ORIGINAL ARTICLE

Experimental analysis of lime putty and pozzolan-based mortar for interventions in archaeological sites

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Abstract The study aims at defining and characterizing a specific restoration mortar for archaeological masonry structures made with traditional materials and to assess the suitability of the mixture compared to other mortars; such a goal is crucial to develop and define interventions in the archaeological sites. The mixture was defined to ensure compatibility with ancient materials and following frequently adopted recommendations at the site, specifically by using: (1) raw materials as similar as possible to the ancient ones; (2) traditional mix design. Therefore, the mixture was made with commercial lime putty CL 90-S type and natural Phlegrean pozzolan, i.e. volcanoclastic material collected from the volcanic area located in the West of Naples in Italy. The precious and limitedly available natural pozzolan used in the experiments resulted in an exclusive mortar which is very similar to the archaeological ones. The mortar has a binder to aggregate ratio 1:3 by volume, according to traditional techniques typically encountered in the ancient Roman city of Pompeii and Vesuvius surrounding area. The evolution of the flexural and compressive strength, elastic modulus, bulk density, open porosity and ultrasonic pulse velocity has been monitored for up to 200 days, based on standard procedures. Moreover, the hardening process was monitored with Differential Thermal Analysis up to 90 days, through the evaluation of phase transitions associated with dehydroxylation and decarboxylation, considering different depths from the external surface of the mortar. The achieved mechanical properties were compatible with those of lime-based mixtures for repair interventions of ancient masonry structures. Moreover, the mortar was found to be well-suited to mitigate cracking, showing a low ratio between its stiffness and load capacity compared to other typologies of mortars used for masonry restoration. Ultrasonic pulse velocity test proved to be a reasonable complementary method to monitor the evolution of the hardened properties of the mortar. Carbonation was found to be still progressing at 90 days. The data presented provide useful and reliable information to approach the complex process of restoration in archeological sites.

Keywords Archaeological Pompeii site · Repair mortar · Lime putty · Pozzolan · Carbonation · Compressive strength · Elastic modulus · Ultrasonic pulse velocity · Differential thermal analysis

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1 Introduction

Restoration interventions of archaeological masonry structures and columns are often based on the use of mortars. As a material for restoration interventions, a mortar should be compatible with the ancient materials, durable and its properties should be well documented [1–3]. In absence of specific requirements for different or innovative solutions, the use of traditional materials and techniques is generally preferred due to their good compatibility with the substrate material (i.e. free thermal dilation, salt content, stiffness and strength apart from aesthetics and authenticity issues)[3]. The production of specifically devised repair mortars by mixing single raw materials on-site should be defined based on an accurate knowledge of the raw materials and the craftsmanship composing the ancient ones. However, the exact definition of the original components often presents economic and technical constraints and, when they are known, it is often impossible to find the same materials.

Several experimental studies on the production of repair mortars based on the composition of the ancient ones are available in the literature. Some studies focused on aerial lime mortars (i.e. lime putty or hydrated lime) with siliceous or calcareous sand [4–8], while others added natural or artificial pozzolanic additions or used hydraulic lime instead [9–12]. Indeed, it is known that aerial lime is one of the most ancient binders used in mortars for masonry [6, 10, 13–17]. Its hardening process takes place through evaporation and carbonation, thus it needs contact with the carbon dioxide in the atmosphere and cannot occur underwater [8]. The carbonation process is gradual and very slow, starting from the outer surface of the joints in the first days after the application until reaching the inner part of the masonry from few months up to more than a year depending on the porosity of the mortar and the units, the wall thickness and the environmental conditions, in particular the relative humidity [7, 18].

Pozzolanic additions have been used with aerial lime for masonry mortars, in the form of natural pozzolans (volcanic ash or specific kind of earth) or artificial ones (crushed terracotta or wood ash)since ancient times, especially in the Roman period. Such additions served the function of reacting with the lime and the water in the mixture to give it hydraulic

properties, and also for improving the final strength of the mortar [9, 12–14, 18, 19]. The addition of pozzolans modifies the hardening process of air lime-based mortars, by introducing the formation of hydration products (calcium silicate hydrates and calcium silicate aluminate hydrates) similar to hydraulic lime or cementitious mortars, but with a slower rate of hardening compared to cement [20].

As concerns the Pompeii site, several studies focused on the petrographic, mineralogical and chemical characterization of ancient mortar-based materials (bedding mortars, plasters and floor mortars). They found that these materials were mainly composed of aerial lime as a binder (i.e. lime putty) and local volcanic aggregates, sometimes with crushed ceramics, limestone sand or marble powder [21–28], while the mechanical properties of such materials nowadays are still poorly studied [29].

Technical sources related to structural interventions at the Pompeii site provide indications from the authority of the site for the production of repair mortars (for instance, technical documentation related to restoration interventions at *the Insula Meridionalis* available from pompeiisites.org). Suggested mixtures shall be made with (1) raw materials as similar as possible to the ancient ones, i.e. lime putty and Phlegrean pozzolan, and (2) mix design consistent with traditional techniques, i.e. binder to aggregate ratio equal to 1:3 by volume. However, comprehensive information on the mechanical performances of this type of materials, which is essential to assess their suitability for different types of interventions and their compatibility from a structural point of view, is still lacking. Goldsworthy and Min [11] investigated similar mortars, made with aerial lime powder, pozzolan from Bacoli (within the Bay of Naples) and water, with the binder to aggregate ratio varying from 1:1 to 1:4, but they only focused on the compressive strength of the mixture. On the contrary, investigations on the evolution of several mechanical and physical properties with the time are available only with regards to other typologies of mortars for interventions on masonry structures (mainly cement-lime blended mortars).

As a part of wide research intended for the definition and implementation of suitable investigation protocols and approaches for the structural interventions at the Pompeii site [30], the present study focuses on the analysis of a repair mortar compliant with the

abovementioned indications by following a carefully controlled procedure; accurately measuring several important mechanical and physical properties and assessing their evolution with the time; analyzing, discussing and comparing the achieved results, most of which were still not available in the literature. The mixture was made with lime putty and pozzolan from the Phlegrean area (i.e. a volcanic area located at the West of Naples, Italy), with a binder to aggregate ratio 1:3 by volume. The evolution of the flexural and compressive strength, elastic modulus, bulk density, open porosity and ultrasonic pulse velocity has been monitored up to 200 days, according to standard procedures. Also, the rate of the hardening process and the evolution of the carbonation front have been specifically studied through the Differential Thermal Analysis.

The significance of this research is related to the distinctiveness of the produced mixture and its similarity with the archaeological mortars, especially regarding the aggregate. Indeed, the natural pozzolan used in the experiments is a precious material whose sourcing and usage is nowadays highly controlled and limited.

2 Outline of the experimental program

2.1 Overall methodology

A repair mortar compatible with the archaeological structures was defined in this study. To this end, the mixture was defined by using: (1) raw materials as similar as possible to the ancient ones, (2) a mix design consistent with ancient traditional ones and (3) a mortar consistency suitable for workability. Then, a comprehensive investigation of the mechanical and physical properties was performed to evaluate the suitability of the mortar as a material for repair in masonry structures. The experimental program involved 13 batches of mortar, 62 prismatic or cylindrical specimens and different types of destructive and non-destructive tests carried out at different ages from 3 days up to 200 days. A total number of 201 tests were carried out. Figure 1 shows prismatic (40 mm × 40 mm × 160 mm, named "P") and cylindrical (60 mm × 120 mm, named "Cyl_A" and 60 mm × 60 mm, named "Cyl_B") specimens used in

this work, while Table 1 reports the full experimental program outline.

