition of the sample, assessing its reactivity and ability to form cementitious phases also requires determining the degree of amorphousness of the sample [45]. Numerous studies have highlighted that both the degree of amorphousness and the particle fineness significantly influence pozzolanic reactivity [45, 46]. Therefore, studies have shown a significant variation in reactivity attributed to differences in chemical composition and amorphous content [36, 47].

As detailed in Table 3, the amorphous content of the particle size fractions ranged from 30 to 62%, which is in close agreement with values reported for other waste-to-energy plants of the same regional area [32]. It is important to note that, for all granulometric fractions, the amorphous phase increased during the milling process (IBA_M), with increases ranging from about 9% to 17% depending on the fraction, despite efforts to minimise milling energy to prevent the mechanochemical activation of mineral phases susceptible to amorphization. As expected, the 4–8- and 8–16-mm size fractions presented the highest amorphous content because of their significant proportion of cullet glass [48], where the amorphous content in the powdered IBA is greater than 60%. Although not as pronounced as the two previous fractions, the remaining granulometric fractions still exhibit a high amorphous content, along with a higher content of fired ceramics and other crystalline mineral phases.

3.2 Pozzolanic activity measurements

Rapid screening tests are commonly used to predict the reactivity of either latent hydraulic or pozzolanic mineral phases contained in SCM with the hydration products of PC. The potential reactivity of SCMs is evaluated using standardized methods such as the

Chapelle and R³ test. According to the literature, IBA generally exhibits lower pozzolanic reactivity than other commonly used SCMs, such as silica fume [49, 50] or metakaolin [51]. Nevertheless, some studies report that its pozzolanic activity is only slightly lower than that of coal fly ash [52]. However, these assessments did not account for the variation in mineralogy and amorphous content across IBA particle size fractions, treating the material as a homogeneous entity without considering the distinct behavior of each fraction.

In this study, the modified Chapelle test, performed in triplicate, was conducted to determine the reactivity of different powdered particle size fractions of IBA with Ca(OH)₂. Pozzolanic reactivity was quantified by measuring the consumption of portlandite, which involves reactive aluminate- or silicate-based mineral phases capable of forming reaction products chemically similar to CSH or CASH cementitious gels. The amount of Ca(OH)₂ consumed per gram of each powdered fraction was calculated using Eq. (3) with the results presented in Fig. 3.

According to French standard NF P 18–513, the amount of CaO consumed must be higher than 660 mg·g⁻¹ of pozzolan (i.e. 872 mg Ca(OH)₂ per g of pozzolan) in order to be considered a pozzolanic material [34, 53]. The results from the modified Chapelle test show that only the IBA_8-16_M fraction (1355 mg Ca(OH)₂·g⁻¹) exhibits a Ca(OH)₂ consumption well above the threshold required for classification as a pozzolanic material. The IBA_4-8_M fraction, with an average consumption of 813 mg

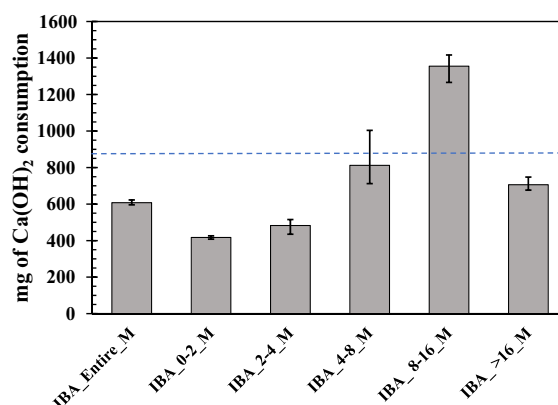


Fig. 3 Pozzolanic activity according to modified Chapelle test for powdered particle size fraction of IBA

Ca(OH)_2 , is very close to this limit, and the maximum value of the replicas also surpasses the threshold set by the French standard. This variation in reactivity closely aligns with the variation in the amorphousness content (see Table 3), where the IBA_8-16_M and IBA_4-8_M fractions, in this order, exhibit the highest contents of amorphous phases, as well cullet glass [14].

Figure 4 presents the results of the modified R^3 test performed on each particle size fraction of powdered IBA. This test quantifies the cumulative heat released during the pozzolanic reaction over a 10-day period, expressed in joules per gram ($\text{J}\cdot\text{g}^{-1}$) of powdered IBA.

