



Fly ash-based one-part alkali activated mortars cured at room temperature: Effect of precursor pre-treatments

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ABSTRACT

One-part or “just add water” alkali activated materials (AAMs) have attracted a lot of attention thanks to the use of solid alkaline activators that makes these materials more suitable to commercialization compared to conventional AAMs (two-part). This is mainly because large quantities of caustic solutions should be handled for producing conventional AAMs. So, one-part AAMs have a great potential for *in-situ* applications. However, heat curing (<100 °C) has been demonstrated to be the best condition to obtain optimized performances of one-part AAMs. This study investigates how to obtain high strength one-part alkali mortars cured at room temperature, considering a newly developed mix design, precursor pre-treatments and curing time. The mechanical performances (*i.e.*, elasticity modulus, compressive and flexural strength) of the developed materials were reported, as well as physical properties, in terms of water absorption, open porosity and pore size distribution and microstructure (by means of FEG-SEM observations coupled with elemental analysis by EDS and FT-IR measurements). Class F fly ash have been activated by potassium hydroxide (KOH) and anhydrous sodium metasilicate. It was found that high strength one-part AAMs can be achieved by activating coal fly ash with a mix of KOH and anhydrous sodium metasilicate at room temperature. In particular, room temperature-cured one-part mortars obtained by pre-treated fly ash exhibited mechanical performance similar to those obtained by heat-cured mortars (at 70 °C, tested after 7 days), reaching a compressive strength (R_c) greater than 60 MPa at 28 days of curing when mechanochemical activation of fly ash was applied.

1. Introduction

Since 2000, a lot of work has been focused on alkali activated materials (AAMs), a newly developed material that is generally obtained from an amorphous aluminosilicate powder activated by alkaline and silicate solutions [1,2]. Many studies have been published investigating mix design and different precursors suitable to obtain AAMs, their mechanical performances and durability, and the environmental sustainability of these new systems [3–16]. The development of the mechanical strength of AAMs has been mainly achieved by two different ways: high- or room-temperature curing. Curing samples at temperatures ranging from 30 [17] up to 120 °C [18] allows developing higher strength at early stage of curing. On the contrary, room-temperature curing, more suitable for *in-situ* application, still need to be optimized as efflorescence and lower compressive strengths are often obtained [18]. Indeed, a strong limit for the diffusion of AAMs, especially in the building and construction field, is the need of alkaline solutions, as they shall be freshly prepared, require special attention in handling and must be

stored in large amount in alkaline-resistant tanks.

Only recently, some studies focused on the synthesis of one-part AAMs, simply obtained by mixing precursor and solid activators with water. The most frequent precursors used in one-part AAM synthesis are fly ash (FA), ground-granulated blast-furnace slag (BFS) and clay minerals which, however, require heat treatment to increase their reactivity [19]. Solid alkali activators in one-part mixes are similar to the ones used in traditional AAMs: anhydrous sodium silicates (Na_2SiO_3) and hydroxides (NaOH or KOH). While traditional AAMs have a $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio in the range of approximately 3.3–4.5, it was found that this range is much wider, from 0.75 up to 6.02 for one-part geopolymers. Considering the $\text{Na}_2\text{O}/\text{SiO}_2$ and $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ molar ratios, they vary in the range 0.16–2.92 and 0.98–1.50 for traditional and one-part AAMs, respectively [19].

Generally, FA-based one-part AAMs are mainly prepared by using class F fly ash (according to ASTM C618 standard) and only few attempts have been made using sodium silicate as solid activator. Suwan et al. obtained binder samples by room-temperature curing with a

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compressive strength of 2.4 and 6.0 MPa after 7 and 28 days of curing, respectively [20]. Yang et al. developed mortar samples by room-temperature curing with a compressive strength of 1.5 and 3.5 MPa after 7 and 28 days of curing. Nematollahi et al. prepared mortar samples by 60 °C curing obtaining a compressive strength of 29.3 MPa after 7 days [21]. Askarian et al. reports that mortar samples activated with sodium silicate cured at 60 °C showed a compressive strength of 12.3 MPa after 7 d; the same mix design cured at room temperature exhibited a compressive strength of 7.0 and 10.9 MPa after 7 and 28 days, respectively [22]. Moreover, it is reported that room temperature-cured one-part AAMs obtained by mixing FA and Na-based solid activator was not able to set in 24 h and did not exhibit any strength even after 3 d [22]. Best results have been found by Hajimohammadi et al. with one-part AAM mortar cured at 40 °C showing a compressive strength of 42 MPa after 7 d [23].

From the cited studies, only low mechanical performances have been achieved when room-temperature curing was applied. In addition, one-part geopolymers are mainly studied considering heat curing, thus limiting the potential application fields of these systems where heating is not allowed (e.g., building materials for *in situ* applications, binders, materials for rendering, restoration, etc.). Therefore, mix-design optimization (i.e., selecting the best molar ratios for activation) is urgently needed for one-part AAMs especially when room-temperature curing is applied and high performances in terms of mechanical, microstructural and durability characteristics, are expected.

For all these reasons, the aim of this paper is to investigate how to develop high strength FA-based one-part alkali activated mortars at room temperature and comparing their behavior when heat curing is carried out. The novelty of this research is based on the following issues: (i) a new mix design based on the use of solid KOH so far never tested in one-part geopolymers at the best of our knowledge; (ii) precursor pre-treatments to enhance its reactivity by mechanical ball milling and mechanochemical activation; (iii) comparison of different curing conditions for the same system: 70 °C and room temperature (RT) curing; (iv) an integrated study based on physical, mechanical, and

microstructural characteristics at different curing time, to evaluate the combined effect of the new mix design and precursor pre-treatments on the development of mechanical properties (Fig. 1).

2. Material and methods

2.1. Materials

Class F (ASTM C618 standard) coal FA were sourced by BauMineral (EFA-Füller®, Germany). The chemical composition, determined by inductively coupled plasma optical emission spectrometry (ICP-OES), is reported in Table 1.