An explanatory nomenclature *B.Ty.C.Te.x.N* was assigned to each test, where:

- B is the number of the batch of provenance (from 1 to 13);
- Ty is the specimen type (fresh mortar, FM, prismatic, P, or cylindrical, Cyl_A or Cyl_B);
- C is the curing condition (A or B as a function of humidity and temperature, being A standard conditions and B controlled carbonation conditions, further detailed below);
- Te is the performed test (Flow test, FT, Bulk Density measurement, BD, Ultrasonic Pulse Velocity test, UPV, Flexural and Compression test, FC, Open Porosity test, OP, Compression test, C, Cyclic Compression test, CC, and Differential Thermal analysis, DTA);
- x is the age of testing (3, 5, 6, 7, 28, 60, 90 or 200 days);
- N is the progressive number of the specimen (1, 2, 3) or sample derived from the specimens for Differential Thermal Analysis (1, 2, 3, 4).

A detailed specification of the performed tests with their relative nomenclature is reported in Annex.

For the raw materials, commercially available lime putty, CL90-S type according to the standard EN 459-1 [38], was used as a binder and volcanic sand from the Phlegrean area, called "pozzolan sand" in the following, was used as an aggregate. A description and brief characterization of the raw materials is provided in Sect. 2.2.

The recommendation provided by Vitruvius in *De Architectura* (*Liber II*, 5 and *Liber V*, 12, 15 A.C.) and by Pliny the Elder in *Naturalis Historia* (*Liber XXXVI*, 175, 77-78 A.C.) have been taken into account for the definition of the binder to aggregate ratio of 1:3. Moreover, 111.7 g of extra water for each 1000 g of pozzolan sand was added to the mixture to obtain a workable mortar, but as stiff as possible, consistently with required performances for a repair mortar [11, 31]. The amount of extra water was defined through subsequent attempts to achieve a plastic consistency corresponding to a flow diameter of 165 ± 10 mm according to the standard EN 1015-3 [32]. For each trial of water to binder ratio, a certain amount of water was added to the mixture and the consistency was defined through the flow test [32].
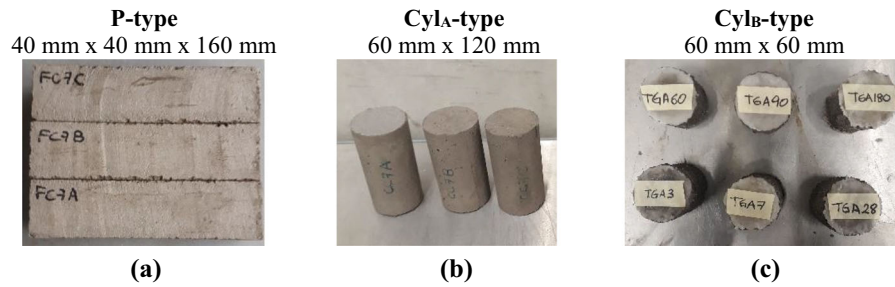
Fig. 1 Types of specimens: prismatic 40 mm × 40 mm × 160 mm, P (**a**); cylindric 60 mm × 120 mm, CylA (**b**); cylindric 60 mm × 60 mm, CylB (**c**)

Table 1 Experimental program outline

Test	Outcomes	Type of specimen	Curing	Age (days)	Total number of tests
FT	Flow diameter (mm)	FM	–	–	2
BD	Bulk density, ρ (kg/m^3)	P	A	5, 6, 7, 28, 60, 90, 200	57
UPV	Ultrasonic pulse velocity, V (m/s)	P	A	5, 6, 7, 28, 60, 90, 200	57
FC	Flexural and compressive strength, f_f and f_c (MPa)	P	A	7, 28, 60, 90, 200	21
OP	Open porosity (%)	P	A	7, 28, 60, 90, 200	15
C	Compressive strength, f_c^* (Mpa)	Cyl$_A$	A	7, 28, 60, 90, 200	15
CC	Elastic modulus, E (Mpa)	Cyl$_A$	A	7, 28, 60, 90	12
DTA	Evaluation of phase transitions	FM and Cyl$_B$	B	3, 7, 28, 60, 90	22
Total number of tests					201

The mixing procedure was performed according to the protocol provided by the standard EN 459-2 [33], by using an automatic mixer constituted of a stainless steel bowl, with 5 L of capacity. The starting time of the mixing process, t_0, was defined as the instant at which water came into contact with the binder in the bowl. Repair mortars are frequently prepared on-site using not standardized methods, which can involve hand mixing by using a bucket and trowel or mechanical by using an electric mortar mixer. However, standardized methods and tools were selected for this work to enhance the repeatability of the results. Once the desired consistency was achieved, the repeatability of the mixing process was checked by repeating the flow test on a new batch of mortar. Finally, the proportion of binder:aggregate:water was defined 1:3:0.5, by volume. Then, to simplify the mixing process the composition by volume was converted into weight, based on the density of the raw mate-rials (given by 275.8:1000.0:111.7 g for 1 kg of pozzolan sand).

For the test specimens, prismatic specimens 40 mm × 40 mm × 160 mm according to the standard EN 1015-11 [34] (Fig. 1a), were used for the measurement of the bulk density and the ultrasonic pulse velocity tests at 5, 6, 7, 28, 60, 90 and 200 days, flexural and compression strength tests and open porosity tests at 7, 28, 60, 90 and 200 days. Cylindric specimens 60 mm × 120 mm, Cyl$_A$-type (Fig. 1b), were used for compressive strength tests at 7, 28, 60, 90 and 200 days and cyclic compression tests for the evaluation of the elastic modulus at 7, 28, 60 and 90 days; finally, five single cylindric specimens 60 mm × 120 mm, Cyl$_B$-type (Fig. 1c), were prepared for the collection of samples to monitor the evolution of carbonation through Differential Thermal Analysis at 3, 7, 28, 60 and 90 days.

Hand compaction method was used for all the types of specimens, according to the procedure indicated in

the standard EN 1015-11 [34] for aerial lime mortars and 40 mm × 40 mm × 160 mm prismatic specimens: the molds were filled in two almost equal layers each one compacted by twenty-five strokes of a tamper. Indeed, the method was applied also for the cylindrical specimens since hand compaction was consistent with the hand laying method of the repair mortar on-site, which does not involve vibration.

The measurement of the bulk density, ultrasonic test, flexural and compression test, open porosity test, compression and cyclic compression tests for the evaluation of the elastic modulus involved the execution of three tests for each age. The same specimens were used for the measurement of ρ and V at 5, 6 and 7 days and then they were used for flexural and compression tests at 7 days. For each of the following ages (i.e. 28, 60, 90 and 200 days), the same specimens were used for the measurement of ρ and V and finally for the flexural and compression tests. Moreover, the same three specimens were used for the cyclic compression tests at 28, 60 and 90 days.

Additional tests were performed for the assessment of the repeatability of the results. In particular, six extra P-type specimens were prepared from the same batch of mortar (i.e. batch 3 as reported in Appendix) and used to assess the repeatability of results with hand compaction compared to mechanical compaction. For that, three specimens were compacted by hand according to the method indicated above and three specimens were compacted by using a jolting apparatus according to the method indicated in the standards EN 459-2 and EN 196–1 [33, 35] (i.e. filling the mold in two layers each one compacted for 60 s). Therefore, the mean bulk density and ultrasonic pulse velocity at 5, 6 and 7 days and the mean flexural and compressive strength obtained at 7 days of the hand compacted specimens were compared with the ones of the mechanically compacted specimens. Additionally, to check the quality of the specimens and the repeatability of the results of specimens obtained from different batches of the same mix, the measurement of ρ and V was performed on three reference specimens at 5, 6, 7, 28, 60 and 90 days (i.e. on the specimens used for the measurement of ρ and V and for the flexural and compression tests at 200 days) and the values obtained were compared with the ones obtained on the specimens tested at every single age.

