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ORIGINAL ARTICLE

Phasing the history of ancient buildings through PCA on mortars' mineralogical profiles: the example of the Sarno Baths (Pompeii)

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Abstract
A total of 83 wall joint mortar samples collected from the Sarno Baths complex in Pompeii (Naples, Italy) were analysed by optical microscopy (OM) and X-ray powder diffraction-quantitative phase analysis (XRPD-QPA) in order to scan the ancient construction phases and modern restorations to which the building was subject. The major issue to overcome in the research depended on the fact that the most part of the analysed mortars was taken from undated structures, while only 35 were collected from dated ones. In order to observe correlations in sample distribution which could reflect ancient building phases and modern restorations, we then processed XRPD-QPA data of the mortars through principal component analysis (PCA). A rational subdivision of the full dataset into a smaller one before performing PCA was a useful step for a proper enucleation of coherent groups. The presence in most of the resulting groups of dated samples also allowed us to place in a precise timeframe the undated ones. This study demonstrates that our approach, integrating the traditional archaeological analysis with archaeometrical methods and statistics, could be adopted as a tool with which to frame the constructive episodes in other ancient buildings in Pompeii as well as at other archaeological sites.

KEYWORDS
multivariate statistics, PCA, petrography, Pompeii, pumice, Sarno Baths, volcanic tephrite, XRPD-QPA

STATE OF THE ART AND TARGET OF RESEARCH

The study of complex ancient buildings relies on the scan of ancient construction phases and modern restorations in terms of relative phasing and, when possible, absolute chronologies. Over the last decades, this was accomplished by integrating traditional archaeological analyses (stratigraphic sequences, building technique examination, pottery study) with analytical data on building materials and absolute dating methods (Mannoni & Boato, 2002).

The archaeometrical examination of mortar-based materials has been adopted as a way for sequencing constructive episodes of ancient or medieval buildings (Crisci et al., 2001, 2002). The fundamental assumption is that the definition of groups of mortars sharing similar composition should reflect different building interventions. Indeed, mortars represent a precise 'recipe' of raw materials (binder, aggregate, water) collected from different sources and mixed at the exact moment they have been employed.

Chemical or mineralogical quantitative data on mortars' compositional profiles are suitable to be treated by multivariate statistics (cluster analysis, principal component analysis—PCA) to highlight the samples' clustering. This methodology has aften been employed in the analysis of mortars from Roman buildings in the Vesuvian area (De Luca et al., 2015; Grave, 2002; Joosten, 1999; Miriello et al., 2010, 2018; Piovesan et al., 2013; Secco et al., 2019), as well as at other sites (De Luca et al., 2013; Gliozzo & Camporeale, 2009; Lezzerini & Giubbilini, 2011; Secco et al., 2018).

Less frequently, multivariate statistics has been applied on data from thermogravimetric analysis (TGA) and differential thermal analysis (DTA) (Leone et al., 2016), on hybrid datasets inclusive of thermal, mineralogical and mechanical data (Moropoulou et al., 2003) or on optical microscopy (OM) data, whose descriptive parameters have been quantified through image analysis (De Luca et al., 2013, 2015; Dilaria, 2020; Grave, 2002; Miriello et al., 2018). Other studies, in which the mortar grouping was only based on qualitative analyses, are less conclusive (Bonazzi et al., 2007; Demauro, 2020; Freccero, 2005; Frizot, 1983; Piovesan et al., 2009).

Some authors also argued that Pompeian mortars' typologies could be considered as indicative markers of specific periods (Joosten, 1999; Miriello et al., 2018). Besides this, a detailed atlas of the compositional features of ancient 'recipes' and modern restoration mortars of Pompeii and the Vesuvian sites is still lacking.

In this research, we analysed, through OM and X-ray powder diffraction-quantitative phase analysis (XRPD-QPA), 83 wall joint mortars from the Sarno Baths, a multistorey complex located in the southern part of *Regio* VIII (2, 17–23) in Pompeii (Naples, Italy), in order to resolve the uncertainties regarding the ancient construction phases and modern restorations to which the building was subject (Bernardi & Busana, 2019; Ioppolo, 1992; Kolowski-Ostrow, 1990). The analysis was completed in three steps. (1) Combining fresh stratigraphic data from recent excavations in the complex, archive photographic documentation and a detailed survey of the walls' constructive techniques, several wall samples were attributed to a specific period. (2) Mortars' XRPD-QPA data were processed through PCA to observe correlations in sample distribution that could reflect ancient building phases and modern restorations. A rational subdivision of the full dataset into a smaller one before PCA was a useful step for the identification of coherent groups. The presence in most groups of dated samples also allowed us to place in a precise period the undated ones. (3) The compositional characteristics of mortar groups were described in detail, according to the results of petrographic and mineralogical analyses.

By this procedure, a series of groups of mortars, which truthfully reflect the numerous constructive and restoration activities of this Roman building throughout the centuries, were defined.

THE SARNO BATHS: ARCHAEOLOGICAL BACKGROUND

The so-called Sarno Baths are a large Roman spa located on the south-western edge of the volcanic plateau of Pompeii (Figure 1, a), in *Regio* VII, *insula* 2, south of the Forum (Figure 1, b). After being buried under the volcanic lapilli of the 79 CE eruption, the building was excavated for the very first time between 1887 and 1890, while its facade was brought to light in the 1950s (Bernardi & Busana, 2019). The quarter occupied by the complex was already used during the Archaic and Samnite periods. Later, during the Late Republican Age (second century BCE), numerous terraced *domus* were built in this area (Carafa, 1997; Zanella, 2012, 2013). In its last building phase, the complex extended over an area of about 3700 m^2, and it was structured into four floors sloping down the volcanic plateau (see Figure S1 in the additional supporting information) and supporting the ground floor accessible from the urban streets, while the upper floor (level $+1$) was not preserved.

