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Valorization of Solid Residues from the Incineration of Expired Medicines in Cement Mortar

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ABSTRACT: The incineration of expired medicines is the most practical method to prevent their accidental use; however, it generates solid residues (bottom ash and fly ash) containing heavy metals and various chemical compounds that are harmful to human health and the environment. This study aims to valorize these residues in the field of civil engineering as additives in the production of Portland cement for mortar manufacturing, in order to reduce cement consumption and environmental impact. The solid residues were characterized using X-Ray Diffraction (XRD), X-Ray Fluorescence (XRF), Inductively Coupled Plasma–Mass Spectrometry (ICP-MS), and Scanning Electron Microscopy (SEM), as well as geotechnical and pozzolanic analyses. The results indicate that the materials are inert and suitable for use as cement substitutes. They were incorporated as partial replacements for cement at 10%, 15%, 20%, and 25%. Mechanical strength and durability against acid attacks were evaluated. The results show that bottom ash and fly ash can partially replace Portland cement up to 10% and 20%, respectively, while improving the mechanical strength and acid resistance of the mortar compared to the control. The proposed valorization provides a simple, economical, and sustainable solution for the management of incineration residues.

1. INTRODUCTION

Pharmaceutical waste, such as expired medicines, can pose a threat to human health, and its management constitutes a significant environmental problem. Therefore, expired medicines must be subject to proper collection and disposal. In Algeria, incineration remains one of the most suitable techniques for the elimination of expired medicines. It makes it possible to reduce the volume and the mass of such waste. However, environmental problems persist. Indeed, the process generates solid residues (bottom ash and fly ash), which constitute incineration by-products. The use of incineration by-products in building materials is very widespread in developed countries due to its ecological impact and economic benefits. These industrial residues are recovered and recycled as a partial replacement for Portland cement in concrete, which reduces greenhouse gas emissions and contributes to the production of more sustainable concrete [1,14]. Recent systematic reviews have highlighted that the conversion of various medical and pharmaceutical waste into supplementary cementitious materials offers dual benefits in terms of circular economy and structural performance [14].

Bottom ash (BA) and fly ash (FA) have already been the subject of several valorization studies in road construction and cement production [2]. These alternatives contribute to reducing pollution and make it possible to meet the needs of certain industrial stakeholders who are seeking ways to eliminate their waste in line with a sustainable development approach. Thus, in the present work, the solid by-products (BA and FA) from expired medicines incineration generated by the Algerian

company "ECFERAL" specialized in incineration are thoroughly characterized with a view to their possible valorization in the civil engineering sector as a partial substitute for Portland cement in mortars.

2. MATERIALS AND METHODS

2.1. Bottom Ash and Fly Ash

The bottom ash (BA) and fly ash (FA) used in this study were obtained from the incineration of three expired medicines: KLAVOX® tablets, KLAVOX® powder for syrup, and BACTROBAN® ointment. The extracted and wet-cooled BA appears as a heterogeneous dark grey gravel. It is composed of a mixture of glass, silica, alumina, limestone, lime, unburnt matter, water, and traces of heavy metals [2]. FA, consisting of fine particles recovered by a dust extractor and/or a filter, represents between 2% and 8% of the total weight of solid residues produced by the incinerator, the remainder being BA. The chemical compositions of both BA and FA are similar; only their proportions differ [3].

2.2. Cement

The cement used in this study is an artificial Portland cement (CPA-CEM I), class 30.17 MPa, prepared using clinker nodules obtained from the cement plant of Sour El Ghozlane in the Wilaya of Bouira (114 km east of Algiers), to which 5% gypsum was added. After grinding, the final powder obtained was sieved through an 80 μm sieve [4, 5]. The Blaine specific surface area (BSS), the apparent density, and the absolute density of the obtained cement are 5421 cm^2/g, 850 kg/m^3, and 3140 kg/m^3, respectively. The chemical and mineralogical compositions of the cement are presented in Table 1 and Table 2, respectively.

Table 1: Chemical composition of cement

Components	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	MnO	K_2O	P_2O_5	TiO_2	SO_3
Content (%)	22.30	4.34	2.47	61.26	2.50	<0.05	0.57	1.00	0.24	0.72

Table 2: Mineralogical composition of cement

Components	C_3S	C_2S	C_3A	C_4AF
Content (%)	47.17	28.39	7.32	7.51

2.3. Sand

The sand used for mortar formulation is a local sand from the Baghlia region in the Wilaya of Boumerdès (50 km east of Algiers). It complies with the "NFP 18-598" and "NF P18-540" standards. The physical parameters and granulometric curve are summarized in Table 3 and shown in Figure 1, respectively. This sand is clean sand and suitable for high-quality mortars [6].

Table 3: Physical characteristics of sand

Physical characteristics	Values
Apparent volumetric mass (g/cm^3)	1.63
Absolute volumetric mass (g/cm^3)	2.34
Fineness modulus	2.38
Water content (%)	0.08
Sand equivalent: ESV (%)	76.67
Sand equivalent: ESP (%)	75.00

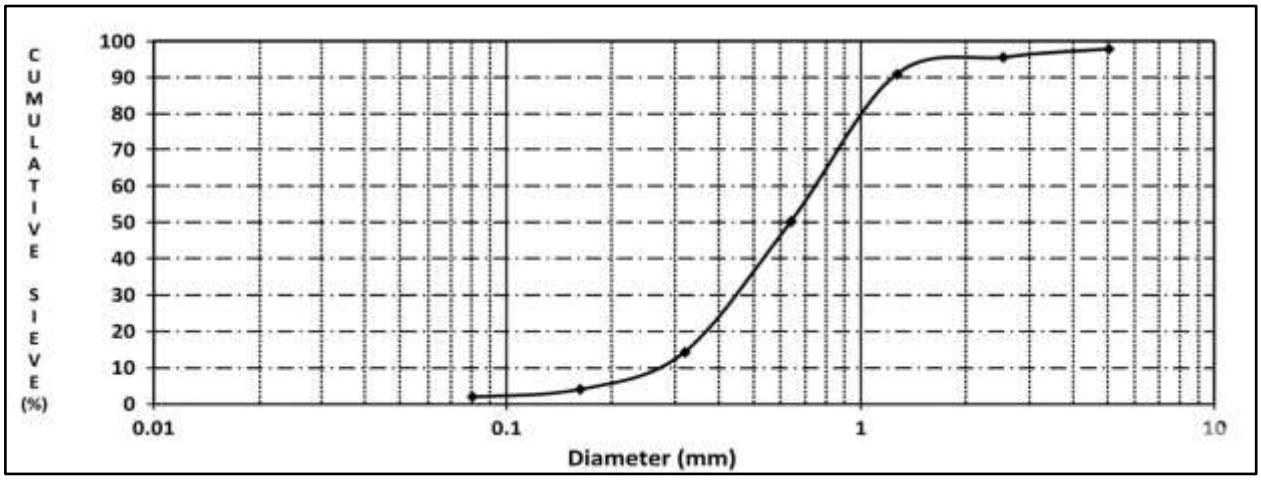


Fig. 1 Granulometric curve of the sand

2.4. Water

The mixing water used in the study is tap water.