Differential Thermal Analysis involved the execution of four tests for each age, according to a specific order for the collection of the samples and testing, to ensure repeatability and effectiveness of the procedure as described in Sect. 2.4. Moreover, before the execution of Differential Thermal Analysis on hardened mortar, the test was performed on single raw materials and fresh mortar. For fresh mortar, two samples were collected directly from the mixing bowl at the end of the mixing processes of batch 4 and batch 5 respectively (as reported in Annex), using a metallic tip. The samples were immediately wrapped in a plastic film and stored in a controlled environment at 20 ± 1 °C and $95 \pm 5\%$ relative humidity. Thus, they were tested within two hours from t_0. The protocol adopted for Differential Thermal Analysis on the raw materials, fresh mortar and hardened mortar involved: a single increasing ramp, from 20 to 1100 °C, with a rate of 10 °C/min [7, 9, 10, 36, 37]. The heating rate, 10 °C/min value was selected to optimize the duration of every single test as well as the accuracy of the results, according to a common value accepted in literature [7–10, 36, 37].

The P-type specimens and the Cyl_A-type ones were stored in a controlled environment at 20 ± 1 °C and $95 \pm 5\%$ relative humidity up to five days, then they were demolded and cured unsealed for the remaining days up to testing in a controlled environment at $20° \pm 1°$ and $60 \pm 5\%$ relative humidity, following the indications of the standard EN 1015-11 [34]. This curing method is referred to in the following as "A". For the CylB-type specimens, specific preparation and curing method was adopted, to allow the carbonation process to start as soon as possible and control its progression. The specimens were cast in cylindrical molds and completely sealed with plastic tape at the top and the bottom surfaces, and stored at 20 ± 1 °C and $95 \pm 5\%$. Since the specimens were too fresh to remove from the cylindrical mold the first day after casting, they were demolded after two days. Thus, to ensure the carbonation process to develop only through the lateral surfaces, immediately after removing the cylindrical molds and plastic tape, the top and the bottom of the cylinders were again completely sealed by putting paraffin layers on them. Then, the specimens were cured in a controlled environment at 20 ± 1 °C and $60 \pm 5\%$ relative humidity. This second curing method is referred to in the following as "B".

2.2 Raw materials

The choice for the selection of raw materials and mix design in this research was based on compatibility requirements with archaeological materials. Studies on ancient Roman building techniques and specific compositional analyses of archaeological mortars from Pompeii found that mortars were mainly composed of almost pure lime putty produced on-site and local volcanic aggregates [21–28]. Based on those findings, a commercial lime putty CL90-S type was selected for this work, since it ensured the least amount of impurity possible compared to the other commercially available types. Indeed, the production of the putty lime on site was not feasible for economic, logistic, time, and safety constraints. In detail, the selected putty was characterized by the following performances certified according to the specification of the standard EN 459-1 [38]: 98% of the granulometry lower than 0.1 mm, CaO + MgO $\geq$ 90%, MgO $\leq$ 5%. CO2 $\leq$ 4% and SO3 $\leq$ 2%. The bulk density of the lime putty was measured according to the standard EN 1015-6 for fresh mortar [39] and resulted in 1.23 g/cm^3.

As concerns the aggregate, a local volcanoclastic material with variable size from ash to lapillus, collected from the Phlegrean area and called "pozzolan sand" in the following, was used in the mixture. This is a volcanic region next to the Bay of Naples where the ancient Roman builders obtained their pozzolan (traditionally known as *pulvis puteolanus*). The pozzolan sand had a bulk density of 1.49 g/cm^3, measured according to the standard EN 1097-3 [40] and a particle size distribution showed in Fig. 2, evaluated according to the standard EN 933-1 [41].

Figure 3 shows Differential Thermal Analysis thermograms of lime putty, pozzolan sand and two samples of fresh mortar. The tests confirmed the purity of the lime putty, with two main heat flow peaks related to the presence of free water and calcium hydroxide. The pozzolan sand was tested without any previous oven-drying. After a slight heat flow peak corresponding to the presence of free water, the sand did not show any significant weight loss in the present range of study. The thermograms of the two samples of fresh mortar were consistent with those for the raw materials, showing two main heat flow peaks related to the presence of free water and calcium hydroxide, respectively.
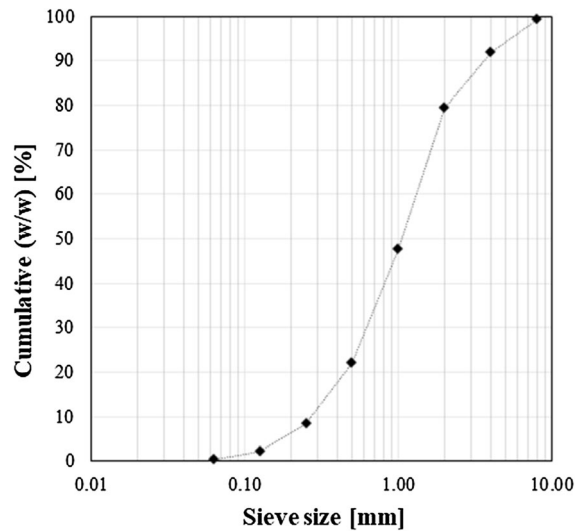
Fig. 2 Particle size distribution (0.063–8.0 mm) of the pozzolan sand
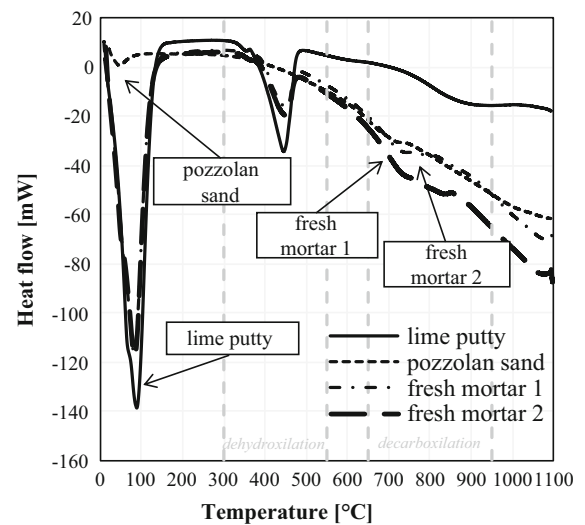
Fig. 3 Differential thermal analysis curves of lime putty, pozzolan sand and the two samples of fresh mortar tested within 2 h from the start of the mixing protocol

2.3 Testing procedures of physical and mechanical properties of the hardened mortar

At each age, the bulk density, ρ, was evaluated for three *P*-type specimens at first, by calculating the mean of the ratios between the weight of specimen and its volume. After that, ultrasonic tests were performed on the same specimens by positioning the probes in the

middle of the specimen along the transversal direction, thus the path length for the calculation of the ultrasonic velocity, V, was equal to 40 mm (Fig. 4a). Standard ultrasound equipment was used, with probes 25 mm of diameter and an operating frequency of 150 kHz. As abovementioned, The measurement of the bulk density and the ultrasonic test were also performed at each age on the reference specimens. After that, the evaluation of the flexural and compressive strength was performed according to the standard EN 1015-11 [34]. The flexural strength, f_f, was evaluated from three-point bending tests performed in displacement control with a rate of 0.003 mm/s at 7 days and 0.006 mm/s at 28, 60, 90 and 200 days (Fig. 4b). The compressive strength, f_c, was evaluated on the two resulting halves of each prismatic specimen from the flexural test using displacement control with a rate of 0.012 mm/s for all ages.

For the evaluation of the open porosity, at each age other three P-type specimens were dried, then they were subjected to water saturation in a vacuum pump (Fig. 4c). The open porosity was evaluated as the mean of the values obtained, calculated as: $(M_3–M_1)/(M_3–M_2)\bullet 100$, were M_1 is the dried weight, M_2 is the immersed weight and M_3 is the saturated weight. This method was applied by adapting the recommendations RILEM TC 25-PEM for stone [42], with the time of immersion and vacuum modified to 3 h.