The ground floor (level $+0$) consisted of three main units with independent entrances and connected to each other: (1) the western one at number 18 of Via delle Scuole presented the traditional sequence of *fauces*, *atrium* and *tablinum* (*a–c*); (2) the eastern one at number 21 on Vicolo Regina was constituted of an entrance, *atrium*, *tablinum* and probably a panoramic terrace (*G–L*); and (3) the third unit, located between the previous two and accessible through the wide corridor at number 20, was planned around a small peristyle (*n*). Level -1 was composed of cisterns and small service rooms opening onto a vaulted corridor (*VI*). Level -2 was occupied by three panoramic residential apartments connected through a decorated cryptoporticus (ι). Level -3 hosted the baths and seven small equally sized rooms with uncertain destination, opening onto a windowed corridor. Level -4 included five differently sized rooms probably with a service function. At this level, the facade of the complex presents a door facing the Sarno Valley out of the city (Bernardi et al., 2019).

In the course of the 19th and 20th centuries, restoration activities, not properly documented, were carried out on the building, compromising on some occasions its original planimetric articulation. In past years, various attempts were made to phase the building history of the baths chronologically based on its planimetric–architectural features (Ioppolo, 1992; Kolowski-Ostrow, 1990; Noack & Lehmann-Hartleben, 1936), without reaching conclusive results.

Between 2016 and 2017, the investigations into the Sarno Baths were renewed by an interdisciplinary team of the University of Padua to perform a complete analysis of the building (Artioli et al., 2019; Maritan et al., 2019). The research later continued in collaboration with the Archaeological Park of Pompeii. New excavations, performed between December 2018 and January 2019, in the *domus* at number 21 and the basement of the facade provided fresh data to retrace the early stages of construction of the complex. In particular, according to stratigraphy, different building episodes were preliminarily recognized at number 21:

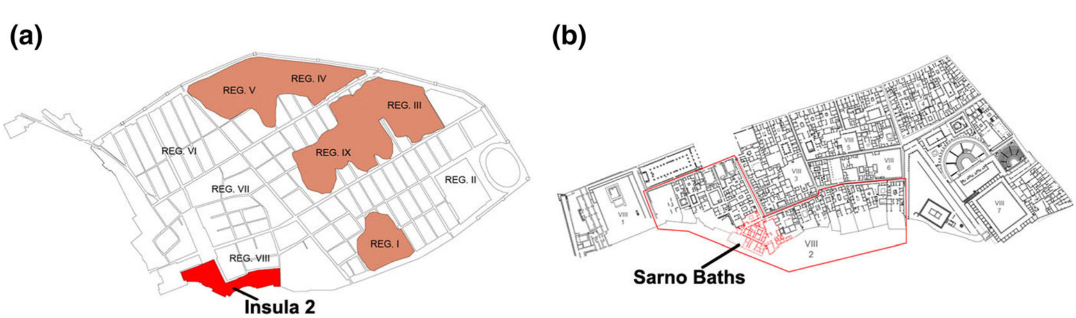

FIGURE 1 (a) Plan of Pompeii showing *Regio* VIII, *insula* 2 (marked in red) within the ancient city; and (b) the Sarno Baths within *insula* 2 (marked with red lines) (modified after Bernardi & Busana, 2019)

- Phase I refers to wall structures detected within a small trench opened in the south-western wing of the *domus*.
- Phase II relates to a series of wall structures and floors probably attributable to two adjacent houses, each provided with a cistern for collecting water.
- Phase III includes the *domus* currently visible at number 21, which was built on the levelling of the previous houses and on the filling of a large pit in the *atrium* of one of the pre-existing houses.

At the current state, the bulk finds collected from the filling of the pit set a *terminus post quem* for the building phase III *domus* during the Augustan age (late first century BCE–early first century CE). Therefore, building phases I and II certainly antedate this moment. However, even though they were distinguished based on stratigraphic relationships, it cannot be excluded that they could refer to two consecutive activities in the development of a unique constructive episode. The complete analysis of the materials collected during the 2018–19 excavations will provide new data to further investigation on the exact chronologies of building phases I and II.

SAMPLING AND SELECTION OF THE ANALYTICAL DATASET

Due to its long and complex history, the Sarno Baths result in an intricate twist of ancient and modern structural elements. The limited extension of stratigraphic excavations and the absence of exhaustive data on 19th- and 20th-century interventions (Bernardi & Busana, 2019), which frequently were accomplished philologically by reusing Roman construction materials and techniques, make the attribution of most of the structures to a specific period difficult.

A former analysis on Sarno Baths' mortars (Secco et al., 2019) collected from the facade and the inner floors (levels −3 and −4), presented a preliminary distinction between mortar types based on their main compositional features. However, there were not enough chronological data to connect each group to a precise historical period.

The excavations executed in 2018 and 2019 provided the occasion to extend the mortar sampling to the structures of level +0 and to the foundations of the facade. At the same time, the study of building techniques and materials and the examination of 20th-century photographic documentation eased the assessment of good chronological constraints for a series of mortar samples collected from other structures of the complex. In this way, most of the 2018–19 mortars were dated, together with some of those already described by Secco et al. (2019).

In this research, we considered for analysis only 83 wall joint mortars (Figure 2) to avoid improper comparisons with mortars from structures having different functions (i.e., revetments or floors).

Most of the samples come from undated structures, but thanks to recent excavations, researches on archive photographic documentation and a detailed survey of wall techniques, we were able to attribute some of them to a specific timeframe. In detail, 19 mortars are surely attributable to ancient building phases. In particular, 10 are dated to the early first century CE (building phases I and II); five between the beginning of the first century CE and 79 CE (building phase III); four samples can be surely ascribed to the Roman period. In addition, other 13 samples can be related to the Roman period with less confidence. Finally, one sample at level +0 relates to a modern restoration of the upper portion of the south-eastern perimeter of the *atrium* at number 18. This wall was surely reconstructed after the 1930s, as in a photograph published in 1936 its upper part was missing (Noack & Lehmann-Hartleben, 1936) (see Figure in the additional supporting information); two other samples from level −3 and its facade were attributed to restoration activities too. In detail, sample M159 is surely linked to an intervention carried out at the end of the 19th century, as proved by the thermoluminescence analysis of a brick