The results for each granulometric fraction exhibit a gradation in pozzolanic activity that closely corresponds to the trend observed in the modified Chapelle test. The size fraction IBA_8-16_M, characterised by a higher amorphous phase content (Table 3), also exhibits the highest pozzolanic activity, with a heat release slightly exceeding $350 \text{ J}\cdot\text{g}^{-1}$ of IBA. Very similar results were obtained for the fractions IBA_4-8_M, IBA_entire_M, and IBA_> 16_M, with 10-day cumulative heat release values of 295, 283, and $277 \text{ J}\cdot\text{g}^{-1}$, respectively. For these powdered IBA fractions larger than 4 mm, the determined activity indices indicate that the material can be classified as pozzolanic, less reactive [36]. These values are very similar to those reported for other commonly used

SCMs, such as fly ash from coal-fired power plants and ground granulated blast furnace slag. Within this same category of low-reactivity pozzolanic materials, other substances with a high amorphous content, such as ground glass or finely ground lightweight aggregates, can also be included.

Significantly lower values were recorded for the finer fractions, with 10-day cumulative heat release values of $237 \text{ J}\cdot\text{g}^{-1}$ for IBA_2-4_M and $185 \text{ J}\cdot\text{g}^{-1}$ for IBA_0-2_M. This trend is consistent with the amorphous phase content of the fractions, reinforcing the correlation between amorphous content and pozzolanic activity. Although milling to obtain a particle size below $90 \mu\text{m}$ induced a measurable increase in reactivity through the mechanochemical activation of certain mineral phases and led to a 9–17% rise in the amorphous phase, together with the expected increase in specific surface area resulting from particle-size reduction, these effects are relatively uniform across all IBA fractions. Therefore, this contribution is considered minor compared to the dominant influence of the initial amorphous phase content.

The SAI is a widely used indirect approach to evaluate pozzolanic activity, based on comparing the compressive strength of pozzolan–cement blended mortars to that of reference mortars made solely with Portland cement [54]. The SAI evaluation is presented in Fig. 5. For reference, the figure includes the 28-day compressive strength of

Fig. 4 Cumulative heat released over 10 days in the modified R^3 test for each particle size fraction of powdered IBA

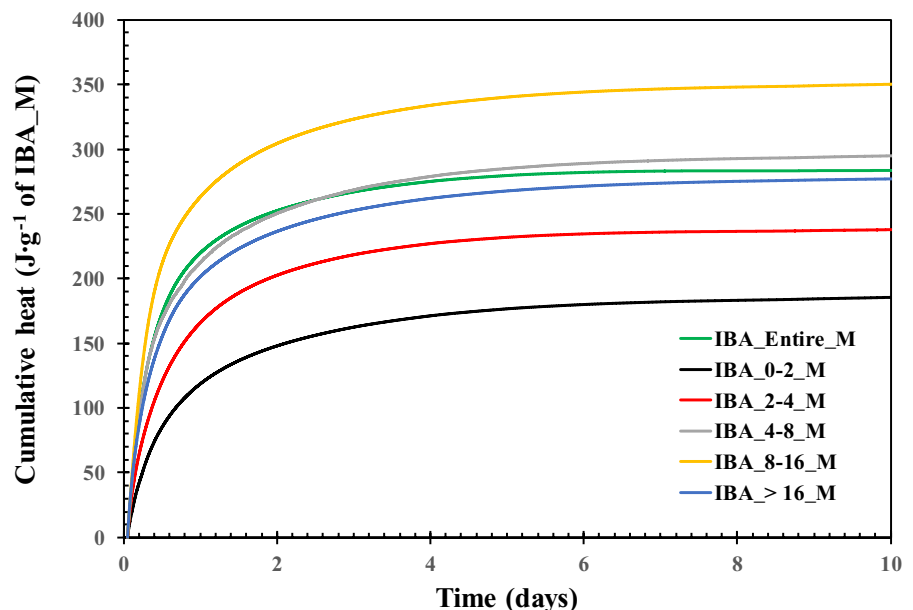
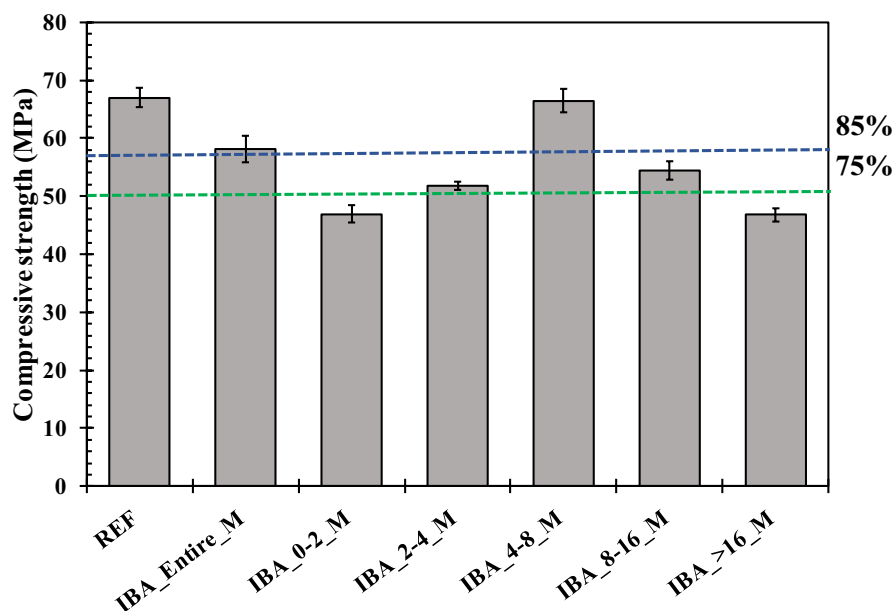