2.5. Analysis and Characterization of Solid Residues

The characterization of BA and FA was carried out on samples that had undergone the following preliminary operations: drying at 105°C, grinding using a ball mill, and sieving through an 80 μm sieve.

2.5.1. Physical Characterization

The morphological observations of BA and FA were carried out using a scanning electron microscope (SEM) JEOL JSM-6360 with magnifications of ×250 and ×1000. Particle size distribution was analyzed by a MASTERSIZER 2000 granulometer equipped with a dry process module. The absolute and apparent densities were determined according to the "NF EN 196-1" standard. The humidity and unburnt contents were determined in accordance with "NF X 31-210" and the circular of 09/05/1994 related to the elimination of incineration bottom ash, respectively. Finally, the Blaine specific surface area was measured according to the NF EN 196-6 standard.

2.5.2. Chemical Characterization

The pH values of BA and FA were determined using a pH meter. The chemical composition of the major elements, as well as chlorides and sulfates, was analyzed by X-ray fluorescence spectrometry (XRF) using an AXIOS PANalytical apparatus. The mineralogical characterization was performed by X-ray diffraction (XRD) using a PANalytical X'Pert Pro diffractometer. The analysis of heavy metals in the eluates was carried out using an ICP-MS (Agilent Technologies 7700X series). A study of the environmental behavior of these residues through a leaching test was also conducted.

2.6. Preparation of Mortar Samples Based on BA and FA

The use of BA and FA as partial replacements for cement led to the preparation of several mortar formulations, which were compared with a control mortar produced under the same conditions. The mortar samples were prepared according to the EN 196-1 standard. The specimens consisted of one part cement, three parts sand, and half a part water (water-to-cement ratio of 0.5). Prismatic mortar specimens measuring 40 × 40 × 160 mm^3 were cast for mechanical strength testing. After 24 hours, the specimens were demolded and cured in water until testing. Three-point bending tensile tests were performed, followed by compressive strength tests on the resulting half-prisms at different curing times (7, 14, and 28 days). Cement was partially replaced by BA and FA at levels of 10%, 15%, 20%, and 25% in the mortar mixtures. A total of eight substituted mortar formulations were obtained and labeled as follows: M10%BA, M15%BA, M20%BA, M25%BA, M10%FA, M15%FA, M20%FA, and M25%FA. These specimens were tested and compared with the control mortar without any addition, denoted as CM. These substitution levels were selected based on previous studies [7-9]. The mass proportions of the constituents for each mortar formulation are presented in Table 4

Table 4: Mass proportions of formulated mortars

Designation of mortars	Cement (g)	Sand (g)	Water (g)	BA (g)	FA (g)
CM	450.0	1350.0	225.0	0.0	0.0
M10% BA	405.0	1350.0	225.0	45.0	0.0
M15% BA	382.5	1350.0	225.0	67.5	0.0
M20% BA	360.0	1350.0	225.0	90.0	0.0
M25% BA	337.5	1350.0	225.0	112.5	0.0
M10% FA	405.0	1350.0	225.0	0.0	45.0
M15% FA	382.5	1350.0	225.0	0.0	67.5
M20% FA	360.0	1350.0	225.0	0.0	90.0
M25% FA	337.5	1350.0	225.0	0.0	112.5

2.7. Mechanical Strength Tests

2.7.1. Flexural Strength Test

The flexural strength test was carried out in accordance with the NF EN 196-1 standard using a TONI TECHNIK machine. The test consists of breaking a specimen placed on two supports. The flexural strength (Sf), expressed in megapascals (MPa), is calculated using the following formula:

$Sf = (1.5 \times Ff \times l) / b^3$ (1)

Where:

• Ff : is the load applied in the middle of the specimen at failure, expressed in Newtons (N).

• b : is the side of the square section of the specimen, expressed in millimeters (mm).

• l : is the distance between the supports, expressed in millimeters (mm).

2.7.2. Compressive Strength Test

The compressive strength of the mortars was determined in accordance with the NF EN 196-1 standard using a TONI TECHNIK machine. The half-prisms obtained after the flexural strength test were subjected to an increasing load using a compression press until failure, as illustrated in Figure 2 and Figure 3. The compressive strength (Rc), expressed in megapascals (MPa), is calculated using the following formula:

$Rc = Fc / b^2$ (2)

Where:

• Fc is the maximum load applied to the half-prism of the specimen, expressed in Newtons (N). • b is the compression surface area of the specimen, expressed in square millimeters (mm^2).

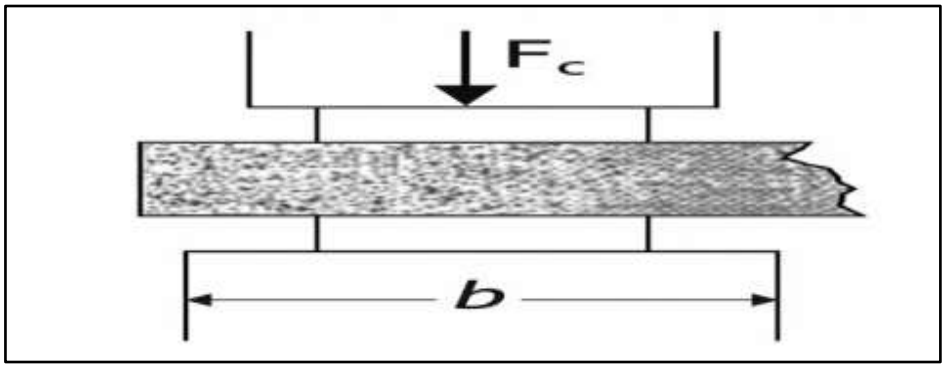


Fig. 2 Diagrams of specimen position for compressive strength test



Fig. 3: Rupture of the half-prisms of a specimen by a compressive strength device.

2.8. Activity Index

According to the ASTM C618 standard, a material is classified as a pozzolan when it meets the following criteria: - Its chemical composition satisfies the relation: $Al_2O_3 + SiO_2 + Fe_2O_3 > 70\%$. - Its activity index (I) ≥ 0.67. The activity index is defined as the ratio of the compressive strength of a mortar containing the supplementary material to that of a control mortar made with 100% cement [10].