Fig. 2 shows the XRD pattern of the as-received FA recorded using a Philips PW 1280 diffractometer, equipped with a Cu-K α (1.5406 Å) radiation source excited with 30 mA and 40 kV. Scanning rate was set at 0.02°/sec for a total 2 θ range of 4–80°. The obtained pattern was smoothed applying a Savitzky-Golay filter. The precursor is highly amorphous, and the two main crystalline phases detected are quartz (SiO₂) and mullite (3Al₂O₃·2SiO₂).

To test the influence of the precursor pre-treatment on the performances of the activated mortars, FA has been treated by a mechanical (FA-MM) and a mechano-chemical process (FA-MC) by means of ball milling using different alumina ball-to-powder (B/P) weight ratios. The milling process lasted 2 h. Particle size distribution of the as-received and mechanically treated FA was determined using a laser diffraction particle size analyzer (Mastersizer 2000, Malvern Instruments). Whereas the mechanical milling involves only FA in order to reduce their particle dimension (FA-MM), the mechanochemical process (FA-MC) consists of ball-milling of a solid blend made of FA and activators (KOH and anhydrous sodium metasilicate) to immediately start the reaction thanks to the milling process. This approach was firstly proposed in a previous study by Matalkah et al. where FA was dry-blended with activators (quick lime, MgO and NaOH) and ball-milled for 2 h [24]. Morphological observations and chemical analysis of the precursors before and after pre-treatments were carried out using a field emission gun

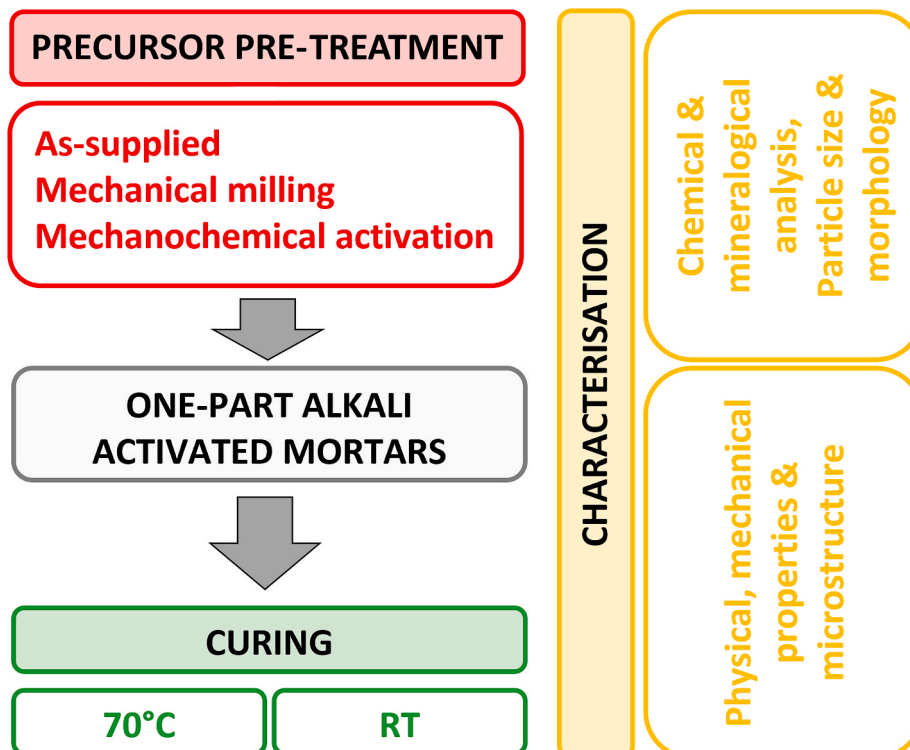
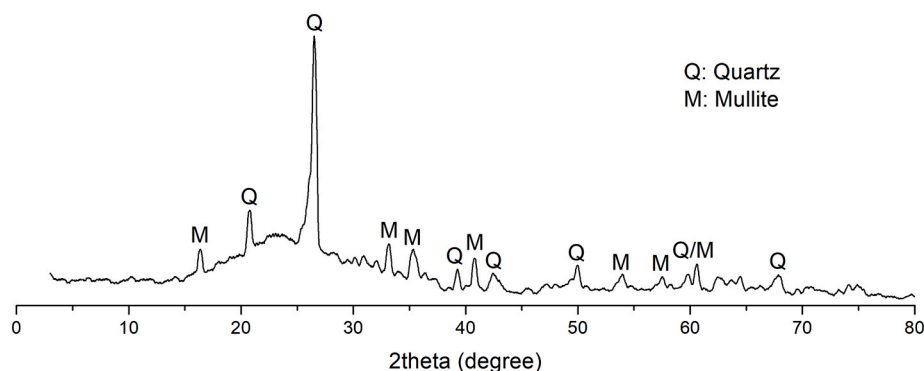


Fig. 1. Scheme illustrating the rational and the parameters investigated in the study.

Table 1

Chemical composition (oxides in wt%) of FA determined by ICP-OES. Loss of ignition (LOI) is also reported.

Mass (%)	SiO ₂	Al ₂ O ₃	TiO ₂	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	SO ₃	LOI
FA	56.58	23.04	0.93	6.11	5.88	1.76	1.14	0.86	0.99	2.71

**Fig. 2.** XRD pattern of the as-received FA.

scanning electron microscope (SEM-FEG, Tescan Mira3).

Anhydrous sodium metasilicate (Na₂SiO₃, Alfa Aesar) with a SiO₂/Na₂O modulus of 0.93 and a density of 1.1 g/cm³ and potassium hydroxide (KOH, Sigma Aldrich) have been used as alkaline activators. Particle size distribution of sodium metasilicate ranges from 200 to 800 μm, as reported in the product data sheet. In order to improve its dissolution in water, it has been milled in a ceramic jar with a B/P weight ratio of 1:1 for 30 min to reduce its particle size prior to sample preparation.