Fig. 5 SAI of mortars evaluation expressed as compressive strength and percentage relative to control mortar (REF)



the control mortar (REF), composed exclusively of CEM I PC as binder. Additionally, guideline lines representing 75% and 85% of the control mortar's compressive strength have been added to facilitate comparison. According to ASTM C618, the acceptance criteria for the SAI ensure that SCMs provide a minimum required contribution to the mechanical performance of mortar or concrete. Specifically, the SAI, calculated according to Eq. (4), must reach at least 75% of the compressive strength of the control mortar, tested under standard curing conditions after 28 days. Given that the blended cements incorporate a 25% substitution of Portland cement, it is expected that the pozzolanic activity of SCM will contribute to a strength increase exceeding 75% of the control mortar's compressive strength. Therefore, achieving 85% of the control mortar's strength is considered an optimal outcome, indicative of an SCM with good pozzolanic activity.

As shown in Fig. 5, three size fractions of powdered IBA, namely, IBA_2-4_M, IBA_4-8_M, and IBA_8-16_M, exhibit a SAI exceeding 75% of the control mortar's compressive strength. Among them, the IBA_4-8_M fraction stands out with a value significantly above 85%, reaching 99% SAI. In contrast, the IBA_0-2_M and IBA_>16_M fractions fall slightly below the threshold, both with SAI values of approximately 70%. The entire fraction (IBA_entire_M), which represents a weighted combination

of all particle sizes, also performs well, achieving an SAI just above 85% (87%).

This trend differs significantly from the gradation observed in the modified Chapelle and modified R^3 tests, where the IBA_8-16_M fraction demonstrated the highest pozzolanic activity. This discrepancy can be attributed to the inherent differences in the nature of the pozzolanic activity tests. In both the modified Chapelle and R^3 tests, the amorphous phases react with an excess of externally added portlandite under well-controlled conditions. In contrast, in the SAI test, the $\text{Ca}(\text{OH})_2$ is generated in situ through the hydration of calcium silicates and aluminosilicates present in the Portland clinker. Furthermore, the modified Chapelle and R^3 tests are conducted at elevated temperatures (90 °C and 50 °C, respectively), which enhance the pozzolanic reactivity of the amorphous phases. Probably, the amount of portlandite generated during clinker hydration is scarce to ensure complete activation of the amorphous phases within the IBA fractions. Consequently, the chemical nature of these amorphous phases, along with their reaction kinetics in alkaline media, may also play a critical role in their activation. For instance, although the IBA_8-16_M fraction exhibits the highest amorphous content (see Table 3), this amorphous phase is mainly composed of primary glass derived from glass cullet and fragments of concrete or cement originating from minor

domestic refurbishments. In contrast, the content of secondary glass is significantly lower in this fraction compared to the finer particle size fractions. Secondary glass refers to newly formed amorphous mineral phases generated at high temperatures [14]. It is typically more fragile and is characterised by high porosity, including gas bubbles, vesicles, cracks, and inclusions of other materials. While it may originate from primary glass, it can also form from the melting of other silicate materials or silica-rich components present in MSW. Consequently, its activation can occur more rapidly and under less strongly alkaline conditions. This observation may account for the fact that the IBA_4-8_M fraction, although possessing a lower amorphous content than IBA_8-16_M, achieves a markedly higher SAI, approaching the value of the control mortar.