However, the potential pozzolanic properties of "BA" have been little discussed in the literature. Regarding coal combustion residues, studies on pozzolanicity have focused more on fly ash because it has the advantage of a fine particle size similar to that of cement. Nevertheless, since 1999, studies have been carried out mainly on the potential use of bottom ash as an additive in cement. The studies conducted by Cheriaf et al. on Brazilian clinker show that it exhibits pozzolanic reactivity (around 88% at 28 days), allowing its use as an additive in the production of Portland cement. Kurama et al. have also shown that by replacing the cement with 15% clinker, an increase in the flexural and compressive strengths of mortars is observed after 56 days of curing. Test methods based on mechanical strength are provided by ASTM standards, independently of standard chemical methods, to determine the effectiveness of a pozzolan. One can simply compare the mechanical strength of two series of mortar specimens, one made by replacing part of the cement with pozzolan and the other with an inert powder. If the mortars containing pozzolana exhibit higher mechanical strength, then the pozzolan is considered active [11].

2.9. Durability Tests Against Acid Attacks

The study of durability against acid attack was carried out on control mortars and mortars incorporating BA and FA incineration residues that exhibited the best mechanical strength. The mortars were prepared in the form of discs in accordance with the NF EN 196-1 standard. After 28 days of water curing, the discs were weighed to determine their initial mass before the acid attack, and then immersed in two 5% acid solutions: a hydrochloric acid solution and a sulfuric acid

solution. These acids are considered among the most aggressive natural chemical agents [12]. The resistance of the discs to acid attack was evaluated in accordance with the ASTM C267-2001 standard [13]. The discs were weighed before immersion and then kept in the acidic solutions for 7, 14, and 28 days. At each specified time, the discs were cleaned three times with tap water to remove the altered mortar and then left to dry for 30 minutes. The dried discs were subsequently weighed at 7, 14, and 28 days (Figures 4, 5, 6, and 7). The resistance of the specimens to chemical attack was evaluated by measuring the mass loss using the following formula:

Mass loss (%) = [(M1 - M2) / M1] × 100 (3)

Where:

• M1 is the mass of the disc before immersion. • M2 is the mass of the cleaned and dried disc after immersion.



Fig. 4: Preparation of mortar discs without additives and with additives



Fig. 5: Curing mortar discs in water for 28 days



Fig. 6: Immersion of mortar discs in acidic solutions

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Fig. 7: Weighing mortar discs after immersion in acidic solutions

3. RESULTS AND DISCUSSIONS

3.1. Physical Characterization

Figures 8 and 9 present the micrographs of BA and FA obtained by SEM at magnifications of ×250 and ×1000. SEM images show that BA consists of angular and porous particles, whereas FA is composed of smooth spherical particles. The packing effect of the spherical particles in FA contributes to reducing its permeability.

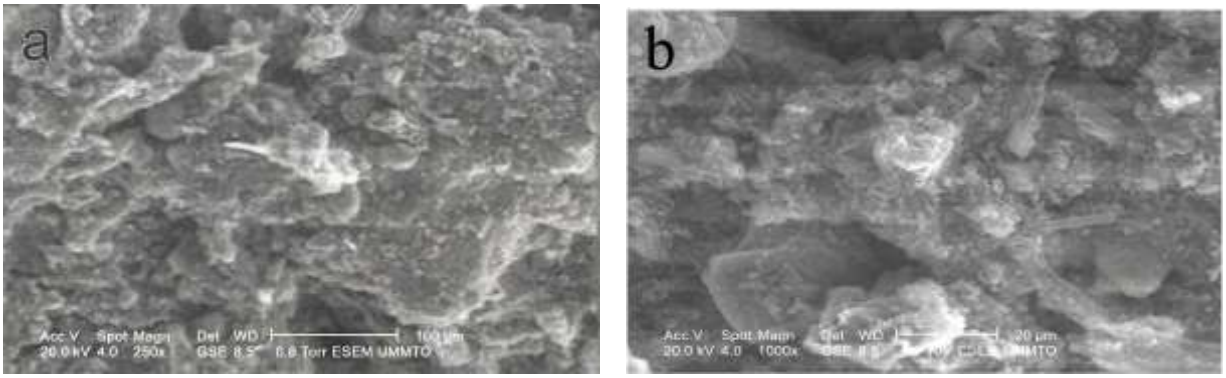
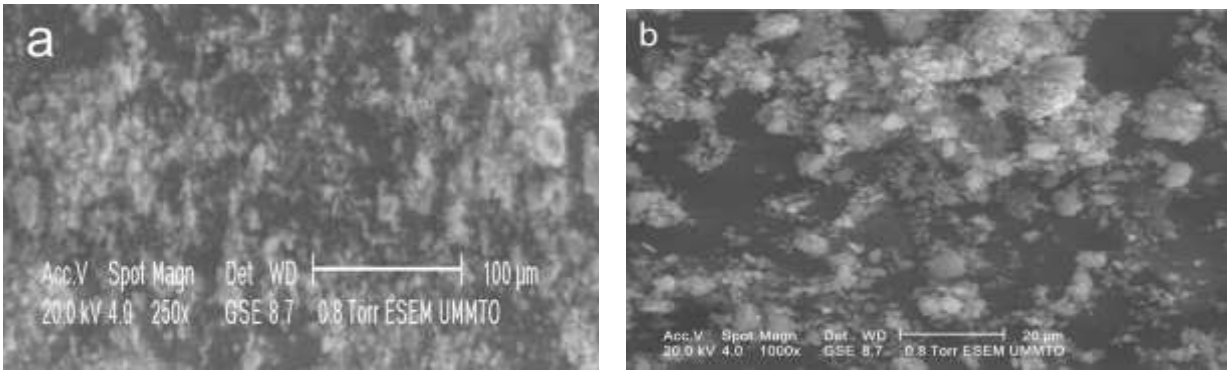


Fig. 8: Micrographs of BA by SEM Magnification × 250 (scale 100 µm) (a) and Magnification × 1000 (scale 20 µm) (b)



Y **Fig. 9:** Micrographs of FA by SEM Magnification × 250 (scale 100 µm) (a) and Magnification × 1000 (scale 20 µm) (b)

It can be observed that, after grinding and sieving, the particle size distribution of FA (from 0.5 µm to 8 µm) is more homogeneous than that of BA (from 0.5 µm to 50 µm), as shown in Figures 10 and 11. This is probably because FA is initially characterized by fine particles. It can be concluded that the fineness of both FA and BA allows their incorporation into building materials for valorization purposes.