Standardized silica sand (EN 196–1:2016), with a specific grain size distribution that ranges between 0.08 and 2.00 mm, was used as fine aggregate for mortar preparation.

2.2. Sample preparation

Mortars have been prepared using the mix design reported in Table 2 with the following molar ratios: SiO₂/Al₂O₃ = 4.70, M₂O/Al₂O₃ = 1.00 and M₂O/SiO₂ = 0.21, where M is the molar sum of the alkaline metals, Na and K. Standardized silica sand (EN 196–1:2016) to precursor weight ratio was equal to 2.7. As-received FA, mechanically ball milled FA and mechanochemically activated FA have been alternatively used as precursor for the preparation of the corresponding mortars, respectively named as M-FA, M-MM, M-MC. The workability was measured by flow table test according to EN 1015–3:1999 standard using a conical ring (d_{max} = 100 mm, d_{min} = 70 mm, h = 60 mm). The average workability value for all the mixes, determined as the percentage of the difference between the average diameter of the spread mixture and the diameter of the conical ring (100 mm) over the same diameter, was 95 ± 10%. The water to precursor ratio (H₂O/FA) of 0.35 was kept constant in all the mortars, except in M-MM samples in which extra-water was added, because of the lower particle size distribution of the milled fly ash.

Authors are aware that the addition of water influences the properties of the hardened samples, however, a sufficient workability was

Table 2

Mix design composition in wt% reporting the concentration of fly ash (FA), sodium silicate (SS), potassium hydroxide (KOH), water (H₂O) and silica sand. Water to fly ash (H₂O/FA) and sand to fly ash (SAND/FA) ratios are also reported.

FA [wt %]	SS [wt %]	KOH [wt %]	H ₂ O [wt %]	SAND [wt %]	H ₂ O/FA	SAND/FA
23.3	3.9	2.2	8.1	62.5	0.35	2.7

necessary to allow sample preparation by casting, thus avoiding a noteworthy air bubble entrapment due to too high viscous mix.

Mixing has been carried out using a Hobart planetary mixer according to EN 196–1:2016. An optimized mixing procedure was followed based on the following steps: 1 min of dry mixing; 1 min during water addition, 11 min at increased speed.

Samples prepared for mechanical characterizations were cast into a 40 mm × 40 mm × 160 mm prismatic molds. The sample casting has been carried out in 2 phases, compacting the slurry with 60 jolts of the jolting apparatus. Cylindrical samples with a diameter of 30 mm and a height of 20 mm were also prepared for testing the physical and microstructural properties. After casting, samples were sealed in plastic bags and cured at room temperature (named RT) or at 70 °C (named 70). All the specimens were kept in sealed conditions for the whole time of curing: 7 days for heat-cured mortars and, 7 and 28 days for RT-cured mortars.

2.3. Characterization

Physical, mechanical, and microstructural analyses were carried out after 7 days for mortars cured at RT or at 70 °C. In addition, RT-cured samples were also tested after 28 days.

Dynamic elasticity modulus has been determined using a MATEST ultrasound tester (C369) on the prismatic beams. Compressive (R_c) and flexural strengths (R_f) were determined by using a Wolpert Probat testing machine applying a 200 kN load cell. Prisms were firstly assessed under the flexural loading and then the broken half samples were tested to compressive strength. Loading rate and testing conditions are in accordance with the EN 196–1:2016.

Physical properties, such as geometrical density, water absorption by total immersion and pore size distribution, have also been determined. Geometrical density has been measured by dividing the dry mass by the volume on cylindrical samples. All reported results are an average of 4 different measurements. Water absorption (WA) test was calculated as the percent increase in weight of the specimens after immersion in ambient temperature water until their complete saturation and/or sample weight does not vary more than 0.1%. Open porosity and pore size distribution has been evaluated through mercury intrusion porosimetry (MIP) test using Pascal Mercury Porosimeters (Pascal 140 & Pascal 240, Thermo Scientific). The MIP measurements were carried out using a contact angle of 141.3° and a Hg surface tension of 480 dyne/cm.

Finally, microstructure of raw materials and one-part mortars was observed using secondary electrons of a field emission gun (FEG) SEM

(Tescan Mira3) coupled with an energy dispersive spectrometer (EDS). Fracture surfaces of one-part mortars were prepared for SEM observation. All the samples were carbon coated before SEM observation to make them conductive. Fourier Transform Infrared spectroscopy (FT-IR, PerkinElmer Spectrum One spectrometer) was carried out on *ad hoc* powder samples sourced by one-part paste specimens prepared following the same procedures (without adding silica sand) described in paragraph 2.2. The analyses were recorded in the range of 400–4000 cm^{-1} with a resolution of 4 cm^{-1} .

3. Results and discussion

This section reports the results about the optimization of mechanical ball milling procedure on FA. Mechanical, physical, and microstructural properties of one-part mortars prepared with as-received, ball milled and mechanochemically activated FA are discussed in light of different curing conditions.

3.1. Optimization of fly ash pre-treatment

Fig. 3 reports the microstructure (observed by FEG-SEM) and the particle size distribution of the as-received FA, as well as after mechanical milling using different B/P ratios (1:1 and 3:1).

The mechanical milling action caused a reduction in the particle size of FA, from a d50 (median value) of 15.7 μm for the as-received FA to 4.1 and 2.8 μm for milled FA with B/P ratios of 1:1 and 3:1, respectively (Fig. 3). B/P ratios higher than 3:1 were also tested, however the relevant particle size distributions and d50 did not significantly change from those determined for B/P equal to 3:1, thus they have not been reported in Fig. 3. The typical spheroidal morphology [25,26] of fly-ash is clearly evident for the as-received particles, whereas fly-ash fragments with sharp edges become largely present increasing the B/P ratio up to 3:1.