Despite the observed differences in pozzolanic activity among the various particle size fractions, powdered IBA can generally be considered a suitable SCM, as it meets the technical requirements outlined in ASTM C618. It is noteworthy that the IBA_entire_M, which did not yield excellent results in the modified Chapelle and R³ tests to determine pozzolanic activity, achieved values exceeding 85% of the compressive strength of the control mortar (REF) in the SAI test. This suggests that, in addition to the formation of cementitious phases generated from the pozzolanic reactions of the amorphous phases in the IBA, there may also be a mechanical effect from the non-reactive mineral phases. These

phases act as fillers, enhancing the compactness of the mortars.

3.3 Environmental assessment

Table 4 presents the leaching concentrations of metal(loid)s, chloride, and sulphate from each particle size fraction of powdered IBA, alongside the threshold limits established by the European Union for waste acceptance at landfills [38]. Before analysing these results, two critical considerations must be taken into account: (i) the IBA used had previously undergone natural weathering outdoors for a period of 2–3 months, a well-established practice to reduce the potential release of contained metal(loid)s [11], and (ii) each granulometric fraction was powdered to achieve a particle size below 90 µm. This significant reduction in particle size resulted in a marked increase in the specific surface area of the IBA powder (see Table 1), which in turn further promoted oxidation, carbonation, and the formation of new mineral phases typically associated with natural aging processes, prior to its use as SCM in the mortar formulation. Consequently, weathering is generally expected to reduce the leaching of metal(loid)s. However, the increased surface area may also enhance chemical reactivity and diffusion-controlled processes, potentially facilitating the release of certain metal(loid)s under specific conditions.

The leaching behaviour of metal(loid)s observed in the different powdered IBA fractions is consistent

Table 4 Leaching concentration (mg·kg⁻¹) for each size fraction of powdered IBA from the EN-12457-2 leaching test, and the regulatory limits for waste acceptance at landfills [38]

	Zn	Cr	Mo	Ni	Cu	Sb	As	Pb	Cd	Hg	Ba	Se	Cl ⁻	SO ₄ ²⁻	pH
IBA_0-2_M	<0.05	0.09	2.97	0.10	5.57	0.69	0.02	<0.01	<0.01	<0.01	0.72	<0.10	5194	6407	10.09
IBA_2-4_M	<0.05	0.10	2.48	0.19	7.67	<i>1.25</i>	0.03	<0.01	<0.01	<0.01	0.36	<0.10	3624	5600	11.53
IBA_4-8_M	0.05	0.02	0.83	0.03	1.91	<i>0.94</i>	0.33	<0.01	<0.01	<0.01	0.05	<0.10	1158	1129	11.75
IBA_8-16_M	<0.05	0.02	1.17	0.02	1.16	<i>0.46</i>	0.29	<0.01	<0.01	<0.01	0.02	<0.10	1077	1585	11.94
IBA_>16_M	0.14	0.09	1.10	0.03	1.80	<i>1.18</i>	0.09	0.02	<0.01	<0.01	0.15	<0.10	1022	1412	11.51
IBA_Entire_M	0.05	0.07	1.02	0.04	3.92	<i>1.52</i>	0.17	0.08	<0.01	<0.01	0.10	<0.10	1566	1676	11.20
Inert	4	0.5	0.5	0.4	2	0.06	0.5	0.5	0.04	0.01	20	0.1	800	1000	–
Non-hazardous	50	10	10	10	50	0.7	2	10	1	0.2	100	0.5	15,000	20,000	–
Hazardous	200	70	30	40	100	5	25	50	5	2	300	7	25,000	50,000	–

(*) Cells highlighted in Bold and Italics represent concentrations exceeding the regulatory thresholds for inert and non-hazardous waste, respectively, as defined by EU landfill acceptance criteria



with previous studies in which the same fractions were used as precursors for alkali-activated cements [32]. Once again, the finer fractions exhibit higher leaching values, in line with their greater metal(loid) content.

As shown, most of the metal(loid)s exhibit concentrations below the regulatory limits for inert waste. However, molybdenum and copper exceed the inert waste thresholds in certain fractions of the powdered IBA, although their concentrations remain below the limits for non-hazardous waste. Only antimony, released from powdered IBA fractions larger than 2 mm, exceeds the threshold established for non-hazardous waste.