The static elastic modulus, E, was evaluated by adapting the method presented in the standard EN 12390-13 [43] for concrete. At each age, at first, a simple compression test, C, was performed on three Cyl_A-type specimens (by using displacement control at a rate of 0.012 mm/s), for the evaluation of the mean maximum load and compressive strength. Then other three Cyl_A-type specimens were tested with five loading/unloading cycles up to one-third of the mean maximum load, in force-control with the rate defined so that the ramps lasted about 60 s. The elastic modulus of each specimen was evaluated from the measurements of three Linear Variable Differential Transducers (LVDTs), placed at the middle of the height of the specimens, on a base of one-third on the height, being supported by two steel rings and spaced of 120°. Before the execution of compression and cyclic compression tests, each specimen was rectified employing thin layers of epoxy resin applied on the top and the bottom surfaces (Fig. 4d).
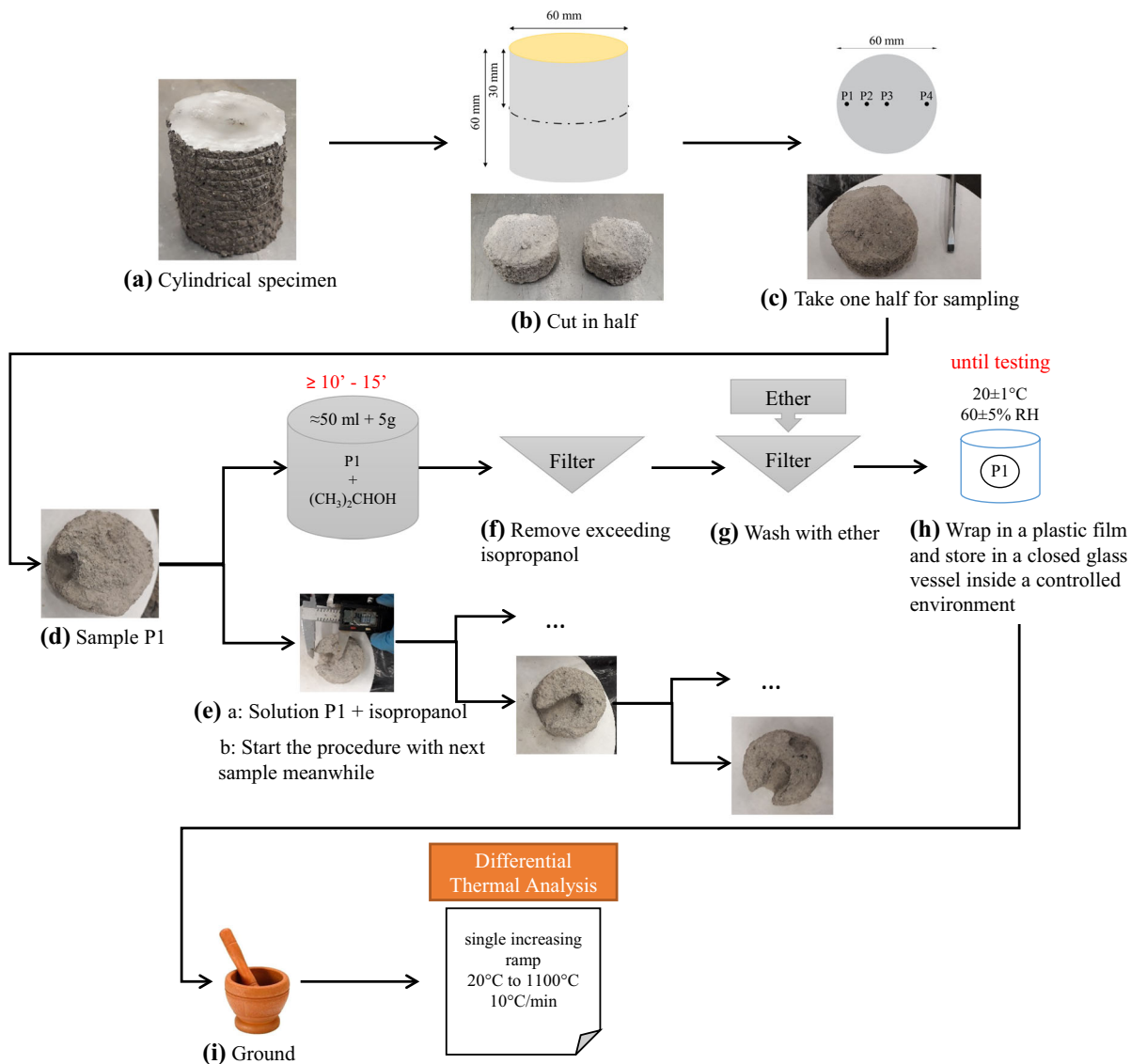
2.4 Differential thermal analysis methodology for the study of the carbonation process

For the execution of Differential Thermal Analysis, one specimen Cyl_A-type was cut in the middle at each age (at half of the height) and 4 samples were collected at different depths of the cut surface of one of the obtained halves, while the other half was stored in a plastic bag (Fig. 5a, b). Each sample had a volume of about 12 mm × 12 mm × 12 mm and was extracted using a metallic device with a slender tip. The samples were collected according to a pre-defined order (i.e. the first on the external surface, the second at the middle of the radius, the third at the core and the fourth on the opposite surface) (Fig. 5c).

After the collection of each sample, a pretreatment was applied to the samples to prevent the evolution of the carbonation. Such procedure was inspired by one of the two methods suggested by Scrivener [36] regarding cementitious materials for the suppression of hydration and removing soluble ions from pore
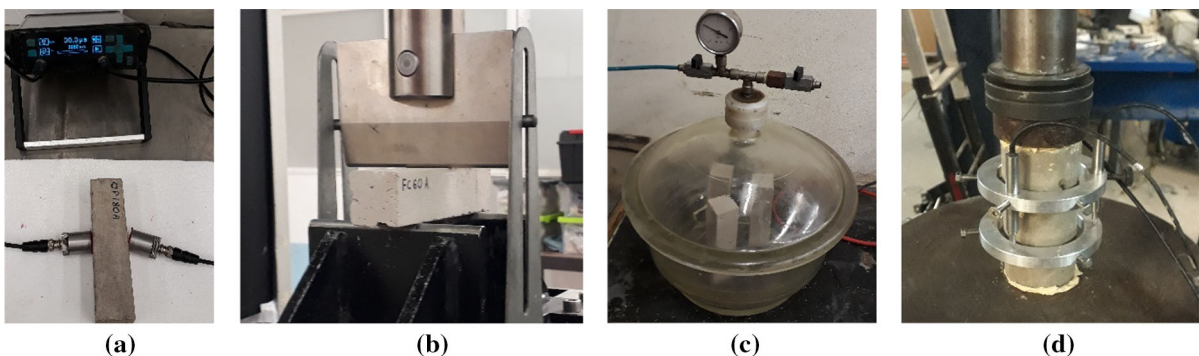
Fig. 4 Test set-ups: ultrasonic pulse velocity (**a**); three-point bending (**b**); open porosity (**c**); cyclic compression (**d**)

Fig. 5 Procedure flow chart for differential thermal analysis tests

solution. It was adapted for stopping carbonation to remove pore water, which is necessary for carbonation to occur [8].

Once the first sample was collected, it was immediately immersed in isopropanol $(CH_3)_2CHOH$ and stirred (about 50 ml of isopropanol for 5 g sample), while the rest of the specimen was covered by a plastic bag. Immediately after the first sample was put in the isopropanol, the collection of the next sample started, and the procedure continued until the fourth sample in the shortest time possible (Fig. 5d, e). Each sample rested in the solution for at least 10 min. Afterward,

starting from the first sample, each solution was filtered, to remove the excess isopropanol (Fig. 5f) and washed (still above the filter device) with ether (easily volatile solvent) to replace the isopropanol (Fig. 5g). Finally, each sample was wrapped in a plastic film and stored in a closed glass vessel (of a volume of about 0.25 L) inside a controlled environment at 20 ± 1 °C and $60 \pm 5\%$ RH (Fig. 5h). All the samples were tested within a maximum of two days from the beginning of the sampling process, for the sake of consistency. Before the execution of each Differential Thermal Analysis, the sample was removed from the

container, furtherly ground with a ceramic bowl and pestle (Fig. 5i). The tests were performed according to the same order of collection and preparation of the samples. This allowed: (1) repeatability of the procedure at the different ages; (2) assessing the effectiveness of the method applied to stop the carbonation, between samples 1 and 4.