As fly ash particle size distribution was determined by analyzing a water powder dispersion, the high solubility in water of the activators did not allow to determine this feature for the mechanochemically treated FA (FA-MC). However, Matalkah et al. assessed that

mechanically milled FA and mechanochemical FA have similar dimensions [24]. Microstructural observations have been also carried out on the mechanochemically activated FA. Differently from when only mechanical ball milling is carried out, mechanochemical process promotes a strong change in fly ash morphology with the formation of subspherical clusters featured by acicular parts. For FA-MC3:1, spherical particles are almost absent (Fig. 4).

Mortar preparation and preliminary tests have been conducted on both mechanically ball milled and mechanochemically activated FA-based series (1:1 and 3:1 ratios). However, this study reports the performance of the mortars prepared with the pre-treated FA with a B/P ratio of 3:1, due to the fact that this process allowed the finest particle size distribution, thus the highest potential reactivity.

3.2. Mechanical and physical properties

Geometrical density (ρ_{bulk}) and mechanical properties, in terms of elasticity modulus (E_d), compressive (R_c) and flexural strength (R_f), are reported in Table 3. At 7 days of curing, RT- and 70 °C-cured samples show a strong difference in mechanical performances, even if density shows comparable values. The mechanical performances of samples cured at 70 °C, also including those exhibited when pre-treated precursors were used (*i.e.*, M-MM and M-MC), are always higher than those shown by samples cured at RT for the same period (7 days). This result is expected because heat curing generally allows a faster development of mechanical properties compared to RT-curing [27].

However, when RT-cured samples were tested after 28 days, higher values of elasticity modulus and strength were measured compared to those measured at 7 days in the same condition. In addition, a slight increase in geometrical density values was also detected. The obtained results for the RT-cured samples after 28 days are more similar to the ones obtained by 70 °C-cured samples after 7 days. Comparable E_d values were obtained, while lower strength values, especially in terms of compressive strength, were recorded for the RT-cured samples. These data show that heat curing allows higher mechanical performances in the early stage of curing and the effect of FA pre-treatments is negligible

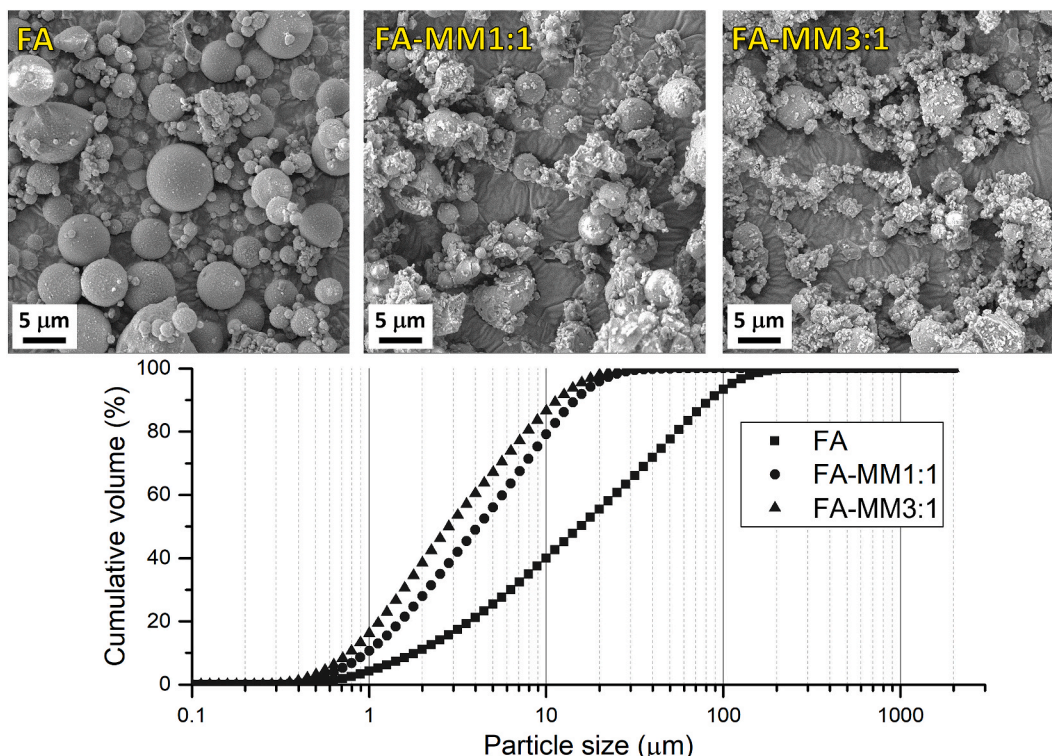


Fig. 3. SEM micrographs and particle size distribution of the as-received fly ash (FA), mechanical milled FA with B/P ratio of 1:1 (FA-MM1:1) and 3:1 (FA-MM3:1).

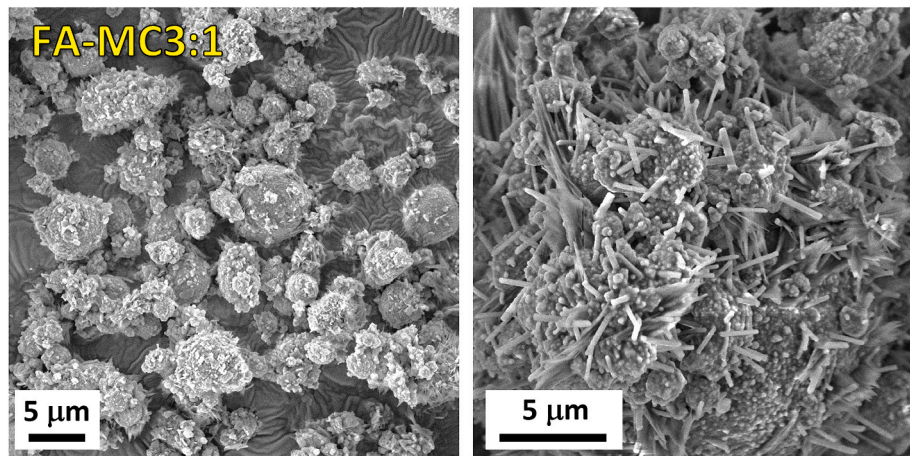


Fig. 4. SEM micrographs at different magnifications of fly ash after mechanochemical activation (FA-MC3:1). A B/P ratio of 3:1 was applied.

