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MULTIANALYTICAL CHARACTERISATION AND PROVENANCE INVESTIGATION OF NATURAL POZZOLANA IN ROMAN LIME MORTARS FROM THE ARCHAEOLOGICAL SITE OF HIPPO REGIUS (ALGERIA)

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ABSTRACT

This research provides the first detailed insight into the composition of hydraulic bedding and coating mortars used in the Roman city of Hippo Regius with the aims of bringing to light the use of natural pozzolanic aggregates for mortars preparation following the legacy of Vitruvius and determining their possible sources. Nine samples of hydraulic mortars taken from different structures have been thoroughly analysed adopting a multi-analytical research strategy combining thin-section petrography, optical cathodoluminescence (CL) microscopy, and scanning electron microscopy (SEM) coupled with energy-dispersive spectroscopy (EDS).
The results indicate the use of pumice clasts with trachytic composition (SiO_2: 58.9-64.4 wt.% and total alkali oxide content: 8.81–14.20 wt.%) as a pozzolanic additive to enhance the strength and hydraulicity of mortars. The comparison of petro-mineralogical features and major element concentrations with literature data reveals the Campi Flegrei caldera (Phlegraean Fields) in the Gulf of Naples, and probably the Baia-Fondi di Baia, as possible area of provenance. This is the first proven case of trade in Phlegraean tephra as a building material to a city of *Africa Proconsularis*, and a new piece of evidence of the spread of Roman technologies to the southern Mediterranean region of the Roman Empire as well as the transfer of their technical expertise to local artisans of the provincial city of Hippo Regius.

KEYWORDS: Hippo Regius, Annaba, Roman mortars, Pozzolana, Phlegraean Fields, Petrography, Cathodoluminescence, SEM-EDS

1. INTRODUCTION

Pozzolans are siliceous or siliceous and aluminous materials that Roman builders added as aggregates to the mix of lime-based binders in order to improve strength and water resistance of bedding and coating mortars. These additives contain little or no cementitious properties but, when finely ground, they chemically react with calcium hydroxide in the presence of water to form compounds with cementitious characteristics (Cadix and James, 2022). Pozzolans used by Romans could be of natural origin (i.e., pyroclastic materials) or artificial, derived from ceramic waste and organic ashes.

This technological improvement marks the advent of hydraulic mortars (Artioli et al., 2019) that were used to prevent infiltrations in several water-related structures and had the ability to cure under water (Ginouvès et al., 1985). Vitruvius mentioned volcanic ash and other aggregates of natural origin as typical additives in Roman building materials in his exhaustive essays on architecture, and whenever natural pozzolana was not available, it was replaced with crushed terracotta obtained from the recycling of ceramic fragments that become reactive at different firing temperatures (Lancaster, 2019). Reactive ashes derived from the combustion of plant and animal-related organic matter have also been used by Roman builders as artificial pozzolanic materials in Mediterranean areas where Punic influences were still present (Secco et al., 2020).

In his overview of Roman technologies, construction methods, and building materials, Vitruvius testified to the astonishing results of *pulvis*, the volcanic ash that came from *Baiae* in the Campania region, as it conferred strength to the constructions and was capable to solidify underwater when mixed with lime and rubble (*De Architectura*, 2.6.1-2). He also mentioned *harenae fossiciae* as a volcanic sand (*De Architectura*, 2.4.1) that could be found in the Latium region. In the following decades, Strabo (*Geographica*, 5.4.6) and Seneca (*Quaestiones Naturales*, 3.20.3-4) both testified to the enhancements provided by adding pozzolanic materials which became a necessary component for maritime harbour construction (Jackson et al., 2017). Later, Pliny the Elder described the volcanic ash as *pulvis puteolanus* (*Naturalis Historia*, 35.166) related to its origin from the Roman city of *Puteoli* in the Gulf of Pozzuoli (D'Ambrosio et al., 2015). Its mention by Pliny the Elder indicates that it was still added to marine concrete during the 1st century AD.

Recently, several studies managed to unravel the secret behind the pozzolanic reaction of volcanic aggregates with lime-based binders and have shown that the interaction produces calcium aluminosilicate hydrates (Jackson et al., 2013; Raneri et al., 2018; Dilaria et al., 2022) that grow as fibrous minerals creating a fibril-rich matrix (Vanorio and Kanitpanyacharoen, 2015) which provides strength and hydraulic properties to the mixtures.