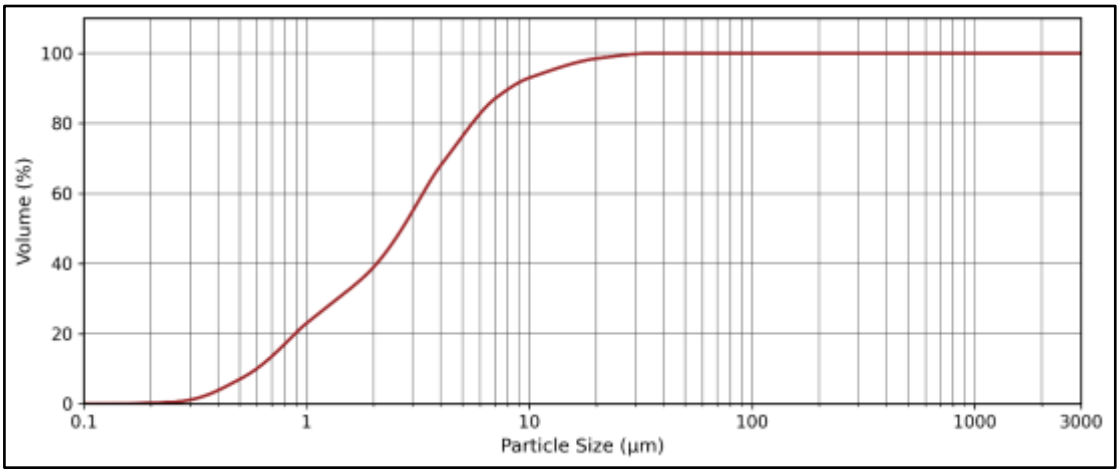


Fig. 10: Particle size distribution of FA

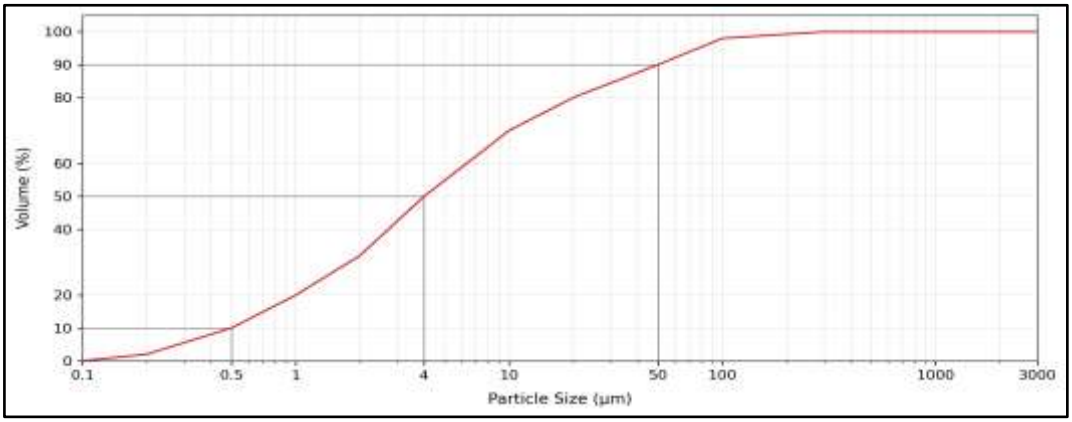


Fig. 11: Particle size distribution of BA

The absolute and apparent densities of FA are lower than those of BA (Table 5), which makes FA a relatively lightweight material. These results are directly related to the morphology and the particle size of FA, which is spherical and homogeneous, whereas BA is an angular and porous material. The moisture content of BA is very low compared to that of FA, which is expected. In fact, FA is recovered wet after incineration, while BA is recovered from the oven after cooling in open air. These materials are therefore suitable for valorization because their moisture contents are below 10%. However, they must be subjected to drying before any use.

The rates of unburned materials in BA and FA are less than 5%, which indicates good incineration efficiency and allows these residues to be classified in the category of recoverable waste "V", according to the circular of May 9, 1994, relating to the disposal of bottom ash. BA and FA have significant BSS, and the BSS of FA is the highest. The result confirmed the greater fineness of FA grains with spherical morphology and solid particles. Fineness is an important material property during mixing, as a larger surface area of the residues in contact with water promotes faster and more complete hydration, leading to higher mechanical strength.

Table 5: Physical characteristics of BA and FA

Characteristics	BA	FA
Absolute density (g/cm³)	3.03	2.15
Apparent density (g/cm³)	0.70	0.30
Moisture content (%)	2.50	9.10

Unburnt rate (%)	2.64	1.05
Blaine specific surface (cm^2/g)	9920	16826

3.2. Chemical Characterization

The results of the chemical analyses of BA and FA are presented in Table 6 and Table 7. The pH of the leachates obtained from BA and FA is alkaline, ranging between 8 and 11. This alkalinity is attributed to the presence of portlandite ($Ca(OH)_2$), a hydration product of quicklime (CaO). The higher CaO content in BA relative to FA likely accounts for the higher pH observed in BA. The concentrations of sulfate ions (SO_4^{2-}) in BA and FA are well below the maximum admissible limit for SO_4^{2-} (10,000 mg/kg) specified in the circular of May 9, 1994, concerning the disposal of incineration bottom ash. Similarly, the chloride contents in BA and FA remain below 800 mg/kg, which corresponds to the maximum allowable limit for inert waste established by the European Landfill Directive 1999/31/EC of April 26, 1999. These results indicate that the investigated residues meet the regulatory requirements for recovery.

Table 6: Physical-chemical parameters of BA and FA

Characteristics	BA	FA
pH	10.60	8.50
Chlorides concentration (mg/kg)	550.50	340.80
Sulfates concentration (mg/kg)	762.50	281.25

Table 7: Elemental chemical composition of BA and FA (XRF)

Element (%)	BA	FA
SiO_2	34.02	49.73
Al_2O_3	19.55	18.75
Fe_2O_3	7.11	15.03
CaO	25.69	4.50
MgO	2.90	1.38
MnO	0.05	0.05
K_2O	2.46	0.75
P_2O_5	0.42	1.93
TiO_2	2.90	4.65
SO_3	0.85	0.65
Na_2O	0.74	1.39
Loss on Ignition (LOI)	2.64	1.05
Total	99.28	99.81

3.3. Mineralogical Characterization

The X-ray diffractograms of BA and FA are presented in Figure 12 and Figure 13, respectively. The main phases detected in both BA and FA are silicates such as quartz (SiO_2), which is present in large quantities in FA. This high quartz content may obscure the detection of minor phases. Hematite (Fe_2O_3) and carbonates, such as calcite ($CaCO_3$), are also identified in the residues. Calcite is more abundant in BA due to the carbonation of lime, according to Reaction 1: $CaO + CO_2 \rightarrow CaCO_3$ (Reaction 1)