3 Experimental results

3.1 Repeatability of results

To assess the repeatability of results obtained with the hand compaction method compared to mechanical compaction, Table 2 reports the mean values of ρ, V, f_f and f_c with the standard deviations and coefficients of variation (CoV) reported in brackets for hand compacted specimens and mechanically compacted ones, produced from the same batch of mortar (i.e. batch 3, as reported in Appendix). Despite a slight difference of results was detected between the two sets of specimens (i.e. ρ and V were higher in mechanically compacted specimens compared to hand compacted ones, the contrary happened for f_f and f_c), they were considered acceptable compared to the variation encountered in each of the individual tests, and also comparable with the variation encountered in the tests performed in experimental studies on aerial lime mortars [7, 11, 37]. Indeed, the percentage differences between the results obtained did not exceed 5% for the bulk density and the ultrasonic pulse velocity, and about 10% for the flexural and compressive strength.

A similar conclusion has been reached as regards the repeatability of the results of specimens from different batches of mortar. Thus, Table 3 reports the

Table 3 Bulk density and ultrasonic velocities of the specimens used for flexural and compression tests at each age and of the reference specimens, with standard deviation and coefficient of variation within brackets

Age (days)	Batch	ρ (kg/m^3)	V (m/s)
7	4	1775 (11) (0.6%)	1239 (50) (4%)
	8	1785 (13) (0.7%)	1323 (6) (0.4%)
28	5	1603 (7) (0.4%)	1601 (41) (3%)
	8	1605 (19) (3%)	1655 (55) (1%)
60	6	1610 (4) (0.2%)	1598 (68) (4%)
	8	1616 (18) (1%)	1679 (78) (5%)
90	7	1621 (5) (0.3%)	1686 (20) (1%)
	8	1620 (18) (1%)	1738 (33) (2%)

mean values of ρ and V with the standard deviations and coefficients of variation reported in brackets for the specimens tested at different ages and for the reference specimens (i.e. the ones used for flexural and compression tests at 200 days as reported in Appendix). Indeed, the differences did not exceed 1% for the bulk density and 6% for the ultrasonic velocity.

3.2 Mechanical and physical properties

3.2.1 Flexural and compressive strength

Figure 6 reports the results of the strengths tests and their evolution in time. Note that the coefficients of variation are reported in brackets and the error bars in the graphs are the corresponding standard errors, calculated as the standard deviation divided by the square root of the number of experiments for each test.

Table 2 Results of the tests performed for hand compacted specimens and mechanically compacted specimens, with standard deviation and coefficient of variation within brackets

Batch	Age (days)	Compaction method	ρ (kg/m^3)	V (m/s)	f_f (MPa)	f_c (MPa)
3	5	Hand	1850 (8) (0.5%)	1208 (66) (5%)	–	–
		Mechanical	1876 (15) (0.8%)	1229 (29) (2%)	–	–
	6	Hand	1797 (16) (0.9%)	1017 (39) (4%)	–	–
		Mechanical	1835 (16) (0.9%)	1023 (52) (5%)	–	–
	7	Hand	1741 (7) (0.4%)	1067 (45) (4%)	0.20 (0.01) (3%)	0.62 (0.08) (13%)
		Mechanical	1786 (11) (0.6%)	1023 (37) (4%)	0.18 (0.01) (3%)	0.57 (0.02) (3%)
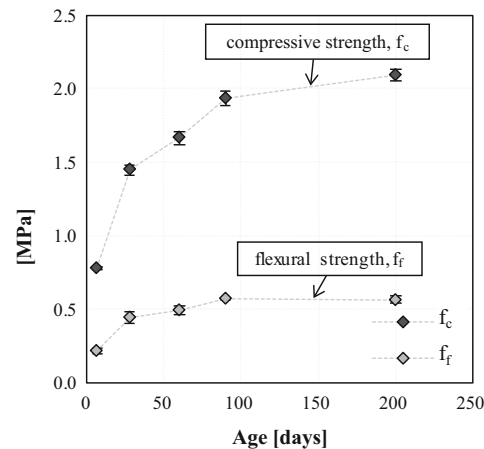
Age [days]	f_f [MPa]	f_c [MPa]
7	0.22 (14%)	0.78 (4%)
28	0.44 (15%)	1.45 (6%)
60	0.49 (9%)	1.67 (7%)
90	0.57 (0.2%)	1.93 (6%)
200	0.56 (8%)	2.10 (4%)

The flexural strength varied from 0.22 to 0.56 MPa and the compressive strength varied from 0.78 to 2.10 MPa. The tests showed similar development of flexural and compressive strength. Indeed, in both cases a substantial increase was showed between 7 and 28 days, then the increase was almost constant between 28 and 90 days, and, finally, the increase was small between 90 and 200 days, with the flexural strength almost constant. These results indicated that the studied mixture developed its flexural and compressive strengths mainly in the first three months, then it slowly increased its compressive capacity up to six months. Similar trends were found in other studies on aerial lime mortars [8, 10, 11].

3.2.2 Open porosity

The mean values of open porosity along with the coefficients of variation at each age are reported in Table 4. No significant variation of the open porosity with the time was detected, except for a slight reduction detected at 200 days. The mean value of the open porosity resulted in 39.6%, with CoV of 0.8%. However, a slightly decreasing trend was observed from 60 up to 200 days. These values and trends are consistent with the ones obtained on similar mortars by Faria et al. [44]. Mortars made with lime

putty typically have higher open porosity compared to mortars made with hydrated lime and lime-cement blended, due to the higher amount of water in such mixtures [12, 45]. Indeed, Cizer [12] found the open porosity of lime putty mortar at 90 days to be 4–7% higher than hydrated lime mortar for different curing conditions.

### 3.2.3 Ultrasonic pulse velocity and bulk density

The bulk density and ultrasonic velocity at each age for the reference specimens are reported in Fig. 7. Note that the coefficients of variation are reported in brackets and the error bars in the graphs are the corresponding standard errors.

The ultrasonic pulse velocity reduced between 5 and 6 days, then it increased up to 90 days and decreased again between 90 and 200 days. The higher velocity at 5 days compared to 6 days was probably related to the higher humidity of the specimens. Indeed, it is known that ultrasonic velocity is higher in water than in air, thus in water-saturated porous media [46]. After that, the increase up to 90 days, was probably related to the build-up of the solid skeleton. Indeed, it is also known that the ultrasonic velocity is higher in solids than in air [46]. Finally, the reduction

Table 4 Results of the open porosity tests

Age (days)	7	28	60	90	200
Open porosity	39.8% (0.8%)	39.4% (0.8%)	39.9% (0.5%)	39.2% (1%)	38.3% (0.5%)

Fig. 7 Evolution of the ultrasonic pulse velocity and bulk density
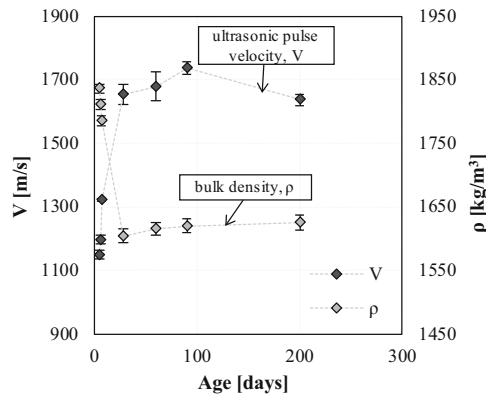
| Age [days]	ρ [kg/m³]	V [m/s]
5	1836 (0.7%)	1266 (2%)
6	1811 (0.7%)	1239 (2%)
7	1785 (0.7%)	1323 (0.4%)
28	1605 (1%)	1655 (3%)
60	1616 (1%)	1679 (5%)
90	1620 (1%)	1738 (2%)
200	1625 (1%)	1637 (2%)

of the velocity between 90 and 200 days was probably related to the loss of water in the specimens.