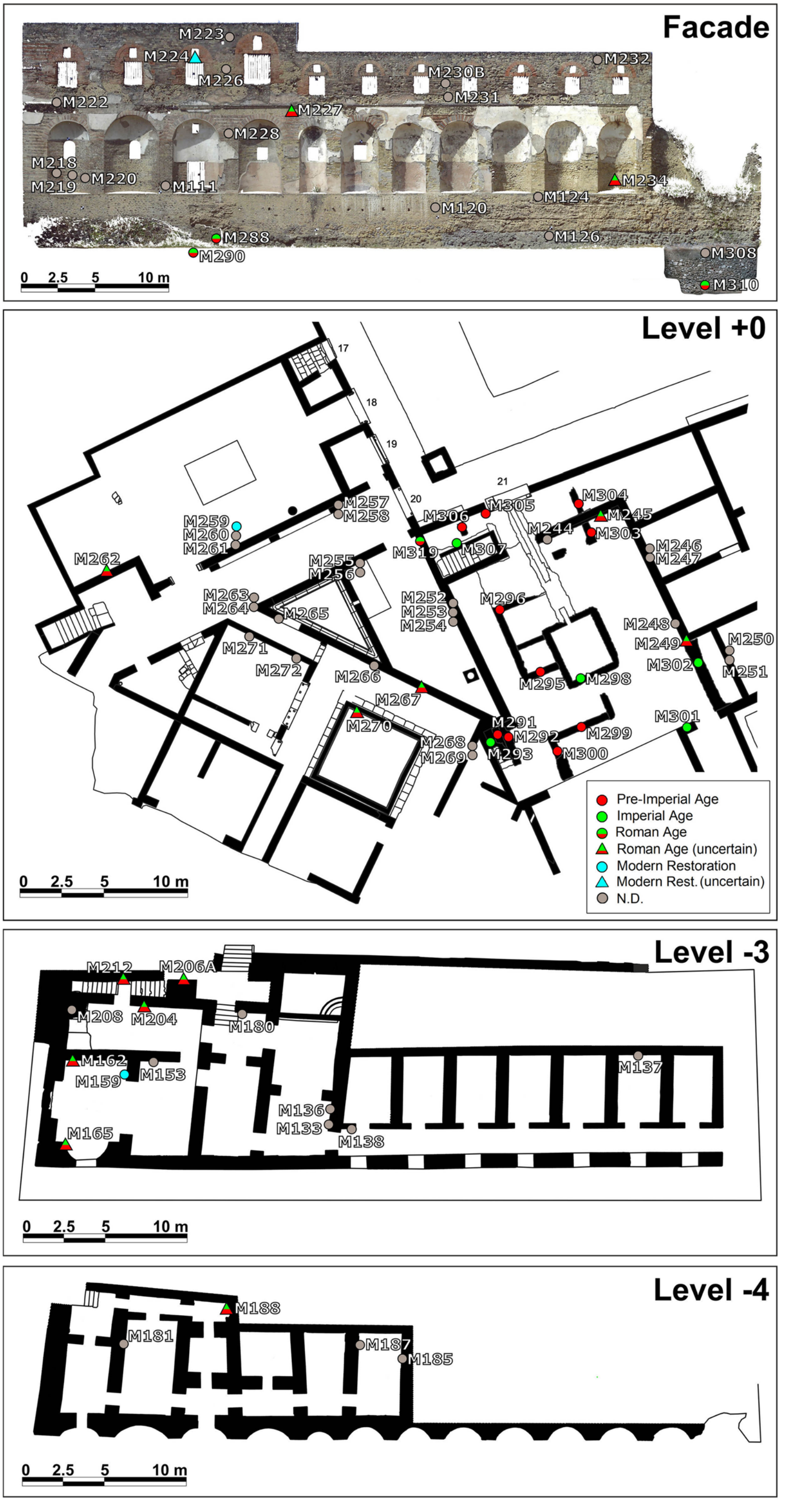

FIGURE 2 Scaled elevation and plans of the distribution of the analysed wall joint mortars with indications, when possible, of their chronology (levels: facade, +0, −3 and −4)

fragment taken from the wall (Secco et al., 2019). The remaining mortars collected from the Sarno Baths remained undatable.

All dated samples were considered as markers of the several ancient and modern constructive episodes of the building, and their compositional characteristics were taken as terms of comparison with connect the undated samples to a specific timeframe.

DATA ANALYSIS

Archaeometrical methods

All 83 mortar samples were investigated through XRPD-QPA (see Table S1 in the additional supporting information). Data were collected using a Bragg–Brentano θ-θ diffractometer (PANalytical X'Pert PRO, Cu Kα radiation, 40 kV and 40 mA) equipped with a real-time multiple strip (RTMS) detector (PIXcel by PANalytical). Data acquisition was performed by operating a continuous scan in the range 3–85 [°2θ], with a virtual step scan of 0.02 [°2θ]. Diffraction patterns were interpreted with X'Pert HighScore Plus 3.0 software by PANalytical, qualitatively reconstructing mineral profiles of the compounds by comparison with Powder Diffraction File (PDF) databases from the International Centre for Diffraction Data (ICDD). Quantitative phase analysis (QPA) was then performed on the sole bulk samples using the Rietveld (1967) method. Refinements were carried out with TOPAS software (v. 4.1) by Bruker AXS (Advanced X-ray Solutions). The determination of both crystalline and amorphous content was calculated by the addition of 20 wt% of zincite to the powders as an internal standard. The observed Bragg peaks in the powder patterns were modelled through a pseudo-Voigt function, fitting the background by a 12-coefficient Chebyshev polynomial. For each mineral phase, the lattice parameters, Lorentzian crystal sizes and scale factors were refined. Although samples were prepared with the backloading technique to minimize preferred orientation of crystallites a priori, during the refinement, any residual preferred orientation effect was modelled with the March Dollase algorithm (Dollase, 1986). The starting structural models for the refinements were taken from the International Crystal Structure Database (ICSD).

The final mineralogical profile of the samples was outlined after the removal of alteration mineral phases, such as gypsum produced by atmospheric weathering of exposed mortars (Leone et al., 2016; Miriello et al., 2010; Sabbioni et al., 2001). The percentiles of the remaining original mineral phases were then normalized at 100% wt (see Table S2 in the additional supporting information). The possible presence of newly formed calcite and analcime as secondary alteration in humid environments (Sánchez-Moral et al., 2005) was considered, but our analyses suggest the correlation with zeolitization of leucites and volcanic tephra for analcime (Ghiara et al., 1999) and with binder-rich mortars for calcite, as described in the results.