Most provenance studies point to the Phlegraean Fields as a main source of pozzolana that was used both in nearby cities such as *Cumae*, *Baiae* and *Surrentum* (Di Benedetto et al., 2018; Graziano et al., 2018; Rispoli et al., 2019, 2021) and in distant cities of the Empire such as *Panormus*, *Chersonesus*, *Soli-Pompeiopolis* and even *Caesarea Palestinae* (Brandon et al., 2005; Stanislao et al., 2011; Vola et al., 2011; Montana et al., 2018). This shows that Roman artisans played a key role in the spread of natural pozzolana across the European and the eastern Mediterranean provinces. However, evidence is lacking regarding its use in the southern provinces such as *Mauretania Caesariensis* and *Africa Proconsularis*.

Hence, this study aims to investigate the use of natural pozzolana in hydraulic mortars from the archaeological site of Hippo Regius, an important harbour for grain export (Stone, 2014) in *Africa Proconsularis*, by conducting a multi-analytical characterisation of natural pozzolans to determine their provenance and highlight the transfer of this Roman innovation to local artisans of this city.

2. ARCHAEOLOGICAL SITE

The ancient city of Hippo Regius is located in the northeastern region of Algeria (Fig. 1), near the mouth of the river Seybouse, at a distance of 2 km from the modern city of Annaba. Between the 12th and 9th centuries BC, the city was founded by the Phoenicians and Carthaginians (Dahmani, 1983), then it became part of the native kingdom of Masinissa (Numidia) after the collapse of the Carthaginian Empire in 146 BC at the end of the third Punic War (Holmes Van Mater, 1970). Hippo Regius was annexed to the Roman Empire in 27 BC after the creation of *Africa Proconsularis* by Augustus to become a *municipium*, later it received the rank of a *colonia* during the Flavian dynasty (Lepelly, 2005) and retained this status until the Vandal invasion in 431 AD (Lassus, 1976). The first archaeological findings were Christian epitaphs and mosaics discovered in 1833 during the French settlement until 1895 when the first excavation, conducted by M. Chevillot and F. G. de Pachtère, brought to light the seafront *villae* (Amraoui, 2017). S. Gsell and E. Albertini joined forces to convince the authorities to acquire the neighbouring lands and impose preservation easements to stop the development of the industrial suburb, which allowed the extension of the excavation area. The investigations conducted between 1925 and 1938 by E. Marec, P. Choupaut and L. Leschi led to major discoveries such as the Christian district, the Roman Theatre and *Forum*, the fountain of the

Gorgon and a second monumental fountain north of the *Forum* (Delestre, 2005b). E. Marec became the lead archaeologist of Hippo Regius between 1947 and 1963, and succeeded in unearthing the Northern Great Baths, the Procurator's House, the Southern Baths, the monument of the *Dii Consentes* and the *Macellum* (Marec, 1954). The last on-site investigation was the archaeological survey conducted between 2002 and 2005 with the aim of implementing a preservation plan for Hippo Regius under the guidance of X. Delestre.

