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Developing Low Cost Eco-friendly Restoration Mortars for Historic Lime-based Stucco and Building Materials

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Abstract

In this work, three formulations based on hydrated lime and eco-friendly additives of powdered brick, fly ash and silica fume were designed to improve repair mortars for historic lime-based stucco and building materials. The microscopic features, physical-mechanical behavior and the microstructure of the prepared mortars were evaluated before and after artificial ageing (by humidity/drying cycles and salt weathering). However, a significant mechanical enhancement was reported for the studied mixes, but the silica fume mix showed a notable failure after salt ageing. The fly ash mix revealed the highest bulk density ratio (1.172 g/cm^3) compared to the lime and silica fume mixes. The silica fume mix recorded the lowest percentage of water absorption (35.56%) and apparent porosity (28.11%). Further, the silica fume mix yielded the highest dry compressive strength value (22.19 kg/cm^2), with an increase reached 31% when compared to the standard lime mix. The results demonstrated that the fly ash mortar is more compatible for sustainable restoration procedures of historic lime-based structures in respect of the physical-mechanical properties.

Keywords

historic lime-based stucco, eco-friendly repair mortars, FE-SEM, compressive strength, salt ageing

1 Introduction

Mortar is a mixture of binding material and aggregates in addition to a required quantity of water used to produce a workable paste for several purposes in buildings. Practically, the desired function of mortar affects its chemical composition and properties [1, 2]. In this direction, many requirements should be met in the restoration mortars of historic buildings such as: reversibility, compatibility, durability and aesthetic characteristics [3].

When applying a restoration mortar for completing and re-pointing historic stucco, compatibility should be considered. For this, a matching between the mechanical, chemical and physical properties of the historic mortars with the restoration mortars is required [4].

Lime mortars have been used since ancient times, but by the application of the Portland cement at the beginning of the twentieth century, the use of lime was obviously limited. In the last decades, the importance of lime has been increased for restoration of historical buildings.

The use of lime has accompanied some challenges associated to the experience of traditional workers and the application procedures of lime. Accordingly, several attempts have been achieved to revive these techniques through scientific and practical applications [5–7].

Further, the low resistance of ordinary lime to moisture, its low mechanical strength and the long setting are further negatives against its extensive application. For this, hydraulic binders and pozzolanic materials are added to lime to enhance its physical-mechanical properties and workability in addition to the compatibility with the historic mortars. These additives allow an excellent improvement to the durability of lime mortars against the environmental impacts [8].

Previous studies have reported that the application of incompatible restoration mortars with historic lime-based structures will cause undesirable damage, likely due to salt crystallization, their low permeability and the high strength. Besides, lime mortars need a long curing time in addition to their low strengths and durability [9–13]. As a consequence, using durable, eco-friendly and compatible restoration mortar is a necessity.

Based on the mentioned above, the present research aims to develop some repair mortars using additives of powdered brick, fly ash and silica fume for restoration

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purposes of historic lime-based stucco and building materials. The proposed mortars were tested with respect to morphological, physical and mechanical properties in addition to evaluate the effect of accelerated artificial ageing on their stability.

2 The used additives with the lime formulations

An evidence on the use of burned clayish additives used with lime mortars has been found since about 3000 BC. But its extensive use as a pozzolanic additive was documented throughout the Roman Empire [14]. Aluminosilicates additives can be in the form of metakaolin, which is resulting from the calcination of kaolinite-rich clay between 450 and 800 °C. Metakaolin is reacting with the slaked lime and water to produce calcium silicates and/or calcium aluminates. According to the country of origin, these additives are called: *Horasan*, *Surkhi*, *Homra* and *Cocciopesto* [15, 16].

2.1 Powdered fired brick

The addition of brick dust accelerates the setting time of lime mortar and produces hydraulic compounds that increase the mechanical strength. More, the application of brick dust enhances the water repellency of lime mortars [17, 18]. Factually, the high amount of reactive silica resulted from the brick powder increases the strength values of mortars [19]. The pozzolanic activity of brick powder depends on many factors such as the glassy phase content, silica content, specific surface area, grain size and the quality of raw materials [20].

Well, the use of brick fragments or dust allows the recycling of wasteful quantities near brick factories and the buildings under construction. So, the producing of repair mortars with brick powder additives will provide eco-friendly mortars as an acceptable alternative to the Portland cement for restoration of historical buildings [21].

2.2 Fly ash

Fly ash (FA) is a by-product derived from the industry of coal combustion and it is characterized by its good crystallinity [22]. Actually, fly ash had been used in Roman monuments in the form of volcanic ash, due to the high similarity in their chemical properties [23]. Recently, the addition of fly ash to mortars has become a trend in the construction field.

Fly ash is consisting mainly of clay-sized glassy micro spheres made of alumina silicate pozzolan. It was used for producing eco-friendly geopolymers through mixing with lime and Portland cement binders [24–26]. The pozzolanic materials contained in the fly ash are hardened after activation with an alkaline reagent. After polymerization, the formed gel offers many advantageous mechanical and physical characteristics to lime mortars [27, 28].

2.3 Silica fume

Silica fume consists of spheres of amorphous SiO_2 and it is resulted from the production of metallurgical silicon and ferrosilicon alloys. The particles size ranges from 0.1 to 0.2 µm. Silica fume is a highly reactive pozzolanic material that improves the workability and strength of the hardened mortars. In recent years, the use of silica fume from polluting waste-products has been applied as a valuable addition for mortars [29].