As expected the bulk density showed a decreasing trend until 28 days, due to the evaporation of water, then a slight increase until 200 days, due to the carbonation process with the formation of calcium carbonate in place of calcium hydroxide, with a higher molar mass compared to the latter [36]. This latter trend was consistent with the decrease observed for the open porosity and the subsequent increase of ultrasonic velocity.

Both for bulk density and ultrasonic velocity the maximum variation was observed between 7 and 28 days, similarly to the flexural and compressive strength.

3.2.4 Static elastic modulus

The mean static elastic modulus at each age is reported in Fig. 8, with the coefficients of variation in brackets and the error bars in the graphs corresponding to the standard errors. The elastic modulus increased

| Age [days]	E [GPa]
7	0.70 (3%)
28	0.80 (8%)
60	0.88 (7%)
90	1.08 (6%)
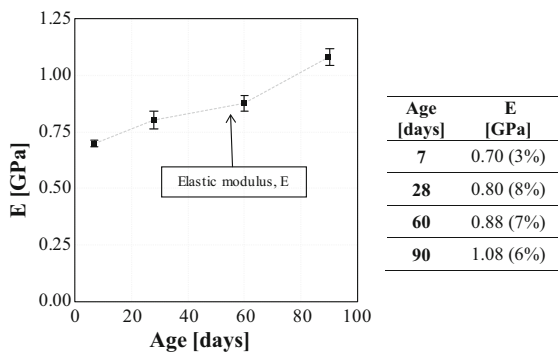
Fig. 8 Evolution of the elastic modulus with time

between 7 and 90 days from 0.70 to 1.08 GPa. Aggelakopoulou [6] found lower static elastic modulus for lime putty mortars with siliceous and calcareous sand at 18 months of curing (i.e. 0.11 and 0.13 GPa). These different results could be related to the different aggregates used in the mixtures (see also Sect. 4).

3.3 Differential thermal analyses

Figure 9 shows Differential Thermal Analysis thermograms obtained from the sample P1 of each cylindrical specimen (i.e. from the surface of each specimen) at each age. A heat flow peak related to the dehydroxylation was evident at three days, getting smaller at 7 days and almost flattened from 28 days,
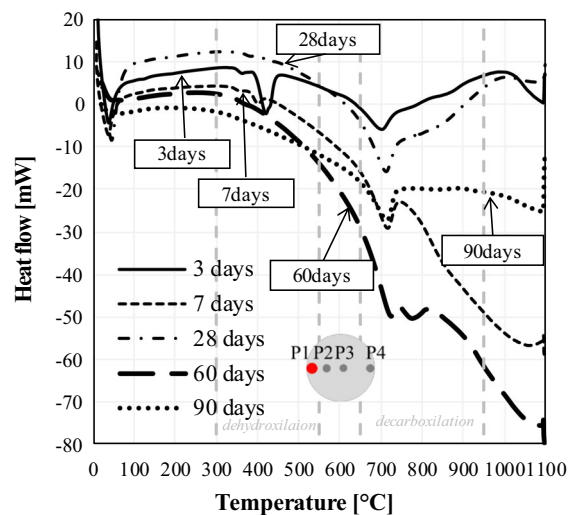
Fig. 9 Differential thermal analysis curves of samples P1 (i.e. on the surface of each cylindrical specimen) at each age

meaning that the calcium hydroxide on the surface of the specimens was completely consumed at that age. A heat flow peak in the range of decarboxylation was observed from 3 days, with an increase in $CaCO_3$ content, alongside the reduction of $Ca(OH)_2$, indicating the evolution of carbonation. Meanwhile, a reduction of free water due to evaporation was observed. Moreover, the weight loss in the temperature range of 50–250 °C from 3 days was further analyzed to investigate the occurrence of pozzolanic reactions related to the formation of hydrated phases [9, 12]. As no heat flow peaks were observed in that temperature range, this has led to conclude that such types of reactions were absent. Despite it was not possible to determine the reason for this unexpected outcome, this is an interesting result defining the behavior o this type of material.

As regards the evolution of carbonation through the depth of the cylindrical specimens, for the sake of simplicity, Differential Thermal Analysis curves obtained from the samples P1, P2, P3 and P4 in the first and the last ages (i.e. at 3 days and 90 days) are reported in Fig. 10. At 3 days, all the samples showed the heat flow peak in the range of dehydroxylation, while the heat flow peak in the range of decarboxylation was evident only for P1 and P4, meaning that at that age carbonation occurred only on the surface of the specimen (i.e. within a depth of about 12 mm). At 90 days, the heat flow peak in the range of dehydroxylation was observed in P2 and P3, while the peak in the range of decarboxylation was still not evident in P3. From that, it may be concluded that at that 90 days of age, carbonation was almost completed only at the edge of the specimen and was still in evolution within the core of the specimen. This was consistent with the results of thermogravimetric analyses by Lawrence et al. [47] and by literature values of carbonation depth in aerial lime mortar specimens evaluated through phenolphthalein [7, 8].

4 Integrative discussion of the experimental outcomes and comparison with literature results

A summary of the main mechanical and physical properties of the investigated mortar, with the coefficients of variation in brackets, is reported in Table 5.

Low values of mechanical parameters were found to be consistent with other lime-based mixtures for repair interventions of historical masonries, ranging about 1 MPa and 2.3 MPa for the compressive strength and 0.5 MPa and 0.6 MPa for the flexural strength after 3 months of curing [7, 10, 13]. In particular, the compressive strength was found to be compliant with the value targeted for lime-based mortar used in recent structural interventions at the Pompeii site (i.e. greater than 1.5 MPa at 28 days) [48]. The values found also comply with the
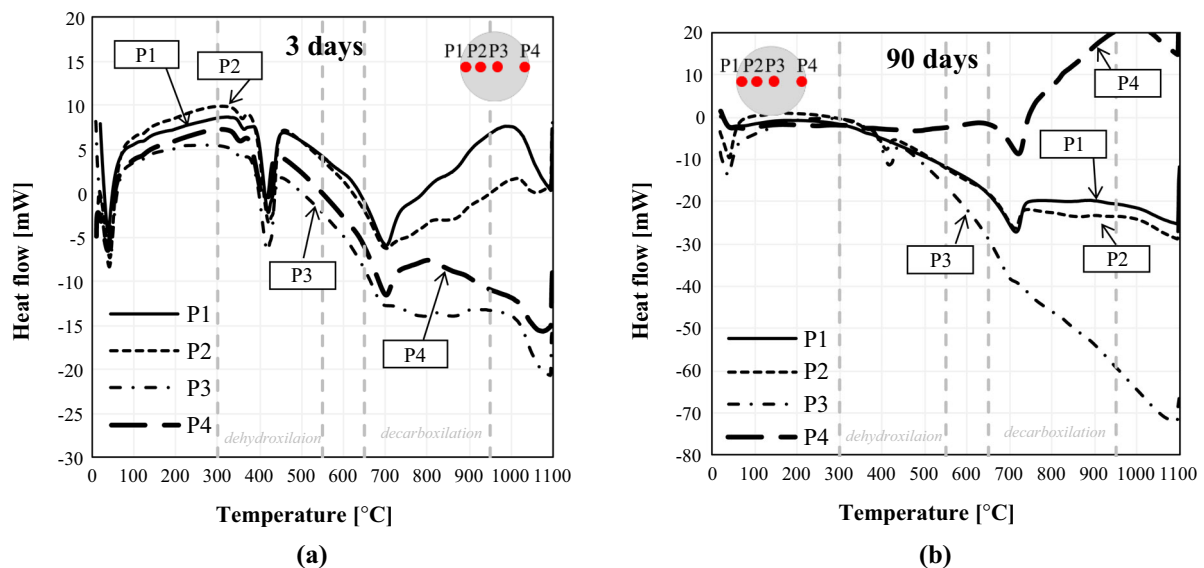
Fig. 10 Differential thermal analysis curves for all the samples at 3 days (**a**) and 90 days (**b**)