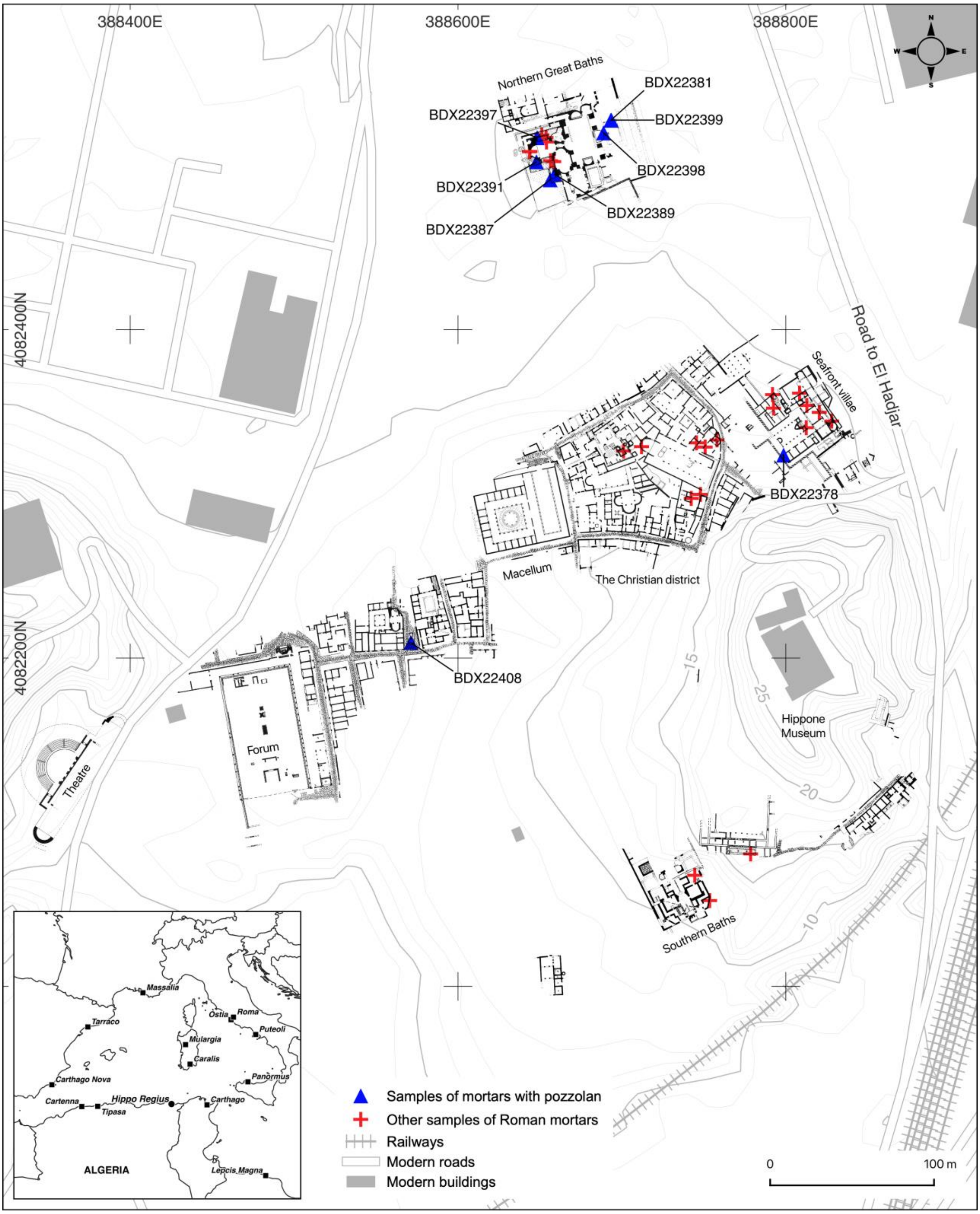

Figure 1. Locations of sampled mortars from different structures of Hippo Regius. Samples with natural pozzolanic materials are represented with blue triangles (archaeological plan adapted from Marec, 1954; Stawski, 1957; Delestre and Tavé, 2005)

The exact chronology of the identified structures remains uncertain, as previous excavations were undertaken following Christian archaeological practices in accordance with Augustine of Hippo (Delestre, 2005a), which omitted stratigraphic and historical contexts throughout the site (Delestre, 2005b). Nevertheless, it was possible to determine a chronology using relative dating methods. E. Marec and J. P. Morel suggested that the construction of the seafront *villae* took place in successive phases between the mid-3rd and the 5th centuries AD (Blanchard-Lemée, 2005), whilst the construction of the Baptistry and *Basilica* occurred, according to the style of the earliest mosaics, during the second half of 4th century AD (Bizot, 2005).

The Northern Great Baths, which cover an area of 2,000 m^2, were completed during the reign of Caracalla (211-217 AD) and dedicated to Septimius Severus as indicated by an inscription found within the structure (Thébert, 2003), whereas the Southern Baths, which cover an area of 1,500 to 2,000 m^2, could be prior to the latter structure, as indicated by inscriptions which date the building to 198 AD (Delestre, 2005c). The *Forum* was built during the second half of the 1st century as suggested by the inscription *C. PACCIUS AFRICANUS PROCOS. AFRICAE.* (78-79 AD) carved on the pavement slabs (Suméra, 2005). Not far from the *Forum* the fountain of the Gorgon and a second monumental fountain were identified, both possibly contemporary with the aqueduct dating between the late 1st century AD and the early 2nd century AD (Lamare, 2019).