Mineralogical analyses were coupled with a detailed petrographic study of mortars (Pecchioni et al., 2014) to confirm the compositional characteristics of samples and to enhance indicative features that cannot be described via XRPD (grain size distribution, porosity, rock-type morphology and texture). The study was performed through a Nikon Eclipse ME600 optical microscope working on transmitted light (TL-OM) and by analysing 30 μm-thin sections, obtained by vacuum-impregnating portions of the materials with epoxy resin and sectioning them transversally.

Statistical treatment

To identify and interpret correlation patterns of samples' mineralogical profiles, XRPD quantitative data were subjected to a treatment by multivariate statistics.

Mortars' descriptive mineral phases were treated via PCA to obtain a small number of linear combinations that adequately describe the original mortar mineralogical profiles. This statistical method extracts a series of principal components that represent the dataset's variability. The sample distribution was reported in a scatterplot according to the value of the first two extracted components (PC1, PC2), as the first two components are those describing the main variance of the dataset, according to the eigenvalues (see Table S3 in the additional supporting information).

To identify groups of mortars relatable to chronologies, a congruent sub-dataset of samples from level +0 and the excavation of the facade foundations was first analysed, because these mortars present better chronological constraints. The PCA on the full sample dataset was then performed.

RESULTS

The PCA performed on the sub-dataset of samples allowed the identification of six groups, whose distribution aligns with the chronologies of the dated samples (Figure 3, a–b).

Samples having both PC1 and PC2 < 0 are ascribed to Gr 1. They are characterized by the strong positive correlation among quartz, sodalite, leucite and dolomite. Two subgroups can be recognized in this quadrant, defined as Gr 1a and Gr 1b.

As detected by XRPD, with respect to all the other samples, Gr 1a mortars are characterized by a pronounced presence of calcite, which is primarily referable to the binder, as revealed by OM investigations. In some mortars, quartz represents an important aliquot ($> 4\%$) compared with the other samples of the dataset. As observed by OM, the aggregate fraction is low and composed of clinopyroxenes of the augite type, plagioclases (labradorite/bytownite), K-feldspars (sanidine) and sporadic leucite-rich rocks. In most of the samples of this group, numerous porphyritic trachyte clasts with feldspar microliths (Kastenmeier et al., 2010; Piovesan et al., 2019) are present.

Gr 1b mortars report the highest concentration of leucite ($> 10\%$) of the dataset detected by XRPD. In fact, OM analysis demonstrated that the aggregate fraction is composed of prevalent leucite-rich volcanic rocks (see Figure S3, a, in the additional supporting information) and tephra (pumices and scoria) coupled with scattered single leucite minerals. A subordinate aggregate fraction is composed of plagioclases, augitic clinopyroxenes and K-feldspars. The quartz component is low.

The presence of the modern restoration sample M259 in Gr 1b suggests considering Gr 1a mortars, entirely represented by undated samples, as related to a restoration activity.

Gr 2 includes all building phase III samples and some generic Roman period samples plotting at PC2 > 0. It can be subdivided into two subgroups, defined as Gr 2a and Gr 2b.

Gr 2a reunites samples having PC1 < 0. As detected by XRPD, in these mortars the quartz fraction is low (around 1–2%), while the amorphous phase is high (usually between 35% and 50%). This latter phase is related to the abundant occurrence of volcanic tephra (pumices and scoria) as demonstrated by OM analysis (see Figure S3, b, in the additional supporting information). Grey tuff clasts of the Campanian ignimbrite (CI) *facies* (39 k.a. BP) and yellow tuff clasts of the CI or Neapolitan yellow tuff (NYT) (12 k.a. BP) *facies* (Kastenmeier et al., 2010; Piovesan et al., 2019) are diffused in the analysis of thin sections (see Figures S3, c–d, in the additional supporting information). Crystals of clinopyroxenes of the augitic type and scattered plagioclases (labradorite/bytownite) are attested in low aliquots as well as sporadic clasts of vesicular phonolite tephrites with hypohyaline texture (Kastenmeier et al., 2010; Piovesan et al., 2019).

Gr 2b groups samples having PC1 > 0. The mortars of this group are similar to those of Gr 2a, but XRPD data report that the amorphous fraction is slightly lower than in Gr 2a (around