3 Materials

The used binders and additives are given in Table 1. The proportions were applied according to Saleh [30]. The used materials are:

- Hydrated lime: hydrated lime produced by HAMCO company for building materials (Cairo, Egypt) was used with the following chemical composition: $Ca(OH)_2$ (min. 80%), MgO (max. 0.05%), SO_3 (max. 0.50%), Cl (max. 0.52%), Fe_2O_3 (max. 0.08%), Al_2O_3 (max. 0.01%), Defects (max. 2.5%), particle size (75 micron or less).
- Fly ash: it is a grey and odorless fine powder composes of spherical particles of alumina silicate pozzolan provided by Sika Egypt company with the following chemical composition: SiO_2 (min. 41.11%), Al_2O_3 (max. 22.15%), Fe_2O_3 (max. 15.20%), MgO (max. 2.11%), CaO (max. 9.05%), SO_3 (max. 1.04%), TiO_2 (max. 0.30%), K_2O (max. 1.90%), and Na_2O (max. 0.93%), loss on ignition (7.80%).
- Silica fume: the used silica fume was obtained from the Egyptian chemical Industries (KIMA company, Cairo, Egypt). It composes of: SiO_2 (min. 92%), Al_2O_3 (max. 1.5%), Fe_2O_3 (max. 1.5%), MgO (max. 1.0%), CaO (max. 0.80%), Granulation (+0.045 mm 1.5%),

Table 1 Composition and proportions of the studied mixes

Proposed mixes	Components	Proportions/mass
Lime mix	Hydrated lime: sand	2:3
Fly ash mix	Hydrated lime: powdered brick: fly ash: sand	2: 1½ : ½ : 1½
Silica fume mix	Hydrated lime: powdered brick: silica fume: sand	2: 1½ : ½ : 1½

C (max. 1.5%), K_2O and Na_2O (max. 2.0%), H_2O (max. 1.0%), loss on ignition at 750 °C (max. 1.5%) color (whitish grey).

- Powdered brick: the remains of fired bricks were collected, then they were milled and sieved to have the brick dust which was added to the proposed mixtures. The following chemical composition was reported: SiO_2 (min. 52.21%), Al_2O_3 (max. 16.07%), Fe_2O_3 (max. 5.4%), MgO (max. 5.4%), CaO (max. 9.8%), SO_3 (max. 1.18%), TiO_2 (max. 0.80%), K_2O (max. 2.6%), and Na_2O (max. 1.7%), loss on ignition (6.30%).
- Sand: the used sand was sieved and washed several times with distilled water to remove any impurities.

3.1 Preparation of the formulations

The dry ingredients were mixed together until the mixture was completely become homogeneous, then the required amount of water was added (water: dry mix ratio was 1:3). Then, the mixture was casted into silicon molds (30 × 30 × 30 mm). The specimens were kept inside the casts for one day. After being removed from the molds, they were cured for four months at 20 ± 2 °C and 65 ± 5% RH (Fig. 1).

4 Methods and measurements

The chemical composition of raw materials was determined by a "Phillips PW2400" X-ray fluorescence (XRF) spectrometer. The microscopic features of mortars were collected using a handheld USB digital microscope (model PZ01, made by Shenzhen Supereyes Co. Ltd, China).

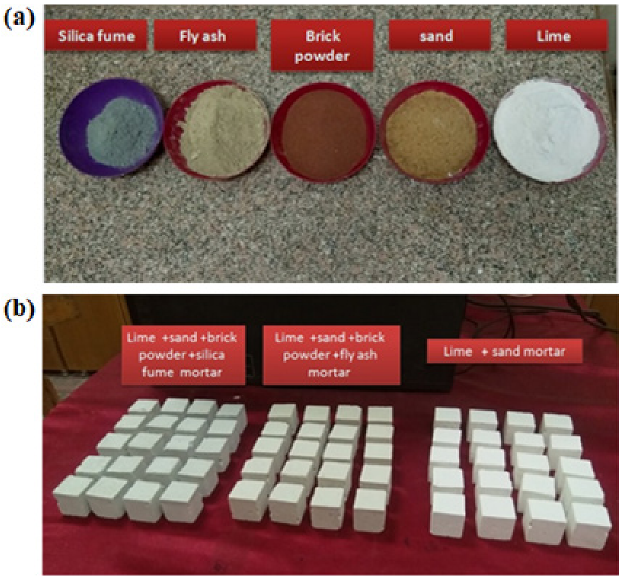


Fig. 1 The raw materials and the proposed mortar cubes

The physical properties of the mixes (bulk density, apparent porosity and water absorption ratio) were measured after four months of curing. The physical properties of the proposed mixes were determined in accordance with ASTM C127-15 [31]. These laboratory tests as well the compressive strength were selected to assess the basic physical and mechanical characteristics of the proposed mixes needed to evaluate their compatibility with historic mortars [32, 33].

The compressive strength of mortar mixes was measured according to ASTM C170/C170M-17 [34] with some adjustments due to the nature of used mortars. Zwick Roell device was used to evaluate the compressive strength of samples with load cell: 10 kN with extensometer: speed 0.5 mm/min. An average of five samples of each mortar was measured.

The microstructure of samples and their micro chemical composition were evaluated using a field-emission scanning electron microscope (FE-SEM) (Philips Quanta FEG 250). EDX chemical analysis of samples was performed in a low vacuum chamber at an accelerating voltage of 20 kV.

The combined thermogravimetric-differential thermal analysis (TG/DTA) was applied by (Shimadzu, DTG 60, Japan), using a rate temperature of 10 °C/min, hold temperature = 1000 °C, atmosphere: nitrogen rate flow 20/min.