From an architectural point of view, one of the common building techniques used in Hippo Regius was the *opus africanum*, where vertical blocks of stone alternating with horizontal blocks are filled in between with smaller blocks of limestone or metamorphic rocks bonded with mortar (Delestre, 2005d). It is also common to find the *opus testaceum*, where baked bricks are bonded with mortar, in the walls and vaults of the Northern Great Baths, in remaining parts of the Theatre, the foundation walls of the fountain of the Gorgon, and the Seafront *villae*. Walls and floors of the Northern Great Baths as well as the interior floors of the *Forum*, the *Macellum*, and the two public fountains were covered with marble veneers. The Christian district's buildings were mostly decorated with mosaics, whereas the private building's floors were either adorned with *opus tessellatum* or coated with *cocciopesto*, a mix of lime mortar and crushed ceramic fragments. Coating mortars were also applied at the junction of walls and floors in pools of the Northern Great Baths and cisterns of both public and private buildings to prevent water infiltration.

3. GEOLOGICAL SETTINGS

Located in the eastern side of the Alpine chain of Maghrebides, the geological framework in the vicinity of Hippo Regius (Fig. 2) is dominated by sedimentary formations ranging from Permian to Quaternary ages which feature calcareous chain, Mauretanian and Massylian flysch nappes, Numidian flysch and sandstones associated with limestone outcrops as well as recent Plio-Quaternary deposits (Vila, 1980). Near the coastline, recent metamorphic formations occurred as a result of collisions between the African and European tectonic plates during the Eocene and the Miocene, creating the Edough Metamorphic Core Complex (Marignac et al., 2016). This complex features the Seraidi Gneiss Unit (high-grade paragneiss), the Marble Complex Unit (high-grade marble, mica schist, and amphibolite) and the Alternance Series Unit showing (medium-grade metapelite with numerous intercalations of marble and feldspathic quartzite). The igneous formations are related to the Neogene magmatic activity and located between the Cap de Garde and Cap de Fer headlands and in the Bougaroun area (Abbassene et al., 2016). They feature mafic rocks (gabbros) and intermediate to felsic rocks (diorites, granodiorites and microdiorites) as well as felsic rocks (microgranites, granites, and rhyolites). The geological formations near Hippo Regius served as local sources to quarry raw materials for the Roman artisans.

The type of marble with medium grain known as *greco scritto* was extracted from the Cap de Garde headland during the Roman imperial period (de Vos, 2003) and is proved to have been used in Hippo Regius as well as the Roman cities of *Cuicul*, *Utica* and *Carthago* for columns, architectural elements and as slabs and veneers on floors and walls (Antonelli et al., 2009). Gneiss was also quarried from the Edough massif and used as large slabs for the pavement of *decumanus* and *cardo* roads, while mica schist was commonly quarried to be used as rubble stones (Delestre, 2005d).