Table 3

Geometrical density and mechanical properties (E_d = dynamic elasticity modulus, R_c = compressive strength and R_f = flexural strength) of the mortars prepared by using as-received and pre-treated fly ash.

Sample	ρ_{bulk} (g/cm ³)	E_d (GPa)	R_c (MPa)	R_f (MPa)
M-FA-70-7	2.16 ± 0.01	37.2 ± 0.2	75 ± 4	7.7 ± 0.6
M-MM-70-7	2.06 ± 0.03	36.1 ± 0.5	83 ± 3	12 ± 1
M-MC-70-7	2.16 ± 0.03	39 ± 2	84 ± 4	7.4 ± 0.7
M-FA-RT-7	2.12 ± 0.03	17.7 ± 0.2	2.1 ± 0.1	0.24 ± 0.02
M-MM-RT-7	2.22 ± 0.05	26.0 ± 0.8	18.0 ± 0.9	2.3 ± 0.2
M-MC-RT-7	2.14 ± 0.04	18.9 ± 0.9	4.4 ± 0.2	0.93 ± 0.02
M-FA-RT-28	2.23 ± 0.02	35.0 ± 0.4	37 ± 3	5.1 ± 0.2
M-MM-RT-28	2.21 ± 0.04	33.4 ± 0.6	51 ± 6	4.6 ± 1.8
M-MC-RT-28	2.32 ± 0.02	42 ± 2	62 ± 3	7.0 ± 0.4

when curing is carried out at 70 °C. In particular, only a slight increase in the elasticity modulus (+4.6%) was achieved when FA has been mechanochemically activated. Moreover, for the M-MM-70-7 sample a slight decrease in both geometrical density and elasticity modulus was recorded, due to water addition in order to enhance the workability of the fresh mix, as reported in paragraph 2.2. In addition, a moderate increase in compressive strength (~+10%) has been detected for both the pre-treated FA-based samples (M-MM-70-7 and M-MC-70-7), and only a consistent improvement in flexural strength (+55%) was obtained for the milled FA-based mortars.

The influence of FA pre-treatments on the mechanical performances of the final products was instead clearly assessed when room temperature curing was carried out (Table 3). Already after 7 days of RT-curing, samples prepared with the milled FA showed an increase in geometrical density (+5%), elasticity modulus (+47%), and compressive and flexural strengths at least 9 times greater than the reference samples (M-FA-RT-7). At 28 days of curing, M-MM-RT-28 exhibited a further increase in compressive strength (+38%). This improvement in mechanical performance is mainly related to a better geopolymerization caused by a higher reactivity of the precursor exhibiting particle size reduction and greater specific surface [24,28].

For mechanochemically activated FA-based mortars, increased mechanical properties have been achieved at 7 days, but in a lower extent compared to M-MM-RT-7 samples (Table 3). This can be related to the fact that milling the precursor together with the solid activators does not allow to reach the same particle size distribution of the mechanically milled FA, due to the high hygroscopicity of the activators (*i.e.*, KOH and sodium metasilicate) and/or the starting of geopolymerization process. However, after 28 days of curing, the mechanochemical activation of the FA is the most effective precursor pre-treatments as it was recorded an increase in geometrical density (+4%), in elasticity modulus (+20%), as

well as in compressive (+68%) and flexural (+37%) strength. As proposed by Matalkah et al. [24], these results are probably linked to an increase of disordered aluminosilicate framework including alkali metal cations due to mechanochemical activation that allow an higher geopolymerization degree.

The results of physical properties, *i.e.*, water absorption and open porosity by MIP, are well in accordance with the reported mechanical properties (Table 3). Fig. 5 shows water absorption and open porosity values (Fig. 5a, c and e) and the pore size distribution (Fig. 5b, d and f). Mortars cured at 70 °C, when both as-received and mechanochemically activated FA have been used, exhibited comparable physical properties (WA of about 6% and OP values of about 10%, Fig. 5a) and very similar pore size distributions (Fig. 5b), thus confirming that precursor pre-treatment does not add any advantage when heat curing is carried out. There was however an exception for M-MM-70-7 with milled FA which showed higher WA and OP values. Its pore size distribution shows the highest total cumulative intruded Hg volume and a bimodal pore size distribution, with most of the pores having a dimension of 0.05 μm and 5 μm. Such a porosity is related to the increased H₂O/FA ratio necessary to allow good workability at the fresh state during sample preparation.

For RT-cured samples, the reference samples did not exhibit strong differences in term of porosity considering 7 (Fig. 5c and d) or 28 (Fig. 5e and f) days of curing, while the M-MM and M-MC specimens cured for 28 days showed a strong decrease in terms of OP and pore size distributions, highlighting in this case the importance of precursor pre-treatments which promote a densification of the matrix at long curing time.

3.3. Microstructural properties

SEM observations of the mortars cured in different conditions (RT and 70 °C) and for different time are reported in Figs. 6–8, showing two micrographs for each sample type at two different magnifications. In the observed microstructures, microcracks are always present and their origin can be due by the following causes: shrinkage during curing [29, 30], exothermal dissolution of activators as proposed by Askarian et al. [22], stress induced by vacuum during specimen preparation and/or SEM analysis. Generally speaking, a quite homogenous and dense microstructure was observed for all the specimens cured at 70 °C for 7 days (Fig. 6), regardless the use or the absence of precursor treatment. EDS measurements were performed in the correspondence of unreacted FA and gel for all the samples and similar results have been achieved: unreacted FA showed a Si/Al ratio of 2, while on gel microstructures a Si/Al ratio of about 3 have been measured, indicating a good grade of geopolymerization [25].