4.1 Artificial ageing of mortars

Artificial ageing was designed on the basis of the climatic conditions in Egypt (temperatures range between average winter minimums of 14 °C (November to April) and average summer maximums of 30 °C (May to October), while the temperature reaches 43 °C in the inland desert areas).

The tested mortars were immersed in water for 3 hours, then they were left to dry until the samples reached a stable weight. Then the samples were dried in an oven at a temperature of 60°(±5°) for 18 hours. After that, the samples were left in the room temperature for 4 hours. These steps were repeated for 20 cycles [35, 36].

The cycles of salt ageing were carried out using sodium chloride salt (NaCl), according to the RILEM 25-PEM-1980 [37]. The samples were dried at 105° for 22 hours. Next, the samples were weighted after reaching the room temperature, then they were immersed in a 14% NaCl solution for an hour. These steps were repeated for 20 cycles.

5 Results and discussion

5.1 Microscopic examination

The microscopic images recorded on the studied mixes are presented in Fig. 2. Sand grains were obviously detected

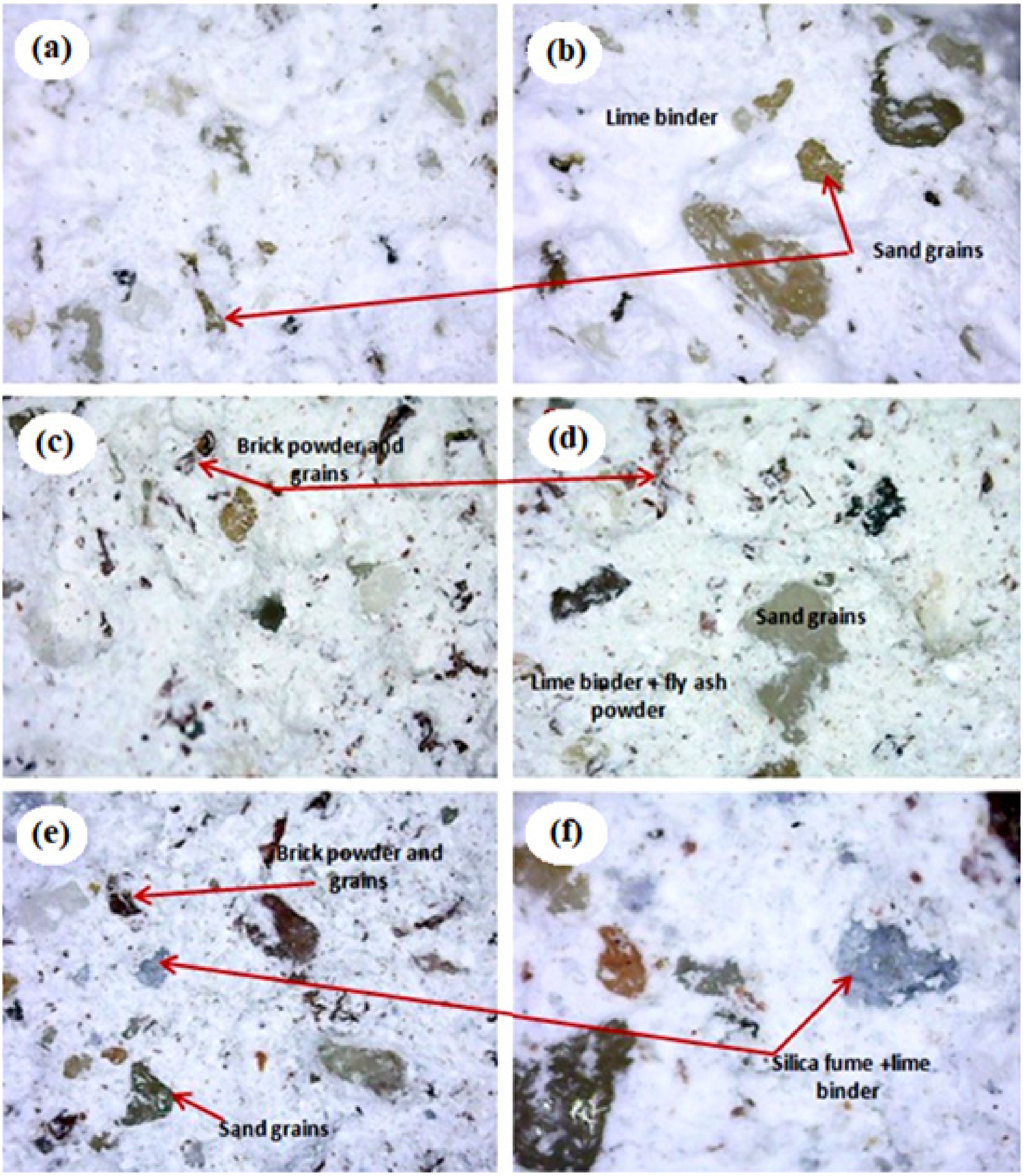


Fig. 2 Microscopic images of the studied mixes: (a) the lime binder after curing, (b) large sand grains within the lime mix, (c) unreacted brick grains within the fly ash mix, (d) large sand grains in the fly ash matrix, (e) brick and sand grains in the silica fume mix, (f) large unreacted silica fume particles

in the three mixes. As well, brick grains and powder were detected in the images of fly ash and silica fume mixes. The observations showed that the fly ash powder is well mixed with lime, while some gray lumps are appeared in the silica fume mix.