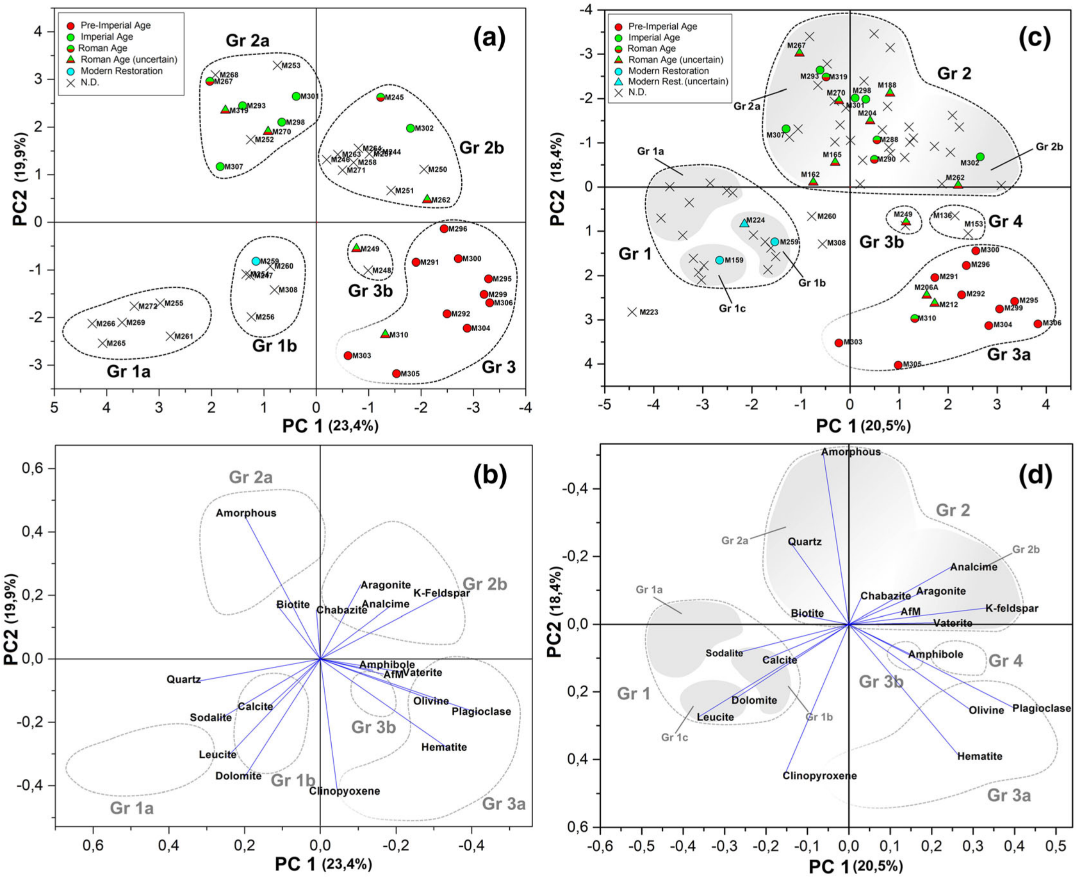

FIGURE 3 Principal component analysis (PCA) distribution of the mortar samples based on X-ray powder diffraction-quantitative phase analysis (XRPD-QPA) profiles: (a) Distribution and groups of the sub-dataset of samples from level +0 and from the recent excavation of the facade; (b) influence weight of the mineral phases, shown as vectors, based on the sub-dataset considered for PCA at point a; (c) distribution and groups of the full dataset of mortars; and (d) influence weight of the mineral phases, shown as vectors, based on the full dataset considered for PCA at point c

30–35%) and the concentration of analcime is pronounced in comparison with the other samples (4.5–7.0%). This phase is probably linked to the natural 'analcimization' of leucite and volcanic tephra (Ghiara et al., 1999). Moreover, with respect to mortars of Gr 2a, we observed by OM a richer concentration of pyroxenes (augite), olivine and volcanic clasts with K-feldspars microlites (sanidine).

Gr 3 includes mortars having PC1 < 0 and PC2 < 0, and it is further divided into two subgroups.

Gr 3a comprises all building phases I–II samples and the generic Roman period sample M310. These mortars are characterized by PC1 > 0.5 and PC2 < 0. XRPD reports a high concentration of plagioclases (labradorite–bytownite) and hematite while the amorphous fraction is lower than in Gr 2a and 2b. As detected via OM, these phases are related to the clasts of dark red vesicular phonolite tephrites (see Figure S3, e, in the additional supporting information), sometimes displaying large phenocrysts of augite, which represent the main coarse aggregate in these mortars. A secondary fraction of the aggregate is composed of large single crystals of clinopyroxenes (augite) and olivine. Volcanic tephra and tuff clasts are sporadic.

Two samples fall on the edge of this group: M303, reporting by XRPD a high concentration of clinopyroxenes (> 30%); and M305, which is richer in leucite (> 8.0%) with respect to other samples of Gr 3a.

Gr 3b is represented by the sole samples M248 and M249, having PC1 and PC 2 around −1. M249 is dated to the Roman period. Both samples can be distinguished by OM for the binder fraction having a dark interference colour, diffused porosity and lower presence of clasts of vesicular tephrite with respect to Gr 3a mortars. Finally, the distribution of large lime lumps and calcination relicts demonstrates an inadequate mixing.

The groups of samples from level +0 and from the excavation of the facade, defined after the statistical treatment of the XRPD profiles, matched with OM observation, reveal a good correspondence with the chronologies of dated mortars. Therefore, this pattern was used for sequencing even the undated samples taken from the inner levels −3 and −4 and from the exposed facade, already analysed by Secco et al. (2019).

The PCA performed on XRPD data of the full dataset reports a sample distribution that matches the previous one (Figure 5, c–d). All the samples related to modern restorations fall in Gr 1 (PC1 < −1.0; PC2 < −0.5), but the distinction of subgroups Gr 1a and 1b is less marked than before. Moreover, a third subordinate concentration of samples, labelled Gr 1c, can be detected inside Gr 1.

Gr 1c mortars, including modern sample M159, are characterized by the results of XRPD by the presence of abundant clinopyroxenes and, in particular, of compact leucite-rich rocks. On the other hand, compared with Gr 1a and 1b, the concentration of the amorphous fraction is lower. This depends on the low presence of volcanic tephra, as indicated by OM analysis.

Three samples (M223, M260 and M308) are outliers inside the quadrant at PC > 0 and PC 1 < 0. XRPD data demonstrate that M223 is different from all the other samples due to the abundance of dolomite (> 5.0%). This could be due to the use of a dolomitic lime component, as revealed by OM analysis.

M260 can be isolated for the scattered presence of reused mortar fragments, as observed via OM analysis (see Figure S3, f, in the additional supporting information).

Finally, M308 can be easily distinguished by OM for the unusual occurrence of single large crystals of leucite (see Figure S3, g, in the additional supporting information), which were documented in M305 of Gr 3a, too. Therefore, it cannot be excluded that M308 could be somehow related to Gr 3a.

Building phase III samples and most of the generic Roman period samples fall into Gr 2 (PC1 > −2.0 and PC2 < 0), whereas the distinction between Gr 2a and 2b samples is not as pronounced as before.

Gr 3a clusters all the samples of the building phases I and II, having PC1 > −0.5 and PC2 > 1.5. Samples M206A and M212, collected from level −3 and already described by Secco et al. (2019), perfectly fall within this group.

Two very close pairs of samples fall between Gr 2b and 3a: samples M248 and M249 (Gr 3b), having PC1 and PC2 values close to 1.0; and a second pair of undated samples (M136 and M153), defined as Gr 4, dropping in between Gr 2b and 3a. On the basis of their distribution in the scatterplot, they could be attributed to the Roman period because they are close to all the other pre-modern era samples. As already described by Secco et al. (2019), they differentiate from all the other samples for their high content of K-feldspars detected by XRPD (38% in sample M153). In fact, OM investigations demonstrated the presence of K-feldspar bearing volcanic rocks (see Figure S3, h, in the additional supporting information) and single scattered minerals of sanidine.