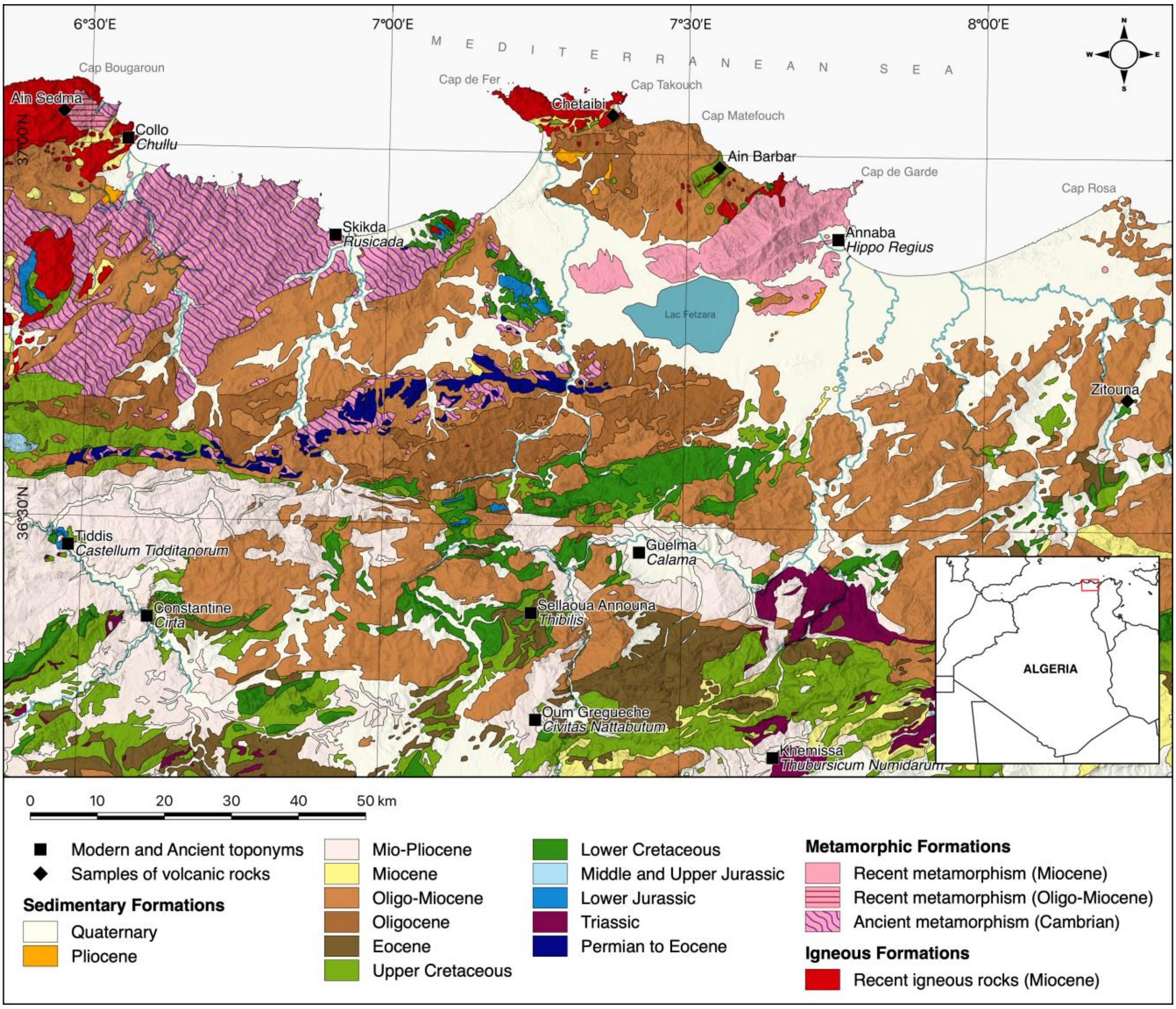

Figure 2. Geological sketch map of NE Algeria showing sedimentary, metamorphic, and igneous formations in the vicinity of the ancient city of Hippo Regius (adapted from Vila, 1980)

4. MATERIALS AND METHODS

4.1. Materials

In this study, thirty-four samples of Roman mortars were carefully collected with a hammer and chisel from the most representative architectural structures of the archaeological site of Hippo Regius (Fig. 1) while ensuring to preserve the integrity of the archaeological remains and avoiding portions altered during the restoration campaigns of 1951 and 1954. The samples are labelled with the BDX prefix, followed by the serial number assigned according to the Archeosciences Bordeaux laboratory register.

Then, a preliminary petrographic investigation was conducted to determine the presence of natural pozzolana within mortars. Twenty-five samples contain only ceramic fragments at various amounts, used as artificial pozzolana, with no evidence of the use of natural pozzolana (pyroclastic materials). On the other hand, nine samples contain volcanic products with vitreous textures: one sample of bedding mortar (BDX22378) collected from the base of western *opus testaceum* wall located in the Seafront *villae* near the breakwater wall of the city; seven samples of bedding and coating mortars (BDX22381, BDX22387, BDX22389, BDX22391, BDX22397, BDX22398, BDX22399) collected from various locations in the Northern Great Baths and one sample of bedding mortar (BDX22408) collected from the base of the foundation wall of Fountain of the Gorgon, as reported in Table 1.

Though samples BDX22381, BDX22387 and BDX22397 have multiple layers with different petrographic features, they will be considered as single layer mortars in this study because the main purpose remains the characterisation of natural pozzolanic aggregates added to these mortars.