5.2 Physical-mechanical properties

The results of the physical properties measured on the studied mortars are clarified in Fig. 3. Bulk density, water absorption ratio and the apparent porosity affect the mortars durability and weathering resistance [38]. It was

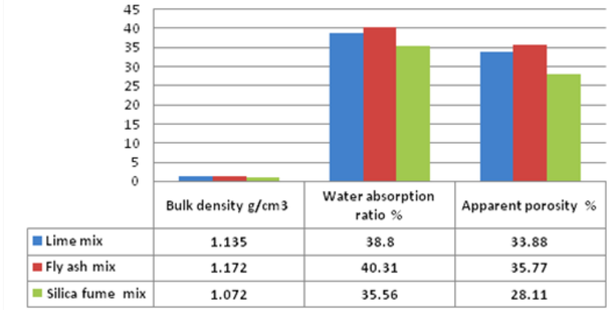


Fig. 3 Results of the physical properties of the mortar mixes

observed that the fly ash mortars showed the highest bulk density ratio (1.172 g/cm^3).

While the silica fume mix recorded the lowest water absorption (35.56%) and apparent porosity (28.11%). The values of water absorption and porosity recorded on fly ash mix showed slightly higher percentages than those of the reference samples. Actually, these results cannot be counted as a serious issue since they may express the physical compatibility of the studied mortars with the historic ones.

According to Giandomenico et al. [39], the porosity is highly influenced by the porous lime content of mortars. However, the mechanical properties are improved due to the few voids at the matrix-aggregates interface in addition to the high air permeability which enhances the carbonation process. The average range of porosity in historic lime mortars is between 26–30% [40] and up to 40% in some structures [41]. Well, the application of durable permeable repair mortars are required in case of highly porous structures.

The compressive strength results of the proposed mixes revealed that the silica fume mix yielded the highest dry/wet compressive strength values, 22.19 and 22.05 kg/cm^2, respectively. However, the values reported on the fly ash mix confirm that it is more compatible as repair mortar (Table 2).

After the immersion in water for 3 hours, the compressive strength results of the three mixes revealed their high resistance towards wetting. The fly ash mix decreased the strength when compared to silica fume probably due to its fine powder [42]. Likely, the quantity of aggregates could also have an effect on the mechanical strength of mortars. But this resistance was decreased after 24 hours of immersion in water, especially for the silica fume mix.

Actually, adding fly ash to lime mortars increases their strength; as the reaction of calcium hydroxide with the fly ash constituents produces several phases such as calcium silicate, calcium aluminates and calcium ferrate which improve the physical and mechanical characteristics of mortars [27]. It was observed that mixing silica fume with lime (15% by weight) increases distinctively the compressive, tensile and shear strength of mortars [29].

5.3 FE-SEM investigation

FE-SEM micrographs provided sufficient information on the binder matrix and the distribution of small grains within the matrix (Fig. 4 (a)).

The detection of smooth and spherical fly ash particles suggests that their hydration reaction with lime and powdered brick was not complete even after 4 months of curing (Fig. 4 (b)). The observed microstructure in the mentioned micrograph reflects the slow reactivity of fly ash with lime when compared to the silica fume additive [43]. While the disappearing of spherical unreacted silica in addition to the detection of new elongated crystals justify that the silica fume particles were completely reacted (Fig. 4 (c)). This probably explains the high compressive strength reported for the silica fume mix [44].

EDX analysis of the lime mix sample (Fig. 5 (a)) revealed the following elements: calcium (Ca), carbon (C), oxygen (O), silicon (Si), which give an indication on the occurrence of calcite and quartz.

EDX spectra of the fly ash and silica fume mix (Fig. 5 (b), (c)) showed an increase in the silica content compared to the lime mix. This result reflects the hydration reaction of silica fume and fly ash pozzolanic products with lime. Specifically, this was the responsible for enhancing the mechanical strength of these two mixes [45]. The detection of iron (Fe) is associated to the components of the powdered brick additive. Based on the morphological observations of the studied samples, the reactivity of fly ash particles was slower than those of silica fume. More, the water released from the chemical reaction leaves behind several pores in the matrix [46–48].

Table 2 Results of the dry and wet compressive strength of mortars

Formulation	Average of dry CS (kg/cm^2)	Average of wet CS (kg/cm^2)		Average of dry CS (kg/cm^2), after ageing
		(3 h immersion)	(24 h immersion)	
Lime	11.77	12.25	9.36	10.08
Fly ash	15.6	13.74	9.59	12.12
Silica fume	22.19	22.05	10.38	16.75