In conclusion, the results of the analysis allowed the definition of eight groups of mortars, coherent with the chronologies of dated samples, and three outliers (undated). It is clear that some mineralogical phases are distinctive for some mortars types, while others are less indicative, such as the newformed phases related to pozzolanic reaction of the samples, affecting most part of the mortars. These are the AFm, in the form of hydrocalumite and hydrotalcite, which are crystalline C-A-H hydrates (Matschei et al., 2007) occurred as a consequence of the reaction between the lime binder and the pozzolanic clasts (i.e., volcanic tephra) in the mortars. Vaterite and aragonite are correlated to AFm as they represent metastable anthropogenic transitional products formed after decalcification and recarbonation of calcium carbonates along with the pozzolanic reaction of the mortars (Jackson et al., 2017; Morandeau et al., 2014).

Mineralogical spectra and petrographic characteristics of representative samples for each group are reported in Figures 4 and 5, and see Table S4 in the additional supporting information.

DISCUSSION

As reported in the previous section, the presence, within the groups, of dated samples links most of the groups to a timeframe. In detail, five groups can be related to ancient building phases (Gr 2a, 2b, 3a, 3b and probably 4) and three groups (Gr 1a, 1b and 1c) to modern restorations.

Among the ancient ones, mortars of Gr 3a are related to the older building phases of the complex (building phases I and II), whose *terminus ante quem* is the Augustan age.

No compositional differences have been observed between building phase I and II mortars. Both of these are characterized by the large presence of phonolitic tephrite aggregates of the Somma–Vesuvio eruptive unit (Langella et al., 2009; Santacroce, 1987), a pillow lava outcropping near Pompeii (Di Girolamo, 1968), which was also employed in the making of the walls from which the mortars derive. These structures are made of compact phonolitic tephrite, the so-called *pietra lavica* (Baiguera, 2007; Piovesan et al., 2019) or its vacuolar *facies*, defined as *cruma di lava*, *lava-schiuma* (Baiguera, 2007; Di Girolamo, 1968; Frizot, 1983) or *foam lava* (Di Girolamo, 1968). These lithotypes were largely employed as constructive materials in Pompeian Republican buildings (Dessales, 2011).

The distribution of Gr 3a samples within the complex (Figure 6) suggests that, before the Augustan age, the space at number 21 was inhabited, and that at number 17 there was a multistorey building sloping down towards the Sarno Valley. As only hypothesized by Secco et al. (2019), this assumption can be now proved by the inclusion in Gr 3a of samples M206A and M212, coming from the internal wall at level −3 that leaned against the rocky outcrop. In addition, the attribution of M310 to Gr 3a may suggest that, already in this period, the substructures of this building could have extended up to the existing facade of the Sarno Baths.

With regard to Gr 2a and 2b mortars, they can both be dated between the Augustan age and 79 CE (building phase III). Even without specific chronological data, the wall stratigraphic relationships and their spatial distribution suggest that Gr 2b mortars could be more recent than Gr 2a mortars.

The compositional difference of Gr 2a and Gr 2b compared with Gr 3a consists in a more pronounced presence (in particular in Gr 2a) of volcanic tephra, comprising pumices and scoria clasts. Tuff fragments in mortars, relatable to the two major Phlegrean eruptive products of the CI or NYT *facies*, likely represents the reuse of chips from building blocks leftover during construction. These lithotypes were widely employed as building stones in some of the walls from which Gr 2a and 2b samples derive (Piovesan et al., 2019). Geochemical analyses are required to properly define the geological *facies* of pumices and scoria, which could be probably the same as the tuffs.

Gr 2a samples appear homogeneously distributed in the complex (Figure 6): they can be found at level +0, as well as at levels −3 and −4 and in the facade. On the other hand, Gr 2b samples are concentrated at level +0, with seven exceptions: three samples coming from the facade (M111, M231 and M288), one (M290) from a collapsed part of the facade found during recent excavations (Furlan et al., 2019), and three from level −3 (M137, M180 and M208). At

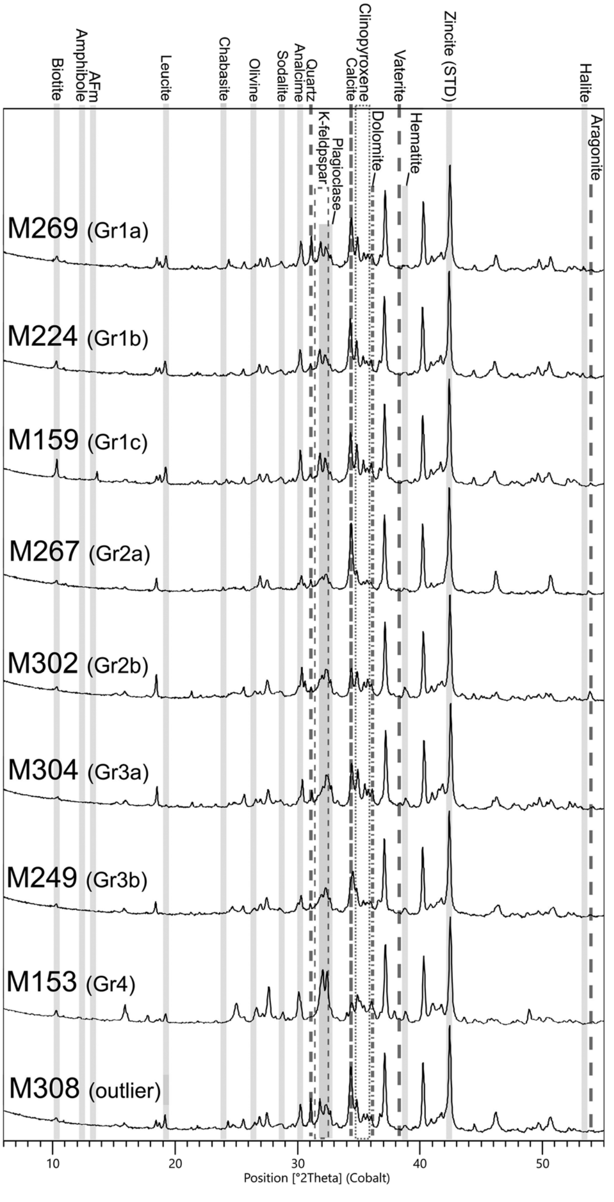

FIGURE 4 Mineralogical spectra of representative samples for each group, showing the d-spacing of the principal peak of all mineral phases other than amorphous. For those mineral phases overlapping their main peaks, an indicative secondary peak has been highlighted (i.e., aragonite, halite)

level +0, Gr 2b mortars are concentrated in the eastern part of the *atrium* at house number 21 and along the south-eastern limit of the *atrium* at house number 18. This distribution suggests that this type of mortar could be related to a building episode which involved just a portion of the complex. Therefore, this activity could be related to restorations carried out after the 63 CE earthquake, but, unfortunately, there are not enough data to confirm this hypothesis.