Table 1. List of mortar samples with natural pozzolana collected from the site of Hippo Regius with an indication of their type, their location, their structure of origin, and their chronology

Sample ID	Typology	Location	Structure	Chronology
BDX22378	bedding mortar	*opus testaceum* wall located in Room B15	Seafront *villae*	280 to 330 AD
BDX22381	bedding mortar	support of marble veneers of the eastern *natatio* (cold-water pool)	Northern Great Baths	211 to 217 AD
BDX22387	coating mortar	eastern coated wall of the *gymnasium*	Northern Great Baths	211 to 217 AD
BDX22389	bedding mortar	corner of *opus incertum* wall in the western service corridor next to the *praefurnium*	Northern Great Baths	211 to 217 AD
BDX22391	bedding mortar	brick arch of the western *praefurnium*	Northern Great Baths	211 to 217 AD
BDX22397	coating mortar	coating of the northern *alveus* (hot-water pool)	Northern Great Baths	211 to 217 AD
BDX22398	coating mortar	quarter circle junction at the base of wall and floor of the eastern *natatio*	Northern Great Baths	211 to 217 AD
BDX22399	bedding mortar	support of marble paving of the eastern *natatio*	Northern Great Baths	211 to 217 AD
BDX22408	bedding mortar	*opus testaceum* (foundation) wall of the fountain	Fountain of the Gorgon	late 1st century AD

4.2. *Methods*

A complete mineralogical and chemical characterisation is necessary to find the provenance of the natural pozzolanic components added to the hydraulic mortars listed above. Therefore, each sample was analysed using a multi-analytical approach consisting of thin-section petrography, optical CL microscopy, SEM image analysis and EDS microanalysis.

First, the selected mortar samples were subjected to microscopic examinations performed on 30 µm thin-sections under a Leica DM2500 P microscope equipped with a Leica DFC420 C digital camera. Microphotographs were acquired under cross-polarized light (XPL) and analysed with Leica LAS EZ software to measure the size of the pozzolanic aggregates as well as their shapes (rounded or angle-shaped aggregates) and their distribution (Arizzi and Cultrone, 2021). The shapes of the aggregates were determined using the visual roundness scale (Powers, 1953) while the distribution within the lime matrices was determined using Shvetsov's diagrams for visual estimation of percentages of minerals in rock sections (Terry and Chilingar, 1955; Coutelas and Büttner, 2009).

Then, polished sections of mortar samples were examined using optical CL microscopy to reveal the processes of crystallisation within the internal textures of minerals (Götze et al., 2013). The samples were placed in a vacuum chamber (0.05 mbar) and bombarded with an accelerated electron beam (acceleration tension: 10-15 kV, current intensity: 190-220 MA, angle of incidence: 30°) to then be examined under the Leica MC125 optical microscope. Microphotographs of light emitted by electron-excited minerals were taken with a Leica DFC420 C digital camera during an exposure time of ±7 s.

Finally, the micromorphology of the pozzolanic aggregates present in studied mortars was examined by scanning electron microscopy (SEM) using the electron microscope JEOL IT500HR in low vacuum mode (pressure: 30 Pa; HT: 20 kV) while chemical compositions were measured using energy dispersive spectroscopy (EDS) microanalysis with two Oxford UltimMax 100 detectors coupled to the electron microscope, operating at 20 kV primary beam voltage and a net acquisition time of 30 s (producing spectra with 4.5–5.2 million counts). Backscattered SEM images and EDS spectra were acquired on thin-sections of the selected mortar samples without carbon coating. Compositional data was processed using AZtec software and chemical quantifications measured by SEM-EDS in mass % were converted to oxide weight %, normalised to 100% so that they could easily be compared with chemical compositions of natural pozzolans known in the literature.

5. RESULTS

5.1. *Thin-section petrography observation*

Microscopic observations revealed a high similarity among the petrographic features of the studied mortars (Fig. 3). They are characterised by lime-based binders with cryptocrystalline to micritic texture, lime lumps and bioclasts; mostly echinoids and foraminifera shells along with algae microfossils which represent unreacted lime and under-burnt fragments of limestone, and various types of inclusions composed of minerals, ceramic fragments, and lithic aggregates. The mineral component of the matrices is composed of individual grains of quartz, alkali feldspar, plagioclase, biotite, muscovite, calcite, hornblende, and clinopyroxene with grain size ranging from 0.2 to 1.8 mm. Crushed terracotta fragments differ from each other and show ceramic matrices of various colours with an aggregate size ranging from 0.1 to 9.0 mm, whilst the lithic clasts' range from 0.3 to 4.0 mm in size and represent fragments of sedimentary rocks (arenite and siltstone), metamorphic rocks (gneiss, mica schist and marble), plutonic rock fragments (microgranite) as well as volcanic rock fragments indicating the presence of natural pozzolana.