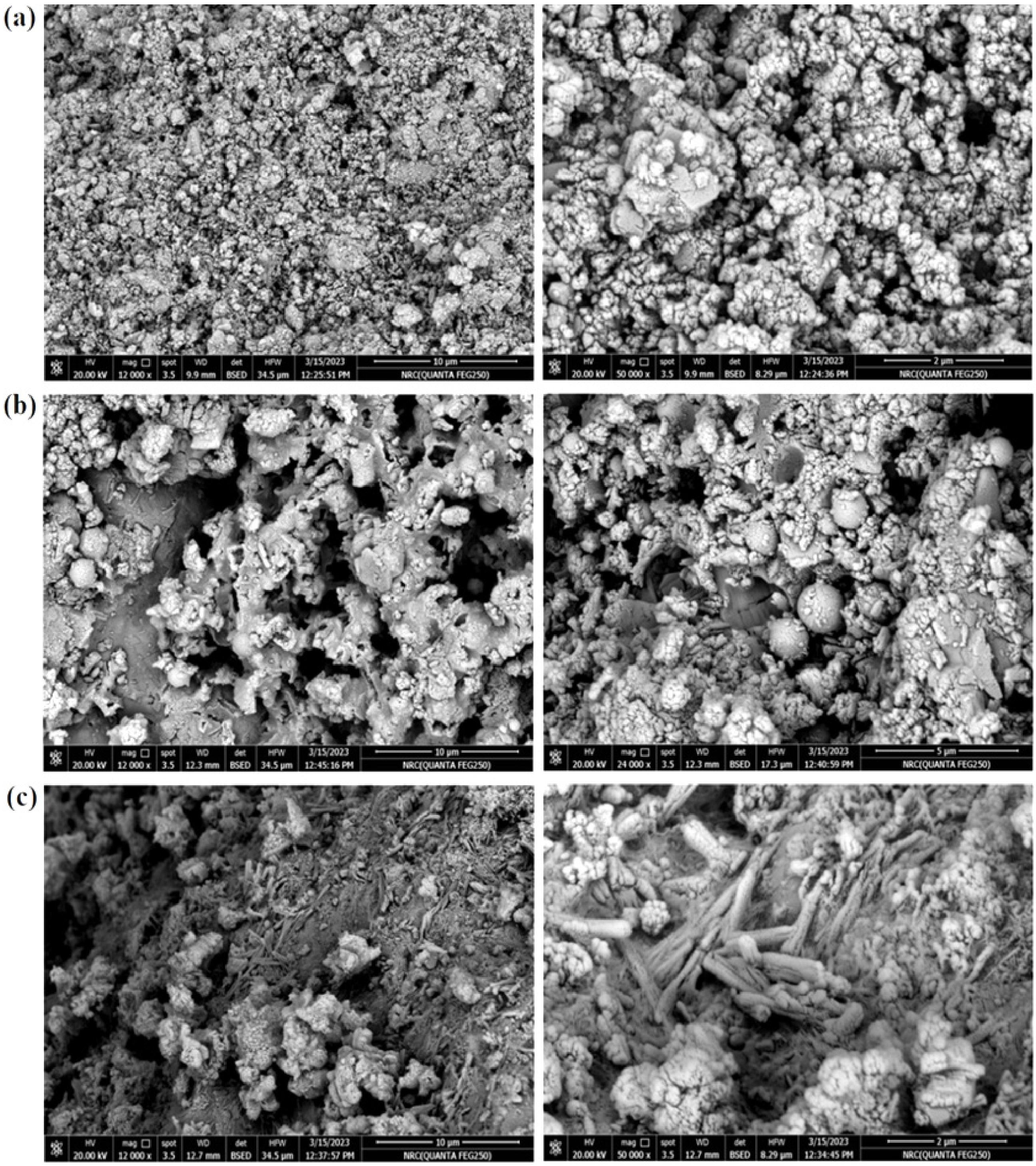


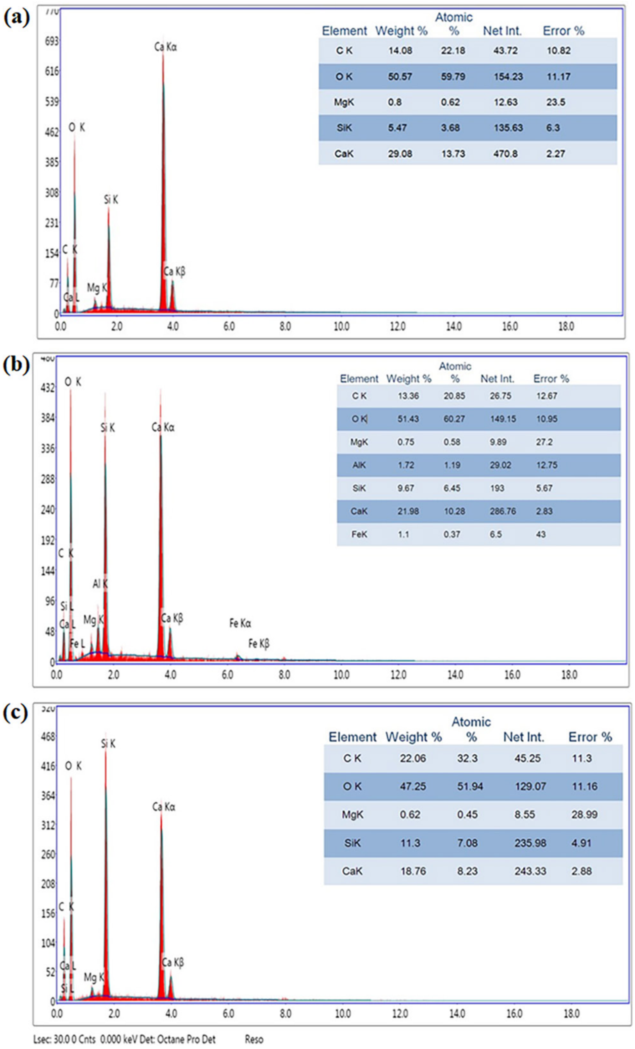
Fig. 4 FE-SEM micrographs of the studied mortars: (a) lime, (b) fly ash, (c) silica fume

5.4 TG/DTA results

When combining the chemical composition of the three mixes with the TG/DTA curves, some results can be concluded. The differential thermal analysis (DTA) of the lime mix (Fig. 6 (a)) is characterized by a peak at about 688 °C which reflects the decomposition of calcite according to the following equation: $CaCO_3 \rightarrow CaO + CO_2$.