One-part mortars cured at RT for 7 days show microstructures characterized by a large number of unreacted FA compared to 70 °C-

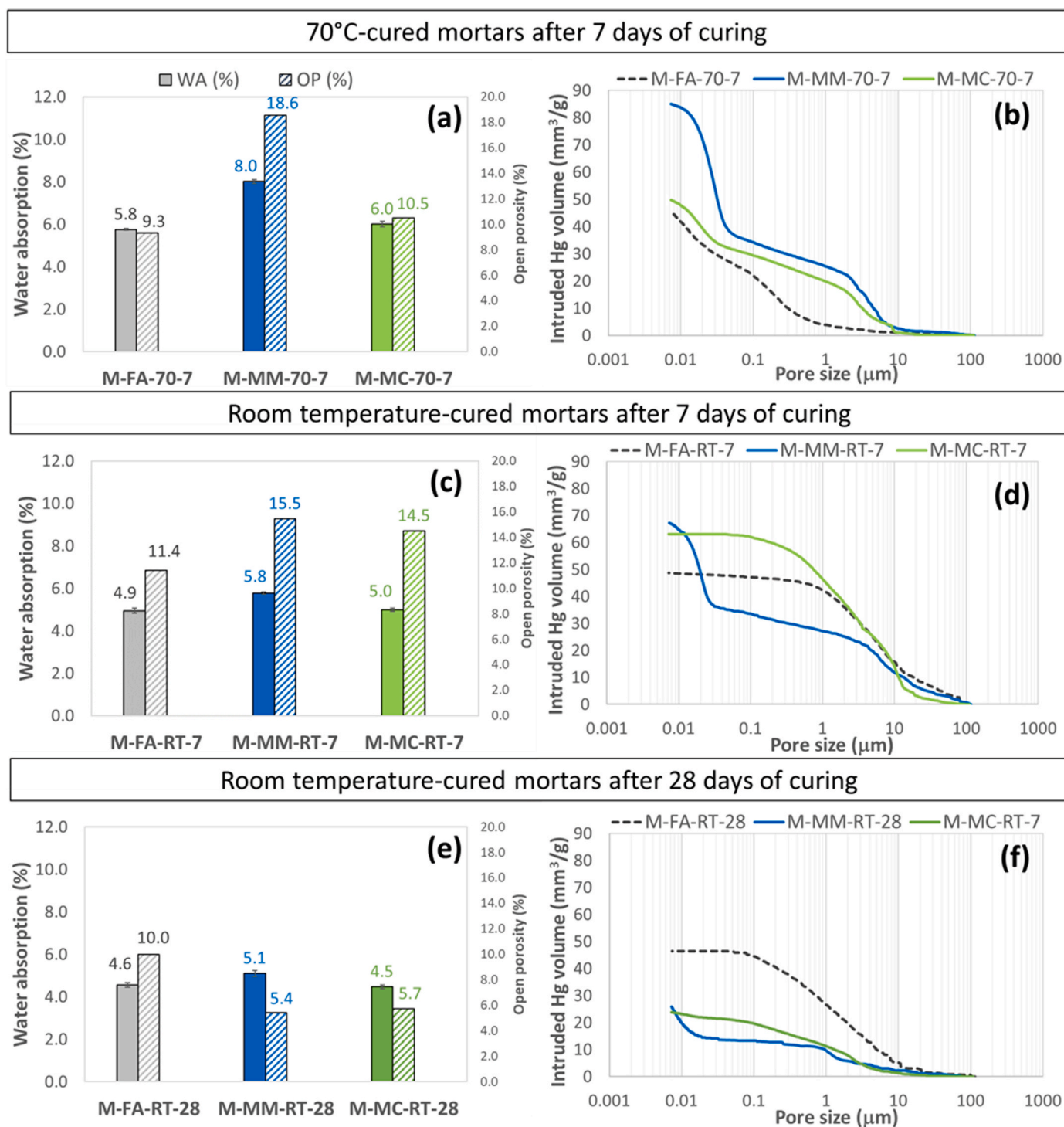


Fig. 5. Physical properties of the mortars prepared by as-received and pre-treated fly ash: (a, c and e) water absorption (WA) and open porosity (OP) values in percentage; (b, d and f) pore size distribution by mercury intrusion porosimeter.

cured samples, as shown in Fig. 7, even if the morphology of M-MC-RT-7 reported in Fig. 7b appears denser thanks to the contribute of the precursor mechanochemical activation. This observation is well in accordance with the mechanical performance and physical properties previously discussed.

Fig. 8 reports the microstructure of samples cured for 28 days at RT. Compared to what reported in Fig. 7, the microstructure of three samples resulted denser and more similar to the ones observed for 70 °C-cured samples (Fig. 6). Very low amounts of unreacted FA particles are evident for M-MM-RT-28 and M-MC-RT-28 confirming the beneficial

action of FA pre-treatments in geopolymerization process.