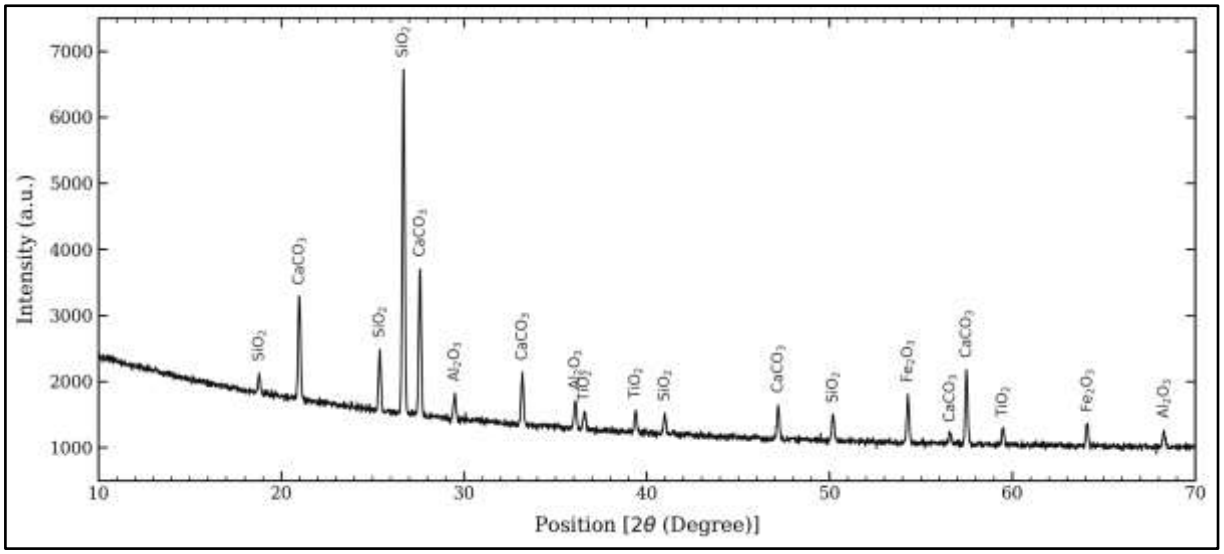


Fig. 12 X-ray diffractogram of BA

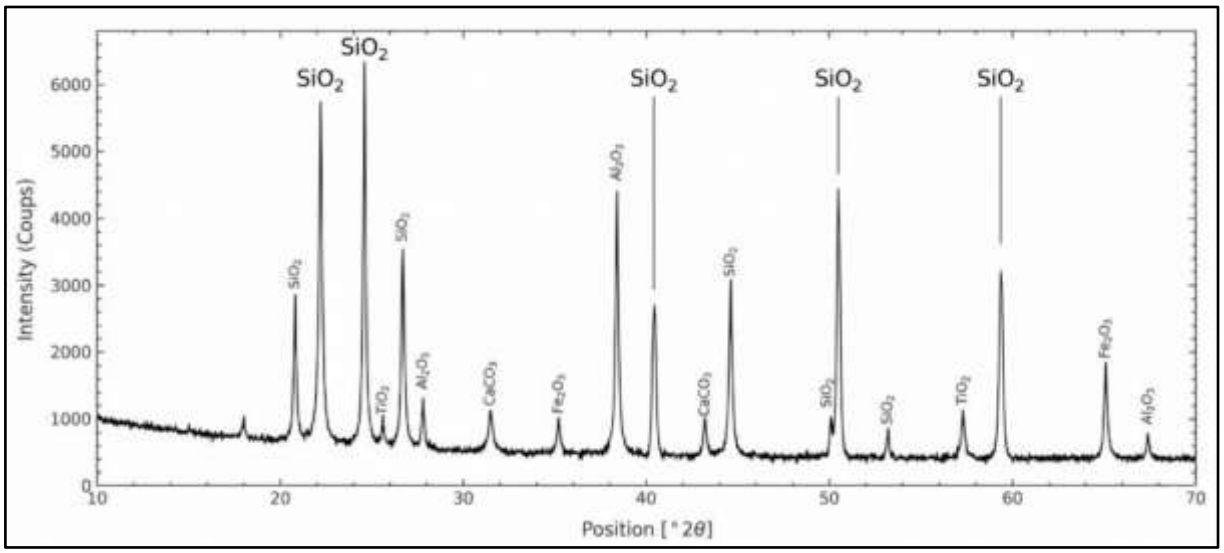


Fig. 13 X-ray Diffractogram of FA

3.4. Enironmental Assessment

As reported in Table 8, the concentrations of elements leached from both BA and FA remain strictly below the inert waste disposal thresholds defined by the European Council Directive 1999/31/EC. These values also satisfy the "recoverable" category limits of the 05/09/1994 circular (Pb ≤ 10 ppm, Cd ≤ 1 ppm, Cr ≤ 1.5 ppm, Hg ≤ 0.2 ppm, and As ≤ 2 ppm). Consequently, BA and FA behave as inert materials, ensuring safe handling for subsequent reuse. Our observations match the mechanism described by Wang et al. [15], where embedding incineration ash into ecological mortars physically encapsulates and chemically binds hazardous heavy metals within the hydration products

Table 8: Heavy metal concentrations in BA and FA leachates

Heavy metals (ppm)	BA	FA	Inert waste threshold(a)	Recoverable waste threshold(b)
Ni (nickel)	0.0012	0.0014	0.40	/
Zn (zinc)	0.0574	0.1484	4.00	/
Fe (iron)	0.0020	0.0004	/	/
Cu (copper)	0.0153	0.0393	2.00	/
Pb (lead)	0.0043	0.0094	0.50	10.00
Cd (cadmium)	0.0002	DL	0.04	1.00
Cr (chromium)	0.0169	0.1106	0.50	1.50
Co (cobalt)	0.0011	0.0015	/	/
Hg (mercury)	ND	ND	/	0.20
As (arsenic)	ND	ND	0.50	2.00

Note: DL = 0.0001 ppm (Limit of Detection). ND = Not Detected. / = No limit specified.
(a) Standards of European Directive 1999/31/EC of 26/04/1999.
(b) Circular of 05/09/1994 relating to the disposal of incineration bottom ash.

3.5. Flexural and Compressive Strengths

Figures 14 and 15 illustrate the evolution of the flexural strength of mortars with different percentages of BA and FA at 7, 14, and 28 days, respectively.