Gr 3b mortars are attributable to the Roman period and, probably, to a later period than the mid-first century CE because both samples come from the eastern wall of the *atrium* of the building phase III *domus* at number 21 (Figure 6).

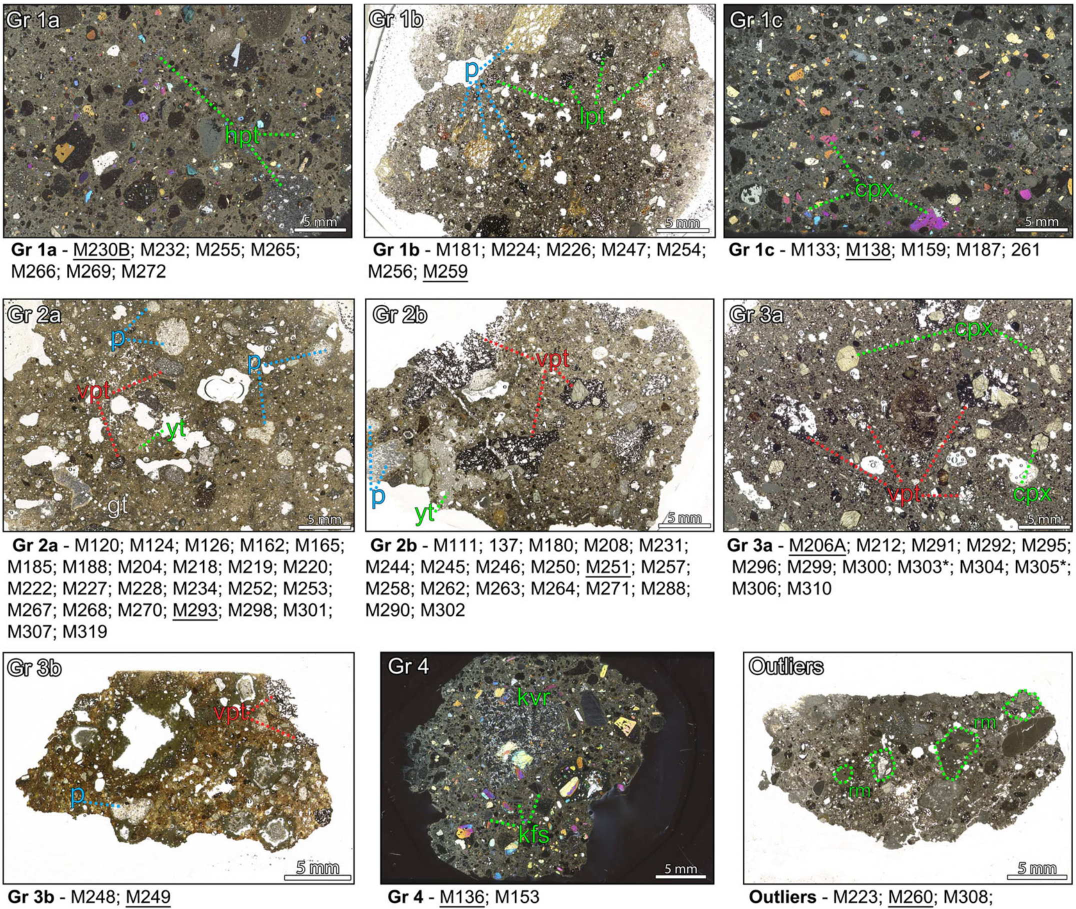

FIGURE 5 Optical microscope working on transmitted light (TL-OM) acquisitions of 30 μm-thin sections of representative samples of the groups. Sample M259 is represented: lpt, leucite phonolite tephrite; p, pumice; yt, yellow tuff clast; gt, grey tuff clast; hpt, hypocrystalline porphyritic trachyte; vpt, vesicular phonolitic tephrite; cpx, clinopyroxene; kfs, K-feldspars; kvr, K-felspar-rich volcanic rock; and rm, reused mortar fragment

Gr 4 is composed of two sole samples from structures at level −3. Mortars of this group could be 'not modern' due to their proximity in the PCA scatterplot to Roman period mortars.

All mortars from Gr 1 (and its subgroups) are related to restoration activities and characterized by the highest concentration in leucite and leucite-rich rocks. This aspect indicates the use of aggregates related to the Vesuvian ultrapotassic products of the 79 CE eruption and younger ones (Morra et al., 2010; Santacroce et al., 2008). This figure is in agreement with Demauro (2020) who considers the abundance of leucite clasts in Pompeian mortars as a marker for the identification of post-1631 CE restoration mortars. Moreover, by the results of the analysis of second and third CE mortars of Villa del Pezzolo (on the Sorrento peninsula), Rispoli et al. (2019) propose for leucite-rich bearing mortars a broader chronology post-79 CE.

Among the modern restoration groups, the oldest could be Gr 1c. Mortars of this group can be linked to the reconstruction of the baths carried out at the end of the 19th century. This hypothesis is demonstrated by sample M159, coming from a partition wall of the *calidarium* which was completely reassembled using modern bricks (Secco et al., 2019). The spatial distribution of Gr 1c samples shows that this restoration was mostly interested in the rooms situated at levels −3 and −4 and a wall at level +0.