Fig. 9 reports the FT-IR spectra with the relevant values of the main vibration bands for FA (black dotted line) and the one-part binders. FA showed the main band at 1043 cm^{-1} related to the stretching vibration of Si–O–Si and/or Si–O–Al, while the other major band at 785 cm^{-1} is linked to Al–O vibration (typical of the Al_2O_3 structure) [22,23,31]. All the one-part binders exhibit the band of stretching vibration of Si–O–Si at lower values than FA precursor, in the range of $950\text{--}980\text{ cm}^{-1}$, thus indicating that the reactive components in the fly ash have been actively participated to the formation of the gel. Moreover, it is noteworthy to

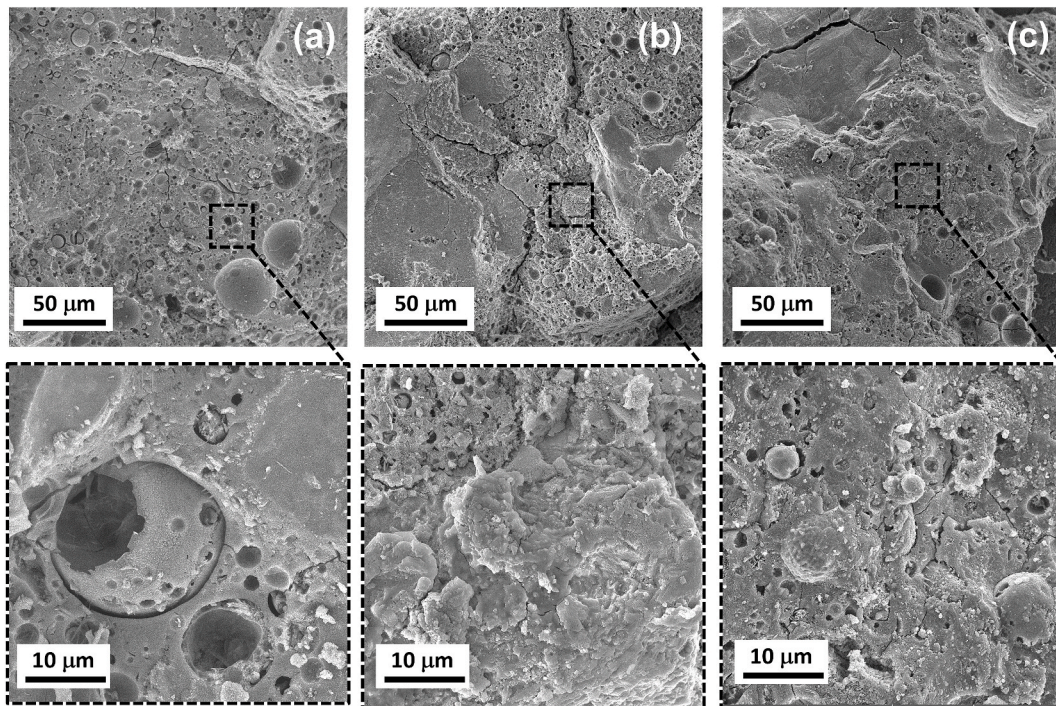


Fig. 6. SEM morphological observation of the 70 °C-cured mortars after 7 days of curing obtained from (a) the as-received, (b) the mechanically ball milled and (c) the mechanochemically activated fly ash.

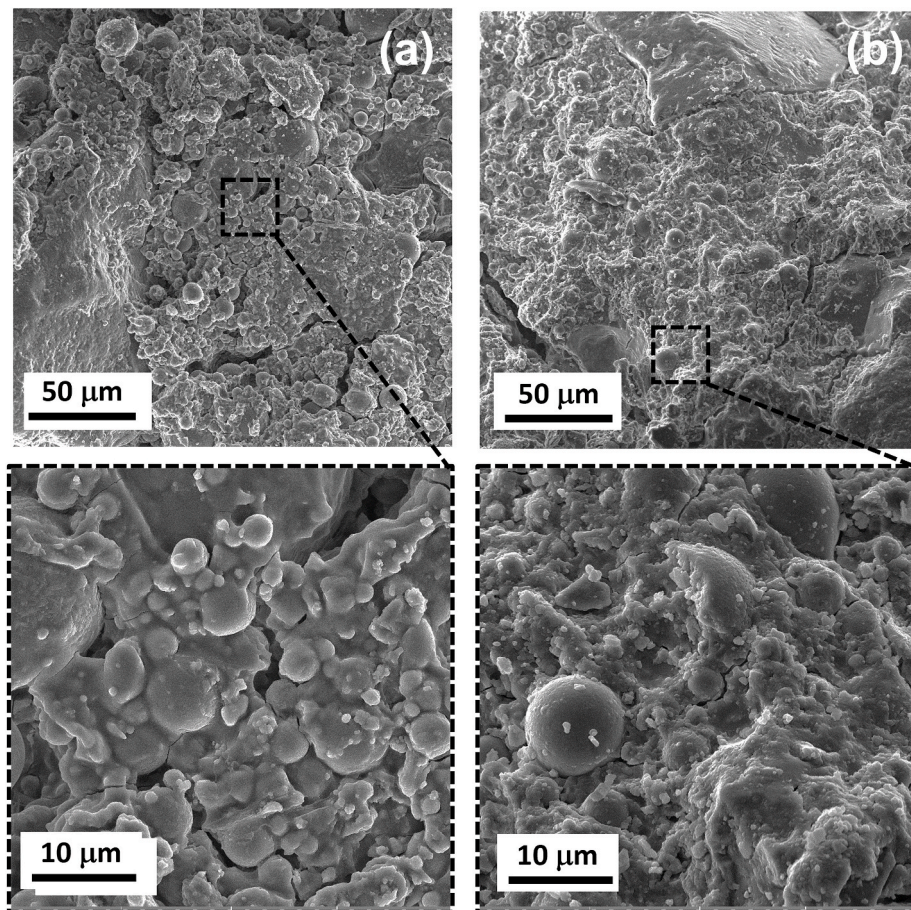


Fig. 7. SEM morphological observation of the RT-cured mortars after 7 days of curing obtained from (a) the as-received and (b) the mechanochemically activated fly ash.