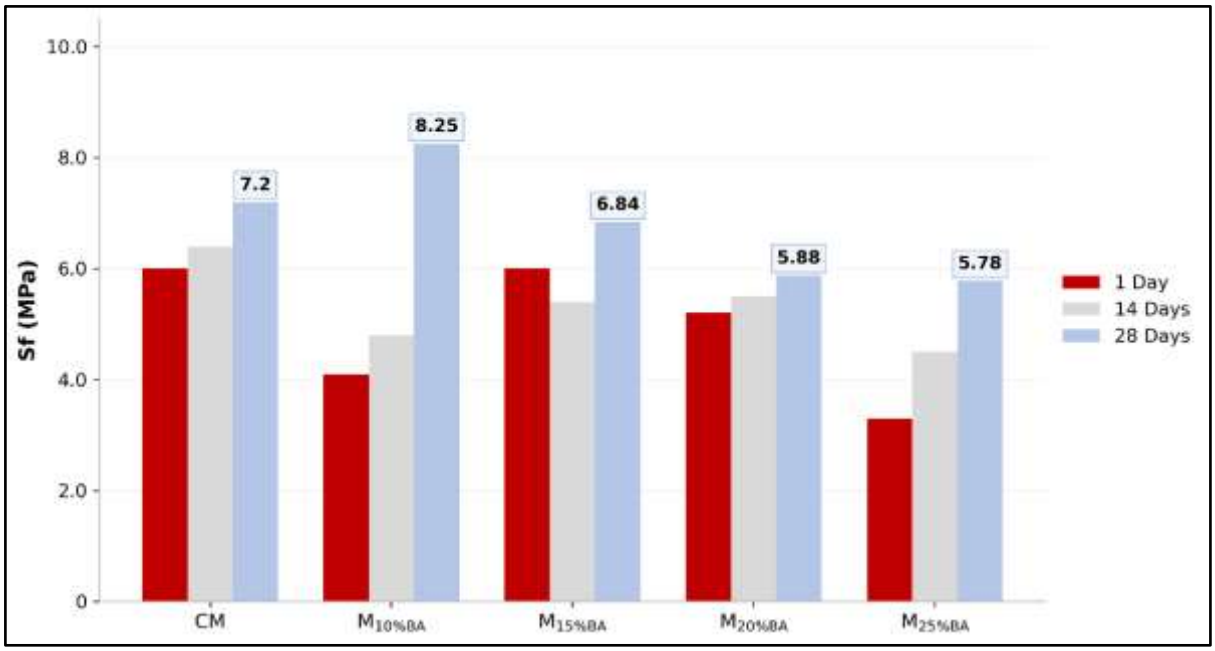


Fig. 14 Flexural strengths (Sf) of BA based mortars

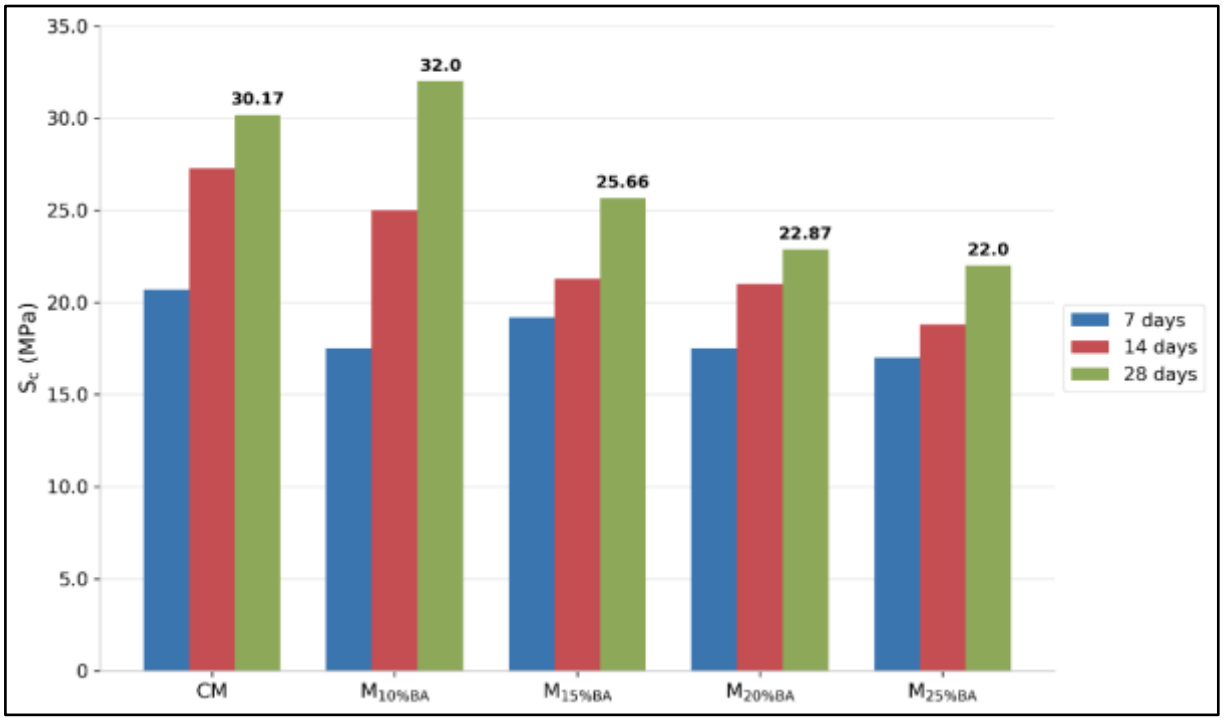


Fig. 15 Flexural strengths (Sf) of FA based mortars

Figures 16 and 17 illustrate the evolution of the compressive strength of mortars with different percentages of BA and FA at 7, 14, and 28 days, respectively.