The thermogravimetric curve (TG) of the lime mix showed a weight loss of about 18% which resulted from the evolution of CO_2 gas [49].

The same peak at about 686 °C was detected in case of the fly ash mix which accompanied with a weight loss of about 16%. No peaks were detected at the dehydration region of the pozzolanic products which may confirm the



Element	Weight %	Atomic %	Net Int.	Error %
C K	14.08	22.18	43.72	10.82
O K	50.57	59.79	154.23	11.17
MgK	0.8	0.62	12.63	23.5
SiK	5.47	3.68	135.63	6.3
CaK	29.08	13.73	470.8	2.27

Element	Weight %	Atomic %	Net Int.	Error %
C K	13.36	20.85	26.75	12.67
O K	51.43	60.27	149.15	10.95
MgK	0.75	0.58	9.89	27.2
AlK	1.72	1.19	29.02	12.75
SiK	9.67	6.45	193	5.67
CaK	21.98	10.28	286.76	2.83
FeK	1.1	0.37	6.5	43

Element	Weight %	Atomic %	Net Int.	Error %
C K	22.06	32.3	45.25	11.3
O K	47.25	51.94	129.07	11.16
MgK	0.62	0.45	8.55	28.99
SiK	11.3	7.08	235.98	4.91
CaK	18.76	8.23	243.33	2.88

Fig. 5 EDX spectra of the mortars: (a) lime, (b) fly ash, (c) silica fume

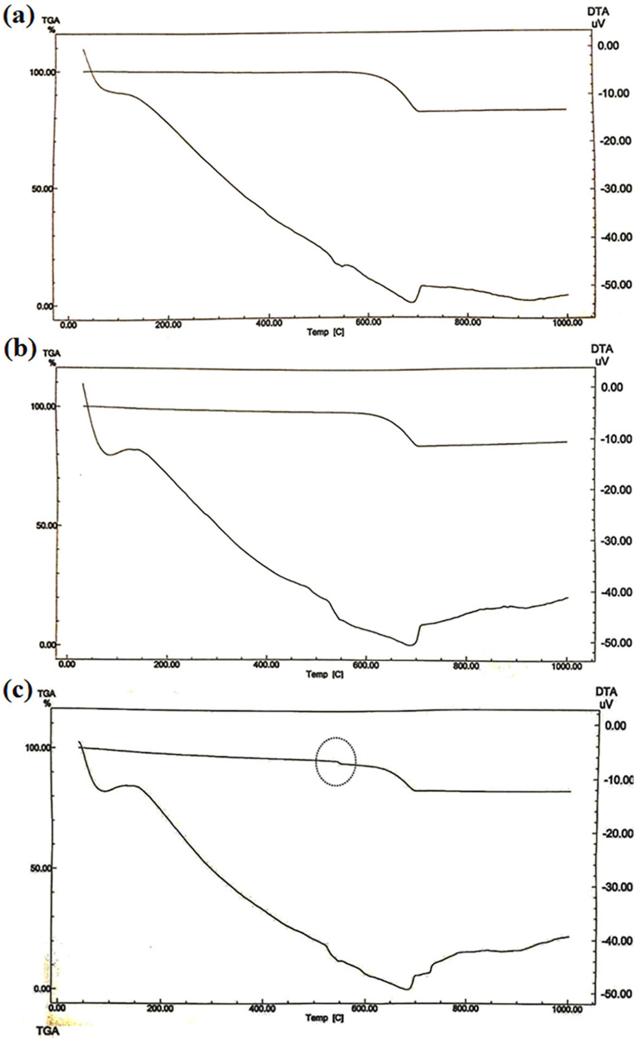


Fig. 6 TG/DTA curves of the mixes: (a) lime, (b) fly ash, (c) silica fume

slow reaction of fly ash and powdered brick with lime (Fig 6 (b)). Actually, these results are in agreement with the FE-SEM observations as previously discussed.

TG/DTA curves of the silica fume mix showed a peak at about 682 °C which is due to the decomposition of calcite while the peak at about 545 °C is associated to the dehydration of the pozzolanic products (Fig. 6 (c)). This suggests that a reaction was occurred between the used additives and lime. It was observed through the FE-SEM micrographs that all the spherical shapes of silica fume have been consumed. This reaction between the pozzolanic additives and lime can result in greatly enhanced durability and strength as previously reported for the studied mortars [15].

5.5 Evaluating the stability after artificial ageing

The visual observations of the studied cubes after artificial ageing showed the stability of both lime and fly ash mixes, while a partial failure was noticed for the silica fume cubes (Fig. 7). No obvious microscopic change was noticed on the proposed mixes after humidity/drying cycles. But many erosions and flakes were noticed on the silica fume samples after salt weathering.