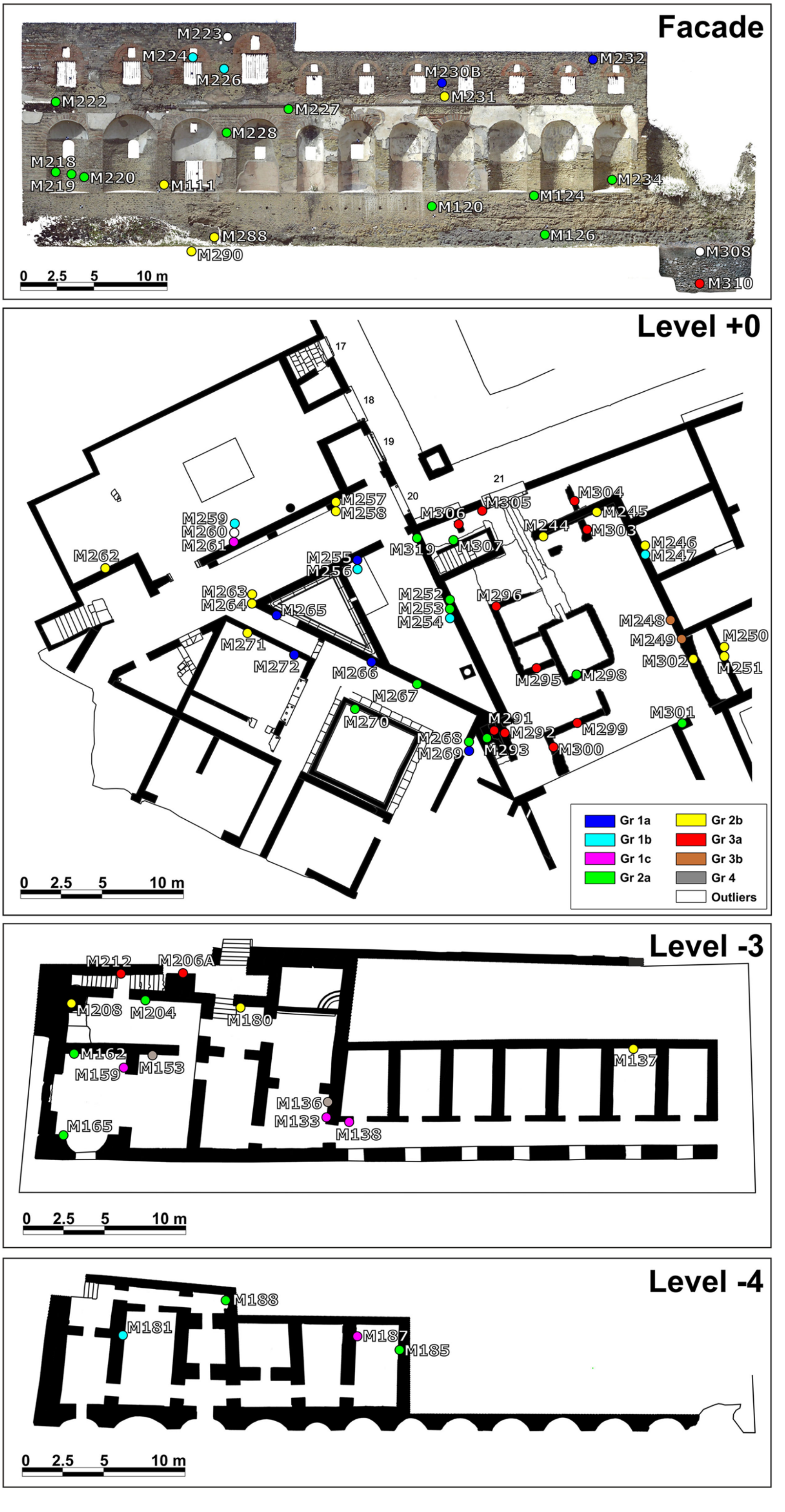

FIGURE 6 Scaled elevation and plans of the distribution of the samples collected from Sarno Baths (levels: facade, +0, −3 and −4) in relation to the groups

On the other hand, Gr 1b relates to the latest restoration after the 1930s: indeed, one of the samples of this group (M259) comes from the south-eastern wall of the *atrium* at number 18, which was partially reconstructed after this date, as demonstrated by the photograph published in 1936 (see Figure S2 in the additional supporting information) (Noack & Lehmann-Hartleben, 1936). The distribution of Gr 1b samples indicates that this restoration episode was mainly interested in the structures located at level +0 and the western part of the upper portion of the facade (Figure 6). The exact chronologies of Gr 1a mortars and outliers M223, M260 and M308 remain indeterminate. Gr 1a mortars could be related to a punctual modern restoration that interested some wall structures at level +0 and at the eastern part of the upper portion of the facade.

These data demonstrate that the entire upper portion of the southern facade was deeply restored in the modern age, as suggested also by the lithological analysis of stone materials and wall techniques (Piovesan et al., 2019). On the other hand, the distribution of the samples at levels −3 and −4 shows that the restoration activities were mainly focused on the internal walls, while the outer ones preserved their original aspect.

Finally, it must be remarked that no restoration mortars were produced using modern cement apart from sample M150 (not considered in this research), coming from a reconstructed vault at level −3 and already described by Secco et al. (2019).

CONCLUSIONS

In this paper we sequenced the building phases of the Sarno Baths by strictly integrating the traditional archaeological analysis with archaeometrical data and statistics. XRPD-QPA data, processed via PCA, were crucial for defining the main features of the mortars and the petrographic analysis allowed us to link the correlations among some mineral phases to the different clasts used as aggregates. We believe that this approach could be adopted as a tool to frame the complex 'history of construction' of ancient buildings not only in Pompeii but also at other archaeological sites.

We also think that the groups we delineated could establish a basis for a 'catalogue' of mortar types produced in Pompeii between antiquity and the modern era. They could constitute an element for absolute dating, but the analysis needs to be extended to other well-dated contexts in the Vesuvian territory in order to be validated.

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AUTHOR CONTRIBUTIONS

SD, CP and MS designed the research; GA, JB and PT supervised the research project; MS and CP performed the samplings; JC, GR and SD performed the samples preparation; MS and SD analysed the samples; SD performed data processing and interpretation; CP performed the architectural analysis and final contextualization of the results; MSB wrote the second section; SD wrote the first, some of the fourth and the fifth to seventh sections, and assembled all the figures and tables; CP wrote the third, sixth and seventh sections; MS wrote some of the fourth section; all authors collaborated in the revision of the manuscript.

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DATA AVAILABILITY STATEMENT

The data that supports the findings of this study are available in the supplementary material of this article.

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