Table 2. Size, shape (roundness grade), and distribution of the volcanic aggregates within mortars samples

Sample ID	Size	Shape	Distribution
BDX22378	0.3-0.6 mm	rounded	+
BDX22381	0.4-3.2 mm	subrounded	+++
BDX22387	0.8-1.1 mm	rounded	++
BDX22389	0.5-1.4 mm	rounded	+++
BDX22391	0.7-5.5 mm	rounded	++
BDX22397	2.2-2.5 mm	subrounded	++
BDX22398	0.9-4.9 mm	rounded	+++
BDX22399	1.4-10 mm	rounded	+++
BDX22408	0.8-15 mm	rounded	++++

Note: distribution percentages are expressed + for less than 1%; ++ for 1% to 3%; +++ for 5% to 10%; ++++ for 15% to 25% (according to Terry and Chilingar, 1955)

The volcanic rock fragments are scattered in the lime-based binders and show aphanitic (Fig. 3a) to porphyritic textures (Fig. 3e) with needle-like microlites and phenocrysts of alkali feldspar embedded in a glassy groundmass. The phenocrysts of feldspar are identified under cross-polarized light as sanidine in which the contrast of light and dark demonstrates the Carlsbad twinning (Fig. 3b), while minor amounts of other minerals are identified as plagioclase and clinopyroxene associated with flakes of dark brown biotite (Fig. 3d). The volcanic rock fragments show similar roundness grades varying from subrounded to rounded, whereas the grain size varies from 0.3 to 15 mm. The visual estimation of percentages of volcanic aggregates in lime-based matrices ranges from less than 1% in BDX2278 to 25% in BDX22408 as reported in Table 2.

5.2. *Optical cathodoluminescence microscopy*

Optical CL microscopy performed on polished sections of mortar samples confirms the identification of mineralogical features (Fig. 4) previously characterised via petrographic observation. The light blue luminescence discriminates alkali feldspar crystals from yellowish green plagioclase (Scholonek and Augustsson, 2016) whereas quartz crystals are optically distinguished by violet-blue weak luminescence.

The volcanic rock fragments show light blue luminescence due to the dominance of minute tabular alkali feldspar crystals and sanidine phenocrysts embedded within the glassy matrices. Rare plagioclase crystals are highlighted with yellowish green luminescence within the volcanic aggregates (Fig. 4b) while secondary crystallisation of carbonates highlighted with orange to red luminescence (Hiatt and Pufahl, 2014) within the glassy groundmass of aggregates (Fig. 4f and 4g), suggests the presence of cavities. The fragments' matrices show a texture variation from aphanitic, porphyritic to vitrophyric texture with an abundant amount of scattered microlites with crystal sizes ranging from 40 to 110 μm and surrounded by glass (Fig. 4c). These observed features (i.e., phenocrysts of sanidine, k-feldspar microlites and pyroclastic textures) are characteristic of igneous rocks which cool down and lose gas so rapidly as the magma arises that atoms in the silicate melt have insufficient opportunity to organise into regular crystals. Instead, the melt solidifies and turns into viscous amorphous glass (Best, 2003).

5.3. *Scanning electron microscopy and chemical analyses*

SEM analyses performed on volcanic aggregates identified as pozzolana within the studied mortar samples reveal a highly vesicular pumiceous micromorphology in backscattered images (Fig. 5) with evident pozzolanic reaction rims between juvenile pumice clasts and lime-based binders, which testifies the mortars' hydraulicity. The high vesicularity of the groundmass is related to rapid exsolution and decompression of magmatic volatiles which occur during explosive eruptions (Houghton and Wilson, 1989). Vesicles show variations of shapes ranging from round (Fig. 5b) to elongate (Fig. 5i), as well as a variation of density which differs from weak (Fig. 5a) to extreme vesicularity with a high glass connectivity (Fig. 5e). The differences in vesicularity in pumice clasts may be explained by slightly different variations of pressure during nucleation and growth of the bubbles which result from variable trajectories in the turbulent gaseous eruption column (Thomas et al., 1994).

In order to determine the provenance of the natural pozzolana, EDS microanalysis was carried out on glassy groundmass of the most representative juvenile pumice clasts from each of the studied mortar samples. Compositional data (Table 3) show that SiO_2 content ranges from 58.9 to 64.4 wt.% indicating an intermediate igneous rock composition with moderate content of silica, and total alkali ($Na_2O + K_2O$) content between 8.81 and 14.20 wt.% which span across the phono-trachytic fields and mostly plot below the boundary line of the phonolitic compositional field (Fig. 6a) of total alkali silica (TAS) diagram for volcanic rocks (according to Le Bas et al., 1986).