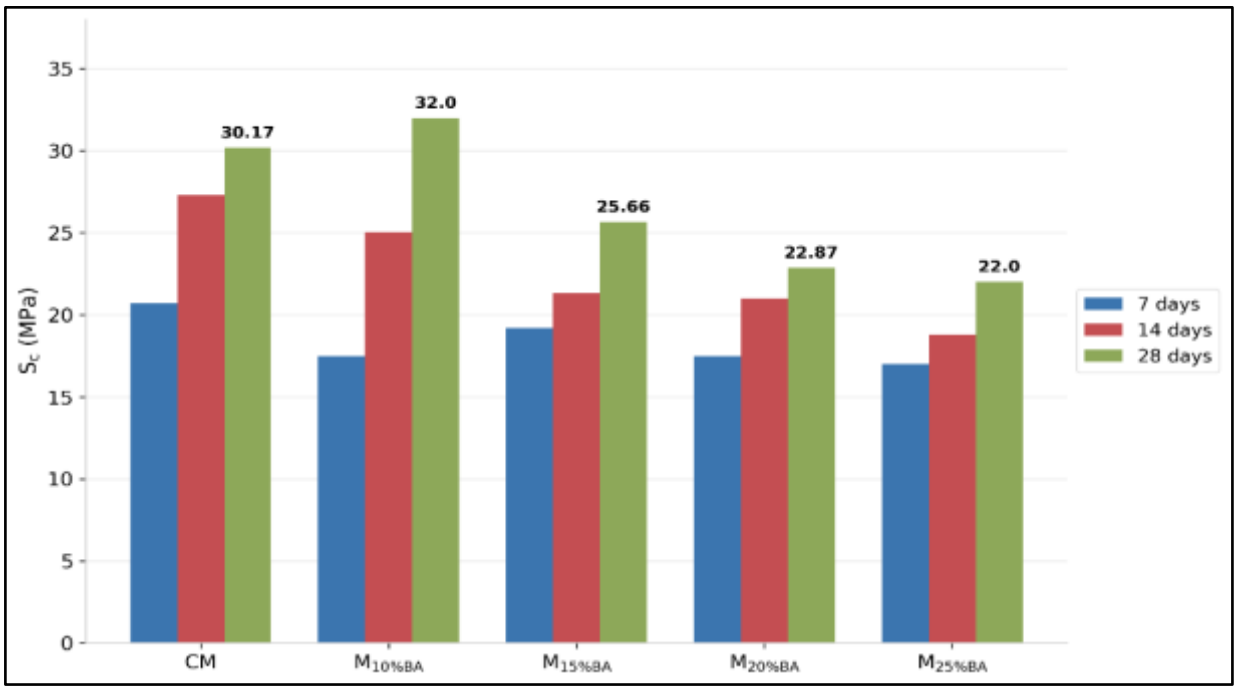


Fig. 16 Compressive strengths (Sc) of BA based mortars

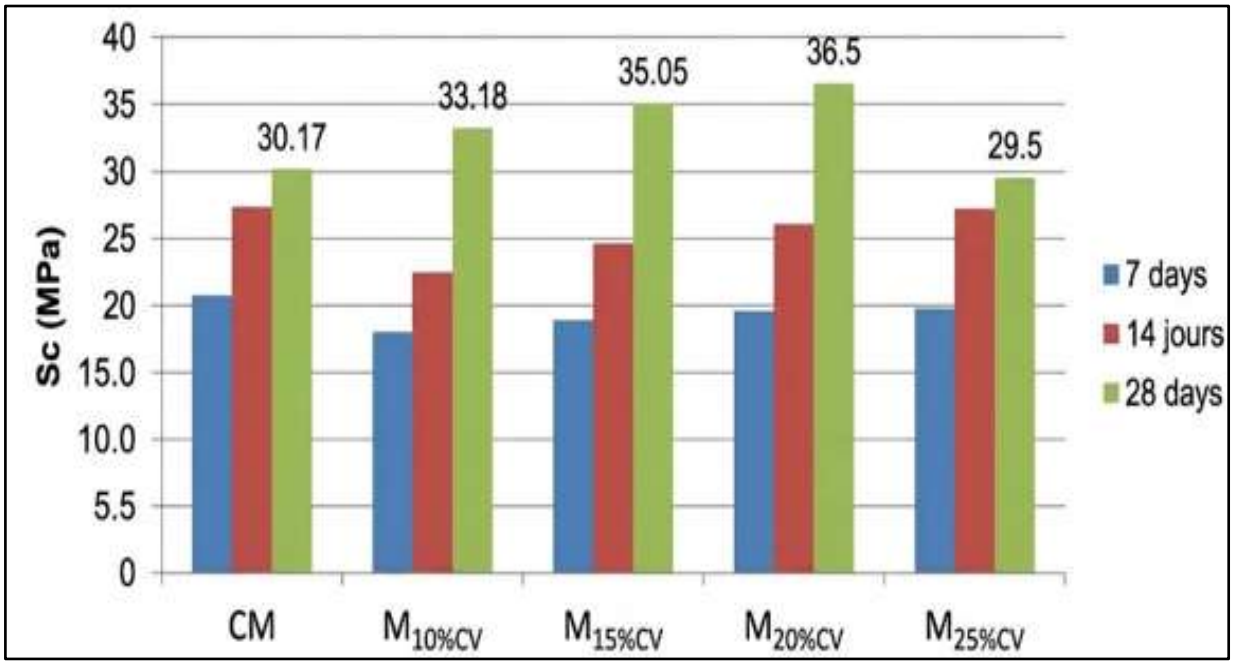


Fig. 17 Compressive strengths (Sc) of FA based mortars

3.6. Pozzolanic Activity Indices of Mortars

Tables 9 and 10 summarize the 28-day pozzolanic activity indices for different cement replacement levels. At 28 days, mortars with 10% BA and 20% FA achieved activity indices of 106.06% and 120.98%, respectively. Since the sum of Al_2O_3, SiO_2, and Fe_2O_3 oxides reached 83.51% for FA compared to 60.68% for BA, FA possesses a more pronounced pozzolanic activity.

The compressive and flexural strengths of all mortar specimens studied at different addition levels increase with age. The compressive strengths of mortar specimens containing BA and FA at 7 and 14 days of curing are consistently lower than those of corresponding control mortars. This reduction in compressive strength can be explained by the interaction between reactive silica and the portlandite released during the hydration of the CPA cement. It gives BA and FA the ability to bind lime. This reaction, known as the pozzolanic reaction, is predominant at early ages (7 and 14 days), leading to a reduction in the hydration rate and consequently to lower strength (i.e., a set-retarding effect).

At later age (at 28 days of curing), compressive and flexural strengths are significantly improved. Mortar specimens containing 10% BA and 20% FA develop strengths comparable to, and sometimes higher than, those of the control mortar, namely 32 MPa for M10%BA and 36.5 MPa for M20%FA. This reflects the long-term physical and chemical effects of BA and FA additions. The incorporation of BA above 10% and FA above 20% affects the workability of mortars, making the fresh mix less workable. This can be explained by the increased water demand during the initial hydration phase. This phenomenon leads to a decrease in both flexural and compressive strengths.

Table 9: Pozzolanic activity index of BA-based mortars at 28 days

Mortar formulation	Activity index, I (%)
M10%BA	106.06
M15%BA	85.05
M20%BA	75.10
M25%BA	73.00

Table 10: Pozzolanic activity index of FA-based mortars at 28 days

Mortar formulation	Activity index, I (%)
M10%FA	109.97
M15%FA	116.17
M20%FA	120.98
M25%FA	97.77

3.7. Resistance to Acid Attack

The durability against acid attack was assessed using a control mortar and on mortars incorporating BA and FA that demonstrated the highest mechanical resistance. The selected compositions correspond to mortars containing 10% BA and 20% FA. Figures 18 and 19 illustrate the evolution of mass loss in hydrochloric acid and sulfuric acid solutions, respectively, at curing ages of 7, 14, and 28 days.

After 28 days of immersion in the two acid solutions, mortars based on 20% FA recorded the lowest rate of mass loss compared to the control mortar and mortars containing 10% BA. This is due to the significant pozzolanic effect of fly ash, which reacts to fix the released lime, thereby reducing the capillary pores through the formation of C-S-H gels, thus limiting the absorption of the acid solution.