The solubility of pH-dependent metals such as zinc, lead, nickel, and cadmium is significantly influenced by the pH reduction associated with carbonation processes. Their solubility is primarily controlled by the formation of corresponding hydroxides and/or carbonates [55, 56]. Additionally, the formation of new mineral phases resulting from the oxidation of iron and aluminium contributes to the adsorption of certain metals, such as arsenic and nickel [56, 57]. Overall, these metal(loid)s exhibit leachate concentrations that are significantly lower than the threshold values set for inert materials.

With regard to copper, it has been reported that its solubility is enhanced by its strong tendency to form complexes with dissolved organic carbon [29, 58], a phenomenon observed even in IBA subjected to weathering for periods exceeding 1.5 years [55]. In the present study, the increase in specific surface area resulting from the grinding process is proposed to enhance the release of the copper-organic complex.

Molybdenum leaching is largely independent of pH under alkaline conditions [58]. The elevated concentrations observed in the leachate are consistent with the higher bulk molybdenum content of the powdered IBA fractions relative to the others, and may also be attributed to molybdenum release during the weathering process, potentially enhanced by the increased specific surface area. In this regard, the higher concentrations of both copper and molybdenum in the finer fractions suggest that these elements are more enriched in smaller particle sizes.

Small amounts of antimony oxides are commonly added as chemical clarifiers and decolourisers during glass production. Since glass cullet is one of the main components of MSW, the presence

of antimony in the different particle size fractions of the IBA can be attributed to its content in primary glass. The formation of calcium antimonates during MSW incineration likely controls both the stability and solubility of antimony over a broad pH range [59]. In this context, the high availability of calcium may promote the formation of these compounds, thereby limiting antimony leaching. In this assumption, the highest available calcium content is found in the finer fractions, whereas in the coarser fractions, where the primary glass content is also higher, calcium is predominantly present as a component of synthetic fired ceramics.

With regard to the leaching of chlorides and sulphates, all particle size fractions of the powdered IBA exhibit concentrations that exceed the thresholds for inert materials but remain below those for non-hazardous waste. Notably, the highest levels of chloride and sulphate leaching were observed in the finest fractions, where a significant proportion of soluble salts is concentrated. This trend in leaching behaviour aligns with the chlorine and sulphur contents determined by XRF analysis (see Table 2), which also indicates that the highest concentrations of both elements are found in the finest fractions. Chloride leaching from SCMs is particularly important because it is a primary factor in the corrosion of reinforcing steel in concrete, which can severely compromise the material's durability and significantly shorten its service life. To ensure the durability of reinforced concrete, international standards such as EN 450–1 [60] stipulate a maximum water-soluble chloride content of 0.10% ($1000 \text{ mg}\cdot\text{kg}^{-1}$ by mass) for coal fly ash intended for use as an SCM. In the present study, all particle-size fractions larger than 4 mm slightly exceed this regulatory threshold.

Table 5 presents the leaching concentrations of metal(loid)s, chloride, and sulphate from each blended mortars formulated with the different particle size fraction of powdered IBA. As shown, the pH of the leachate for all blended mortars is above 12, controlled by the portlandite formed during the hydration of Portland cementitious phases. Only molybdenum exhibits leaching concentrations slightly above the limit established for inert materials ($0.5 \text{ mg}\cdot\text{kg}^{-1}$), while all other metal(loid)s, chloride and sulphate remain below the corresponding threshold values (see limits in Table 4).

Table 5 Leaching concentration ($\text{mg}\cdot\text{kg}^{-1}$) of blended mortars formulated with each size fraction of powdered IBA from the EN-12457-4 leaching test

	Zn	Cr	Mo	Ni	Cu	Sb	As	Pb	Cd	Hg	Ba	Se	Cl ⁻	SO ₄ ²⁻	pH
IBA_0-2_M	0.06	0.13	0.46	0.02	0.17	<0.01	<0.02	0.16	<0.01	<0.01	0.01	<0.10	123	13	12.31
IBA_2-4_M	<0.05	0.09	0.54	0.04	0.16	<0.01	<0.02	0.12	<0.01	<0.01	0.01	<0.10	103	42	12.19
IBA_4-8_M	<0.05	0.05	0.70	0.01	0.08	<0.01	<0.02	0.09	<0.01	<0.01	<0.01	<0.10	79	56	12.24
IBA_8-16_M	0.06	0.04	0.65	0.01	0.10	<0.01	<0.02	0.07	<0.01	<0.01	<0.01	<0.10	77	38	12.14
IBA_>16_M	<0.05	0.05	0.45	0.01	0.10	<0.01	<0.02	0.09	<0.01	<0.01	0.01	<0.10	71	33	12.16
IBA_Entire_M	<0.05	0.07	0.63	0.01	0.04	<0.01	<0.02	0.07	<0.01	<0.010	<0.01	<0.10	100	36	12.26