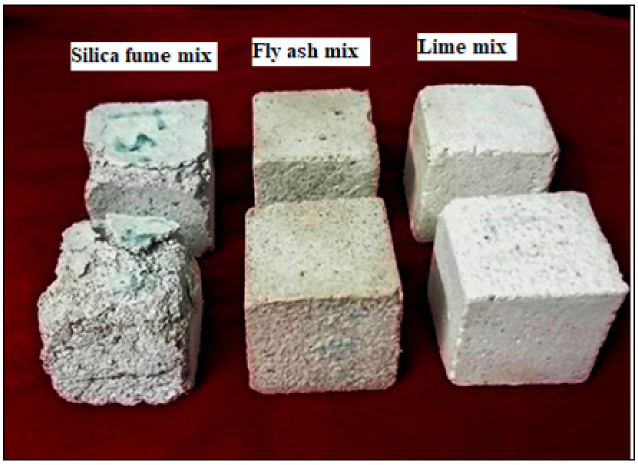


Fig. 7 The studied mortars after the artificial ageing

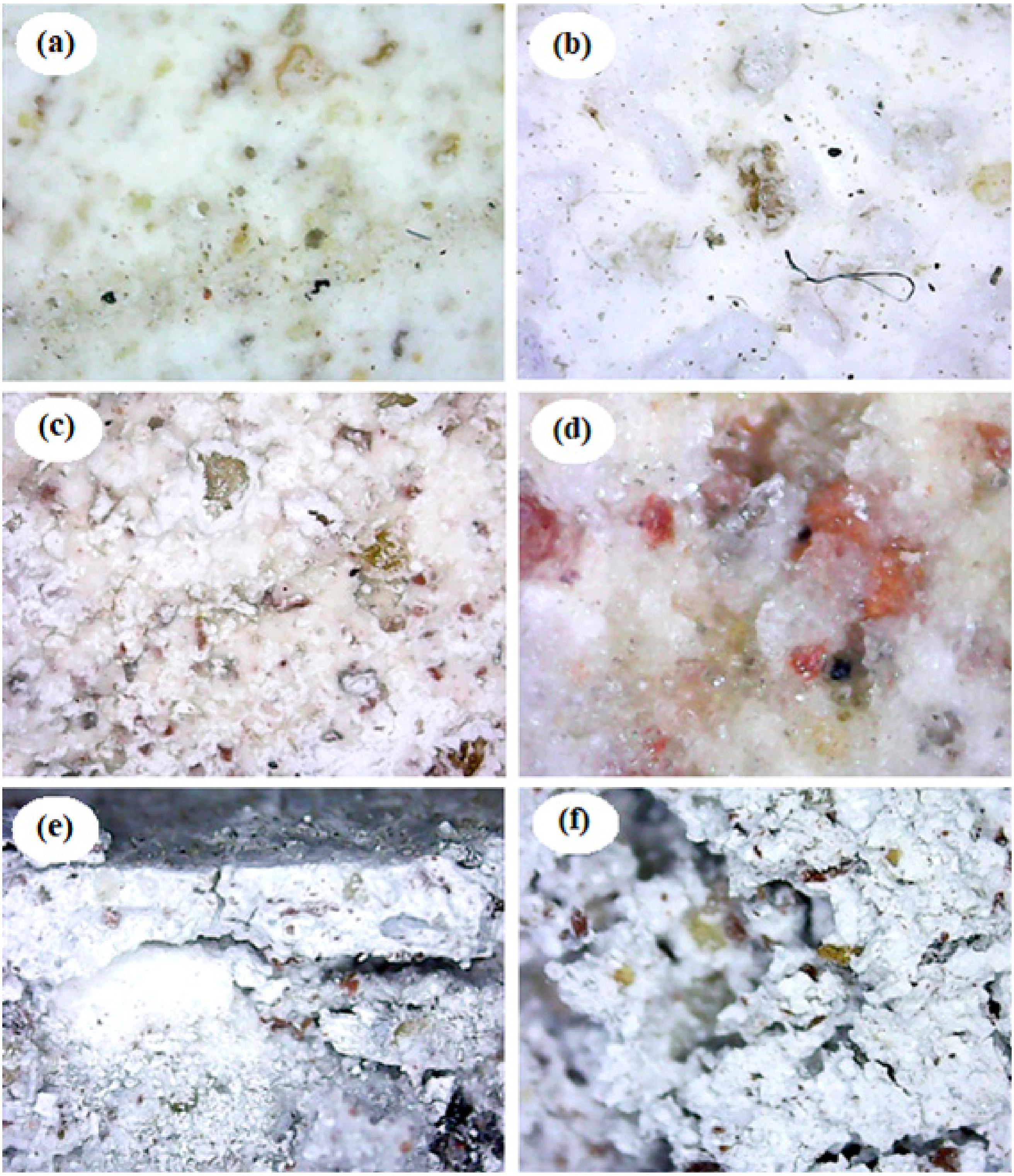


Fig. 8 Microscopic images of the studied mixes after artificial ageing: (a) compacted lime matrix, (b) stability of the lime mix, (c) few etching in the fly ash samples, (d) glassy surface of an aged fly ash mix, (e) several cracks in the silica fume samples, (f) highly etched matrix of silica fume after aging

Conversely, the lime and fly ash mixes showed an obvious durability towards the artificial ageing despite some salt crystals were observed on the surface of the sample (Fig. 8). As shown in Table 2, the compressive strength after humidity/drying cycles showed that the tested mixes achieved acceptable values, especially for lime and fly ash mixes. These results reflect the durability of the fly ash and lime mixes. Despite the acceptable compressive strength of the aged silica fume mortars, serious deterioration aspects on the outer surfaces have been observed.