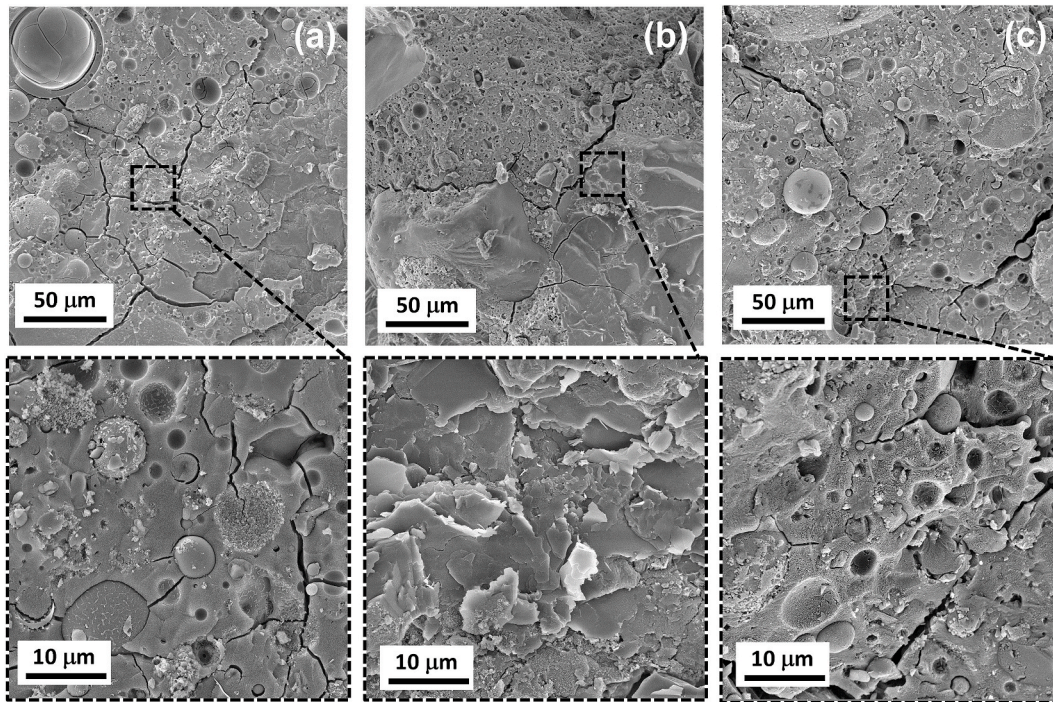


Fig. 8. SEM morphological observation of the RT-cured mortars after 28 days of curing obtained from (a) the as-received, (b) the mechanically ball milled and (c) the mechanochemically activated fly ash.

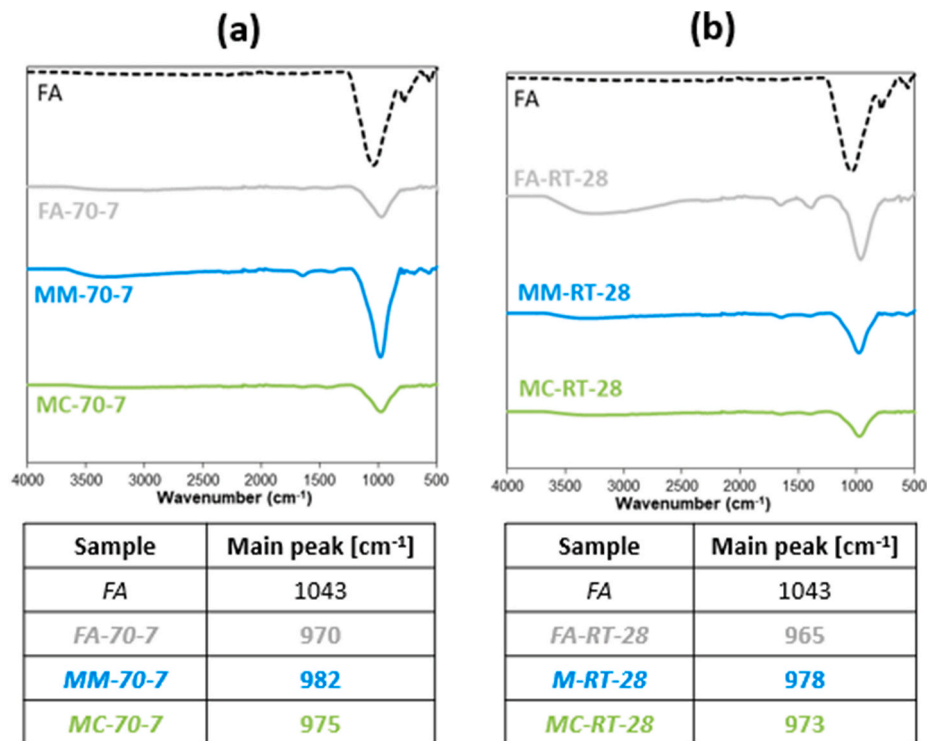


Fig. 9. FT-IR spectra of one-part paste specimens prepared with the as-received and pre-treated fly ash cured in different conditions: (a) at 70 °C for 7 days, and (b) at room temperature for 28 days. Values of the main vibration bands are also reported in the table below each spectrum.

highlight that the shift of the peaks is similar regardless the different curing conditions (70 °C tested after 7 days and RT tested after 28 days), thus indicating that a similar degree of geopolymerization has been reached.

4. Conclusions

High strength one-part alkali activated mortars by activating coal fly ash with a mix of KOH and anhydrous sodium metasilicate at room temperature have been achieved in this study. In particular, the effects of fly ash pre-treatments, such as mechanical milling and

mechanochemical activation, and the comparison between room-temperature and heat curing conditions have been investigated taking into account the mechanical performances, the physical and microstructural properties of final products.

The following conclusions can be drawn from this study:

- the solid activation mixes and the relevant mix designs developed in this research were suitable to obtain one-part geopolymers with satisfactory performances, as demonstrated by the mechanical, physical and microstructural characterization;
- one-part mortars obtained by pre-treated fly ash, cured at room temperature for 28 days, exhibited mechanical performance almost comparable to those obtained by specimens cured at 70 °C;
- the most performing product, reaching a compressive strength (R_c) greater than 60 MPa at 28 days of curing, has been obtained by mechanochemical activation treatment of fly ash.

Declaration of competing 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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