The low concentrations of metal(loid)s, chloride, and sulphate observed in the blended mortars can be attributed to the combined effect of the dilution of the powdered IBA, the encapsulation provided by the cementitious matrix, and the higher availability of calcium, which promotes the formation of insoluble mineral phases that limit the solubility of certain metal(loid)s (e.g., antimony) and sulphate.

4 Conclusions

The chemical and structural analysis of powdered municipal solid waste incineration bottom ash (IBA) fractions indicate their potential as supplementary cementitious materials (SCMs). The main components identified were SiO_2 , CaO and Al_2O_3 , with their proportions varying depending on particle size. Coarser fractions contained more SiO_2 , while finer ones had higher CaO content. Although none of the fractions met the ASTM C618 requirement of more than 70 wt.% combined SiO_2 , Al_2O_3 and Fe_2O_3 for classification as pozzolanic materials, the coarse fractions showed values close to this limit, reaching around 66 wt.%. Additionally, the amorphous content ranged from 30 to 62%, with the 4–8 mm and 8–16 mm fractions showing the highest values due to their high content of cullet glass. These results suggest that, even if the chemical criteria are not fully met, some IBA fractions may still show pozzolanic behaviour due to their high amorphous content.

The results from both the modified Chapelle and R^3 tests consistently demonstrate a gradation in pozzolanic activity among the powdered IBA fractions, closely related to their amorphous content. In the modified Chapelle test, only the 8–16 mm fraction exceeded the minimum $\text{Ca}(\text{OH})_2$ consumption

threshold required for pozzolanic classification, with a value of $1355 \text{ mg}\cdot\text{g}^{-1}$. The 4–8 mm fraction also showed promising reactivity, with an average consumption of $813 \text{ mg}\cdot\text{g}^{-1}$, approaching the limit defined by the French standard. In the modified R^3 test, the same fractions displayed the highest reactivity. The 8–16 mm fraction released over $350 \text{ J}\cdot\text{g}^{-1}$ of cumulative heat after 10 days, followed by the 4–8 mm and >16 mm fractions, with values of 295 and $277 \text{ J}\cdot\text{g}^{-1}$, respectively. These results reinforce the strong correlation between pozzolanic reactivity and the amorphous content, particularly in fractions rich in cullet glass.

Three powdered IBA fractions (2–4 mm, 4–8 mm, and 8–16 mm) and the entire fraction exceeded 75% in the strength activity index (SAI), with the 4–8 mm fraction reaching 99% of the control mortar's compressive strength, indicating good pozzolanic performance. Unlike trends in the Chapelle and R^3 tests, this result may be due to differences in test conditions and the chemical nature of the amorphous phases, where the finer fractions contain more reactive secondary glass, enabling higher activation despite lower overall amorphous content.

The environmental assessment of the blended mortars shows low metal(loid)s leaching, mainly due to the dilution of powdered IBA, its encapsulation within the cementitious matrix, and the availability of calcium, which promotes the formation of insoluble phases that reduce solubility. At the end of their life cycle, all particle size fractions meet the regulatory limits for inert materials, except for molybdenum, which slightly exceeds the established threshold.

Overall, the results indicate that powdered IBA fractions larger than 4 mm have strong potential for use as SCMs. These fractions contain relatively high amounts of SiO_2 , CaO , and Al_2O_3 , as well as a



significant proportion of amorphous phases, which contribute to their reactivity. They also demonstrated good mechanical performance, with strength activity indices exceeding 75% of the reference mortar. From an environmental perspective, the blended mortars formulated using IBA fractions larger than 4 mm exhibited low leaching of metal(loid)s and the lowest levels of water-soluble chloride, remaining well within acceptable regulatory limits. These findings support the selective use of IBA fractions larger than 4 mm as safe and effective SCMs, offering a sustainable alternative in cementitious formulations.

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Data availability Data will be made available on request.

Declarations

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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