The effect of fly ash in the cementitious matrix strengthens its chemical resistance to acid attack, resulting in a reduction in mass loss for mortars containing 20% fly ash compared to other mortars. The mass loss observed for all mortars in both acid solutions is due to the fact that, after hydration, cement releases a considerable amount of free calcium hydroxide, which can be leached out. The attack mechanisms follow Reaction 2 and Reaction 3:

$$Ca(OH)_2 + 2HCl \rightarrow CaCl_2 + 2H_2O \quad \text{(Reaction 2)}$$
$$Ca(OH)_2 + H_2SO_4 \rightarrow CaSO_4 + 2H_2O \quad \text{(Reaction 3)}$$

The results also show that hydrochloric acid is more severe than sulfuric acid. In fact, the calcium chlorides formed are highly soluble salts in water and very aggressive towards Portland cement. However, gypsum reacts with non-hydrated tricalcium aluminate (C_3A) to form ettringite, according to Reaction 4. Gypsum and ettringite cause internal expansion, which leads to cracking of the mortars.

$$3CaSO_4 \cdot 2H_2O + 3CaO \cdot Al_2O_3 + 26H_2O \rightarrow (CaO)_3 \cdot (Al_2O_3) \cdot (CaSO_4)_3 \cdot 32H_2O \quad \text{(Reaction 4)}$$

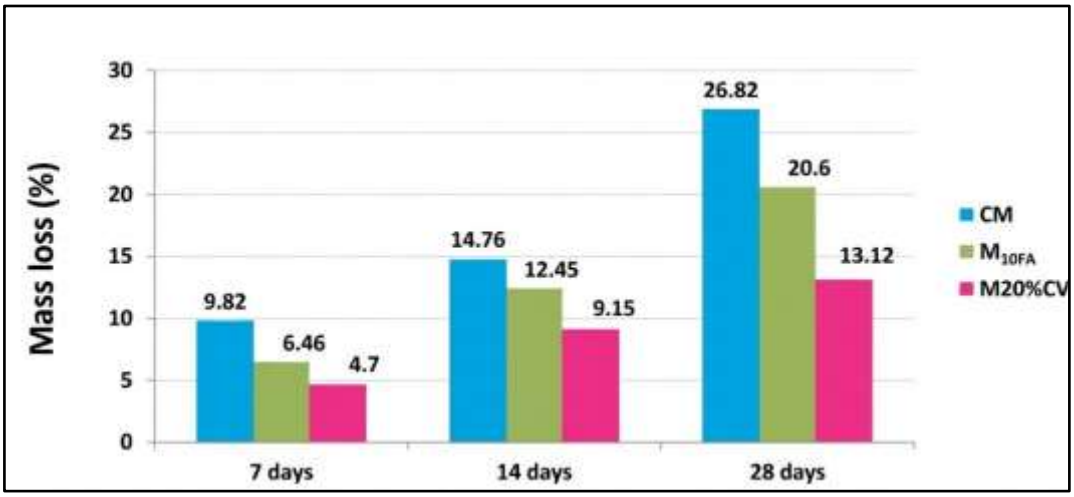


Fig. 18 Evolution of the mass loss of mortars in a 5% HCl solution

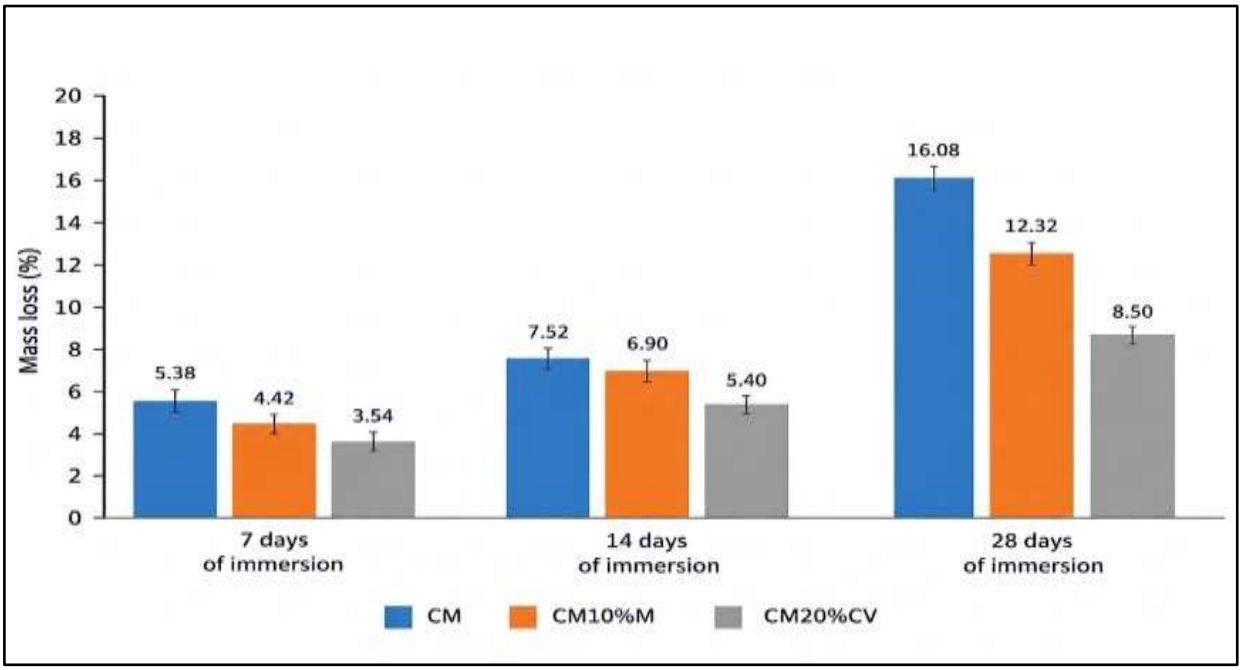


Fig. 19 Evolution of the mass loss of mortars in a 5% H_2SO_4 solution

4. CONCLUSION

This work evaluated the potential of utilizing bottom ash (BA) and fly ash (FA) from expired medicine incineration as partial replacements for Portland cement in mortars. The experimental data confirms that both by-products qualify as inert and recoverable waste materials. Mortars formulated with 10% BA and 20% FA exhibited higher long-term flexural and compressive strengths than the control mix, confirming the combined physical and chemical effects of these additions.

The incorporation of BA and FA as pozzolanic additives helps protect the cementitious matrix against deterioration under acid attack by promoting the formation of a denser and more compact cement paste. This effect is manifested through the fixation of $Ca(OH)_2$, the reduction in capillary pore size, and the decreased permeability of the mortars. Finally, the results show that the BA and FA residues studied in the present work can be used as supplementary materials to Portland cement, with optimal replacement levels of 10% for BA and 20% for FA.

Declarations

Conflict of Interest: The authors declare that they have no conflicts of interest.

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