Table 5 Summary of the investigated physical and mechanical properties

Age (days)	f_f (MPa)	f_c (MPa)	Open porosity (%)	ρ (kg/m^3)	V (m/s)	E (GPa)
5	–	–	–	1836 (0.7%)	1266 (2%)	–
6	–	–	–	1811 (0.7%)	1239 (2%)	–
7	0.22 (14%)	0.78 (4%)	39.8% (0.8%)	1785 (0.7%)	1323 (0.4%)	0.70 (3%)
28	0.44 (15%)	1.45 (6%)	39.4% (0.8%)	1605 (1%)	1655 (3%)	0.80 (8%)
60	0.49 (9%)	1.67 (7%)	39.9% (0.5%)	1616 (1%)	1679 (5%)	0.88 (7%)
90	0.57 (0.2%)	1.93 (6%)	39.2% (1%)	1620 (1%)	1738 (2%)	1.08 (6%)
200	0.56 (8%)	2.10 (4%)	38.3% (0.5%)	1625 (1%)	1637 (2%)	–

requirement for mechanical compatibility with ancient mortars [13, 18]. Moreover, according to what was found by Faria et al. [44], the use of the lime putty in the mixture and the relatively high value of open porosity obtained may positively affect the durability of the mortar in terms of salt resistance, other than its workability. As regards the ultrasonic pulse velocity, despite it did not provide a direct correlation with the physical and mechanical properties of the mixture, its development trend was found to be a useful complementary method to follow the general development of the hardening process being consistent with the evolution of the hardened properties of the mortar.

Flexural and compressive strengths showed a similar evolution with the time. The values of the flexural strength were compared with the compressive strength at each age as reported in Fig. 11a. This could be a useful tool for a primary estimation of one of these parameters, known the other, being the flexural strength approximately equal to 30% of the compressive strength. A similar correlation was found by Haach et al. [49] for lime-cement blended mortars with different lime:cement proportions and type of aggregates.

Moreover, the compressive strength obtained at each age from the standard prisms' halves (i.e. the P-type specimens), f_c, were compared with the ones obtained from the cylindric specimens (i.e. Cyl$_A$-type), indicated as "f_c^*", as reported in Fig. 11b and the relevant table. This correlation is particularly interesting from an engineering point of view since it is well known that the compressive strength of concrete and mortars is affected by the shape and size of the tested specimens. The ratio f_c^*/f_c was found to vary between 0.45 and 0.60. This was consistent with what found by Parsekian [50] according to whom mortars with a strength less than 4 MPa show a reduction of the strength of cylindrical specimens compared to prism halves between 37 and 49%, which is much lower than the usual relation adopted for concrete to relate cube and cylinder specimens.

The evolution of the stiffness of a mortar (elastic modulus) compared to the evolution of its load capacity (compressive strength) could be taken as an indicator of the development of its capacity to accommodate larger movements without cracking and could be useful for comparisons with other mortars. Indeed, mortars with lower values of the

Fig. 11 Relationship between the mean compressive strength obtained from prism halves, f_c, and the mean flexural strength, f_f, (**a**) and between the mean compressive strength f_c and the mean compressive strength obtained from cylindric specimens, f_c^*
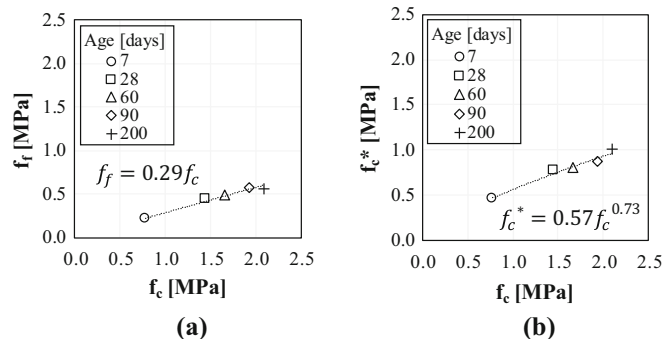
Age [days]	f_f/f_c	f_c^*/f_c
7	0.28	0.60
28	0.31	0.53
60	0.30	0.48
90	0.30	0.45
200	0.27	0.48

ratio E/f_c may have a lower probability of cracking, which could be crucial in repair interventions, especially for constructions in archaeological sites which are typically characterized by a low strength capacity and high deformability. In the present study, the ratio E/f_c was found to vary in the range 900–500, with the highest value at 7 days, as shown in Fig. 12. The figure also shows the comparison with the available data derived by literature concerning different types of mortar that can be possibly used in restoration. In particular, data related to cement-aerial lime blended mortars [45], natural hydraulic lime mortars [37] and cement-natural hydraulic lime blended mortars [49] were used for the comparison. Figure 12 also specifies the mix proportions for each mortar (Cement:Lime:Sand) and the water:binder ratio by volume. Note that as regards data from Haach et al. [49], mixtures made with fine coarse sand were considered. The mortar investigated here showed lower values of the ratio E/f_c compared to the other mixes. This provides quantitative evidence of the higher suitability of the investigated mortar for the restoration of ancient masonry structures.

5 Conclusions

In this study, a comprehensive characterization of a restoration mortar for ancient structures is presented. The goal of the study is the definition of indications for appropriate interventions on archaeological constructions. A mortar made with lime putty and pozzolan from the Phlegrean area, a volcanic region next to the Bay of Naples where the ancient Roman builders obtained their pozzolan (1:3 by volume) was investigated. The natural pozzolan used in the experiments is very precious and limitedly available and allowed to obtain an exclusive mortar that is very similar to the archaeological ones. The choice of the raw materials and the amount of water, the mixing procedure, the preparation and storage of the specimens are discussed in detail. The main physical and mechanical properties were investigated through standard methods at different ages from 5 up to 200 days, meanwhile, the hardening process was studied through Differential Thermal Analysis from 3 to 90 days at different depths from the surface of the cylindrical specimens. The experimental program involved 13 batches of mortar, 62 prismatic or cylindrical specimens and a total number of 201 destructive and non-destructive tests.

The main outcomes of this research can be summarized as follows:

- the mechanical properties of the mortar, specifically designed to be compatible with ancient structures, were similar to those of other types of lime-based mixtures for repair interventions of ancient masonries;
- the mean flexural and compressive strength obtained from standard prismatic (40 mm × 40 mm × 160 mm) were found to be 0.56 MPa (CoV 8%) and 2.10 MPa (CoV 4%) at 200 days;
- flexural and compressive strength showed a similar increasing pattern at different ages; the flexural
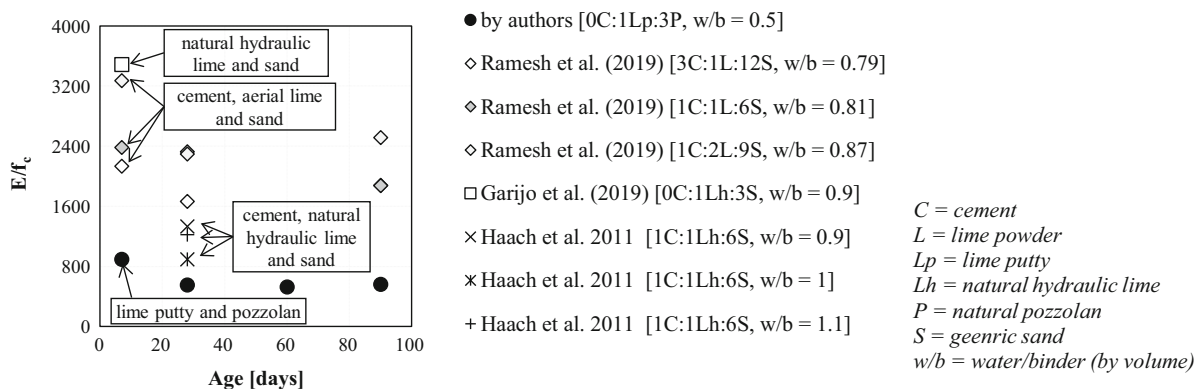
Fig. 12 Evolution of the ratio of elastic modulus to the compressive strength with time and comparison with values presented in the literature concerning different types of mixtures

strength resulted to be approximately equal to 30% of the compressive strength;