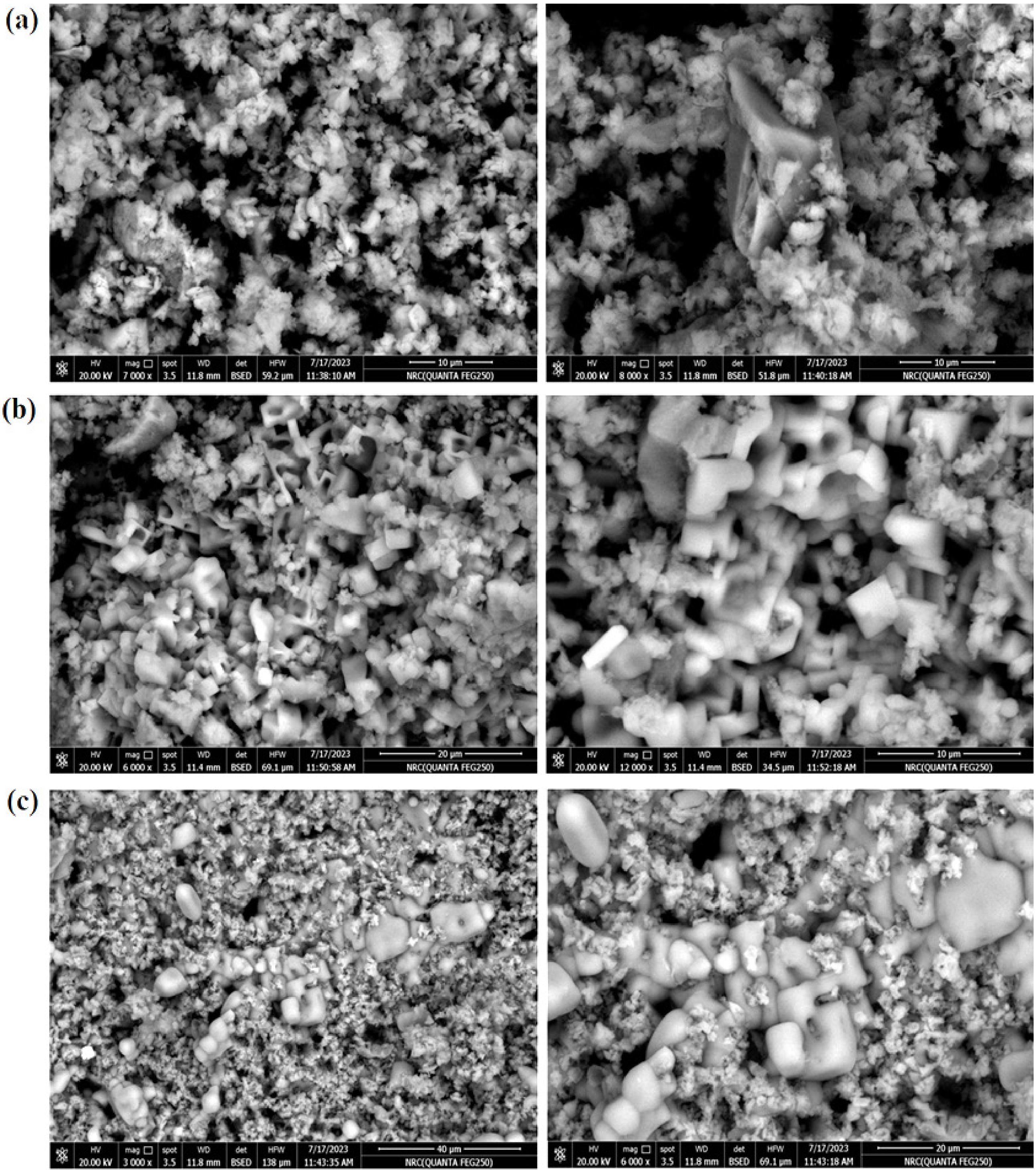


Fig. 9 FE-SEM micrographs of mortars after salt weathering: (a) lime, (b) fly ash, (c) silica fume

The microstructural investigation of the studied mixes after salt weathering is given in Fig. 9. In case of lime mix, a high resistance was observed due to the compacted inner matrix (Fig. 9 (a)). Fluffy salt accumulations were observed on the outer surface without occurring any alteration to the mortar microstructure.

A dense salt coat together with unreacted spheres and crystalline plates of the geopolymer constituents were determined through the FE-SEM images of the fly ash mix (Fig. 9 (b)). Small cavities together with waxy salt coat can be noticed in the silica fume mix (Fig. 9 (c)). Obviously, the salt ageing caused a notable damage to the silica fume mortar while only small itching was occurred in case of lime and fly ash mortars.

6 Conclusions

Based on the obtained observations on the studied mortars, the following conclusions can be drawn:

- The fly ash-based mix showed the highest bulk density ratio when compared to the lime and silica fume mixtures. The silica fume mix recorded the lowest percentage of water absorption and apparent porosity.
- The compressive strength measurements on the three mixes revealed that the silica fume mix yielded the highest values.
- Despite the high compressive strength of the silica fume mortars but a serious damage was documented after salt ageing.
- FE-SEM micrographs on the silica fume mix confirmed its complete reaction with the lime binder. The observations allowed to conclude that a slow reactivity was occurred in case of the fly ash mix.
- EDX analysis on the silica fume mix showed a significant increase in the silica content which reflects the hydration reactions of pozzolanic products with lime.
- TG/DTA analyses on the silica fume mix reflected the complete reaction occurred between the silica fume and lime. While the absence of any peaks in case of the fly ash mix is probably due to its slow reactivity.

To conclude, the physical-mechanical results of the fly ash-based mortar (2 hydrated lime: 1½ powdered brick: ½ fly ash:1½ sand) suggest its consistency as restoration mortar for historic lime structures in the Mediterranean climate environment. A further encourage on the employment of eco-friendly additives to the restoration mortars is highly recommended.

Progressing on to further study on the compatibility of these formulations with different types of historic mortars can help in the practical restoration interventions.

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