- the compressive strength obtained from cylindric specimens was found to be approximately equal to 45–48% of the compressive strength obtained from standard prism halves between 60 and 200 days;
- the values of the ratio between the elastic modulus and the compressive strength (E/f_c), which provides a rough evaluation of the capacity of the mortar to accommodate larger movements without cracking, ranged between 0.60 at 7 days and 0.48 at 200 days and resulted to be lower than other types of mortar (i.e. cement-aerial lime blended mortars, natural hydraulic lime mortars and cement-natural hydraulic lime blended mortar) used for masonry restoration; this makes the mortar under investigation more likely to mitigate cracking in restoration works;
- open porosity was not found to be significantly affected by the hardening process, with a mean value of 39.6%, CoV of 0.8%. The relatively high mean value was found to be consistent with similar mortars;
- the evolution of the ultrasonic pulse velocity was found to be a reasonable complementary method for the monitoring of the development of the hardened properties of the mortar;
- Differential Thermal Analysis analysis on the surface of the specimens (until about 12 mm) showed that the carbonation already started in that part from 3 days; calcium hydroxide was mainly consumed between 3 and 7 days, then the carbonation degree slowly increased until 60 days;
- Differential Thermal Analysis performed across the surface of the specimens showed that carbonation was still in evolution at intermediate depth (between approximately 12 and 24 mm) and in the core of the specimens at 90 days.

This study intended to provide a reliable framework for the design of structural interventions in archaeological sites based on the use of a mortar produced on-site from single raw materials. The designed mixture is intended to fulfill both the physical and mechanical compatibility with the archaeological structures and to comply with the traditional techniques found in ancient Roman cities in the area of Naples and similar archaeological contexts.

Declarations

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

Appendix: Experimental program matrix

Batch	Type	Curing	Test	Age (days)	No. of tests	Nomenclature of the test
1	FM	–	FT	–	1	1.FM.FT.1
2	FM	–	FT	–	1	1.FM.FT.2
3	P	A	BD	5	3	3.P.A.BD.5.1/2/3
			BD	6	3	3.P.A.BD.6.1/2/3
			BD	7	3	3.P.A.BD.7.1/2/3
			UPV	5	3	3.P.A.UPV.5.1/2/3
			UPV	6	3	3.P.A.UPV.6.1/2/3
			UPV	7	3	3.P.A.UPV.7.1/2/3
			FC	7	3	3.P.A.FC.7.1/2/3
	P-MC*	A	BD	5	3	3.P-MC.A.BD.5.1/2/3
			BD	6	3	3.P-MC.A.BD.6.1/2/3
			BD	7	3	3.P-MC.A.BD.7.1/2/3
			UPV	5	3	3.P-MC.A.UPV.5.1/2/3
			UPV	6	3	3.P-MC.A.UPV.6.1/2/3
			UPV	7	3	3.P-MC.A.UPV.7.1/2/3
			FC	7	3	3.P-MC.A.FC.7.1/2/3
4	FM	–	DTA	Within 2 h from t_0	4	4.FM.DTA.1
	P	A	BD	5	3	4.P.A.BD.5.1/2/3
			BD	6	3	4.P.A.BD.6.1/2/3
			BD	7	3	4.P.A.BD.7.1/2/3
			UPV	5	3	4.P.A.UPV.5.1/2/3
			UPV	6	3	4.P.A.UPV.6.1/2/3
			UPV	7	3	4.P.A.UPV.7.1/2/3
			FC	7	3	4.P.A.FC.7.1/2/3
	P	A	OP	7	3	4.P.A.OP.7.1/2/3
5	FM	–	DTA	Within 2 h from t_0	4	5.FM.DTA.2
	P	A	BD	28	3	5.P.A.BD.28.1/2/3
			UPV	28	3	5.P.A.UPV.28.1/2/3
			FC	28	3	5.P.A.FC.28.1/2/3
	P	A	OP	28	3	5.P.A.OP.28.1/2/3
6	P	A	BD	60	3	6.P.A.BD. 60.1/2/3
			UPV	60	3	6.P.A.UPV. 60.1/2/3
			FC	60	3	6.P.A.FC. 60.1/2/3
	P	A	OP	60	3	6.P.A.OP. 60.1/2/3
7	P	A	BD	90	3	7.P.A.BD.90.1/2/3
			UPV	90	3	7.P.A.UPV.90.1/2/3
			FC	90	3	7.P.A.FC.90.1/2/3
	P	A	OP	90	3	7.P.A.OP.90.1/2/3
8	P	A	BD	5	3	8.P.A.BD.5.1/2/3
			BD	6	3	8.P.A.BD.6.1/2/3
			BD	7	3	8.P.A.BD.7.1/2/3
			BD	28	3	8.P.A.BD.28.1/2/3
			BD	60	3	8.P.A.BD.60.1/2/3

Batch	Type	Curing	Test	Age (days)	No. of tests	Nomenclature of the test
			BD	90	3	8.P.A.BD.90.1/2/3
			BD	200	3	8.P.A.BD.200.1/2/3
			UPV	5	3	8.P.A.UPV.5.1/2/3
			UPV	6	3	8.P.A.UPV.6.1/2/3
			UPV	7	3	8.P.A.UPV.7.1/2/3
			UPV	28	3	8.P.A.UPV.28.1/2/3
			UPV	60	3	8.P.A.UPV.60.1/2/3
			UPV	90	3	8.P.A.UPV.90.1/2/3
			UPV	200	3	8.P.A.UPV.200.1/2/3
			FC	200	3	8.P.A.FC.200.1/2/3
	P	A	OP	200	3	8.P.A.OP.200.1/2/3
9	Cyl_A	A	C	7	3	9.CylA.A.C.7.1/2/3
	Cyl_A	A	CC	7	3	9.CylA.A.CC.7.1/2/3
10	Cyl_A	A	C	28	3	10.CylA.A.C.28.1/2/3
	Cyl_A	A	CC	28	3	10.CylA.A.CC.28.1/2/3
			CC	60	3	10.CylA.A.CC.60.1/2/3
			CC	90	3	10.CylA.A.CC.90.1/2/3
11	Cyl_A	A	C	60	3	11.CylA.A.C.60.1/2/3
12	Cyl_A	A	C	90	3	12.CylA.A.C.90.1/2/3
	Cyl_A	A	C	200	3	12.CylA.A.C.200.1/2/3
13	Cyl_B	B	DTA	3	4	13.CylB.B.DTA.3.1/2/3/4
	Cyl_B	B	DTA	7	4	13.CylB.B.DTA.7.1/2/3/4
	Cyl_B	B	DTA	28	4	13.CylB.B.DTA.28.1/2/3/4
	Cyl_B	B	DTA	60	4	13.CylB.B.DTA.60.1/2/3/4
	Cyl_B	B	DTA	90	4	13.CylB.B.DTA.90.1/2/3/4

*MC indicates that the specimens were mechanically compacted

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