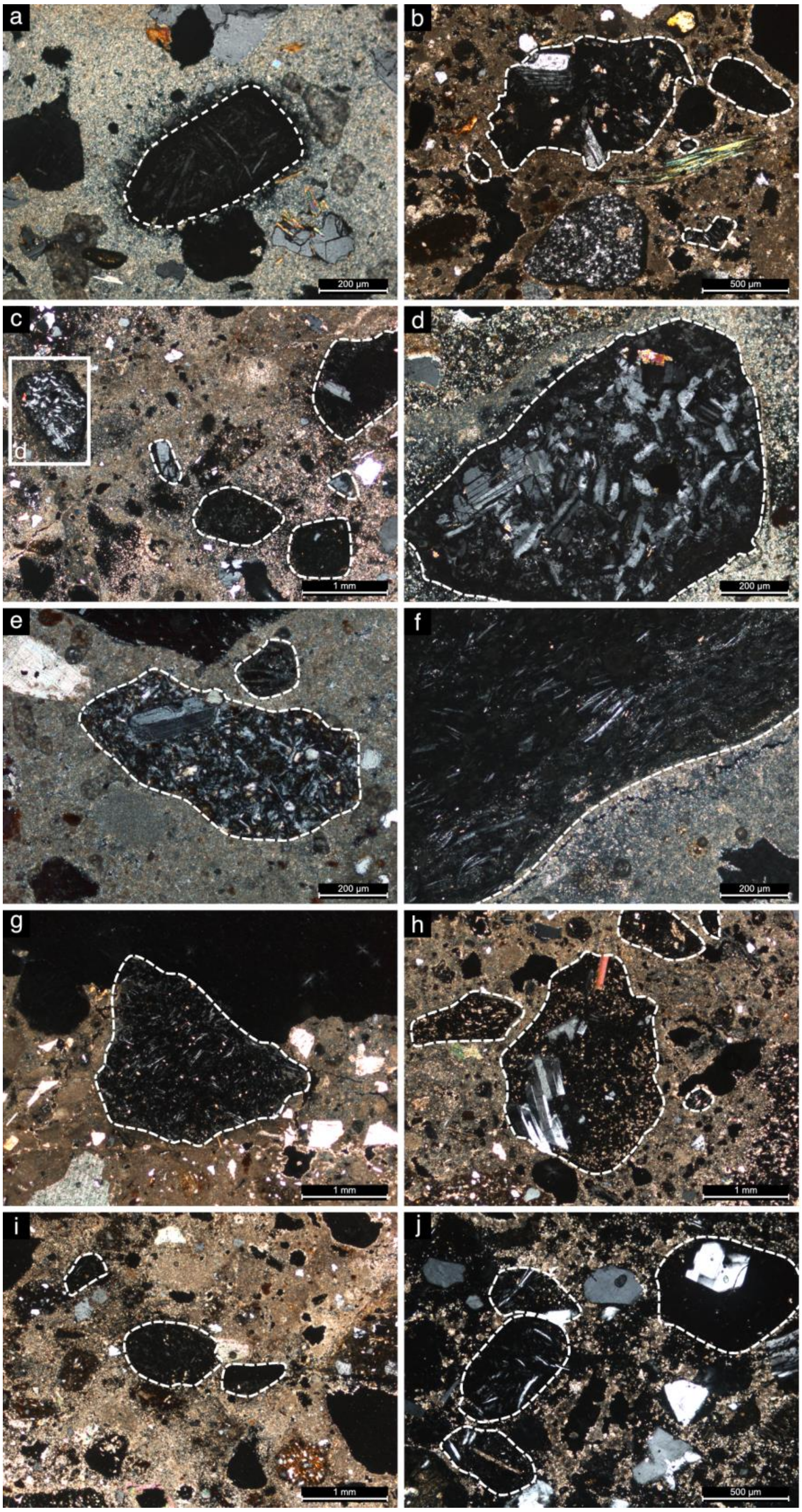

Figure 3. Optical microscopy images (XPL) showing mortar components with volcanic rock fragments highlighted by dashed lines: (a) glassy groundmass with needle-like microlites surrounded with reaction rim in BDX22378; (b) sanidine phenocrysts embedded in a glassy groundmass in BDX22381; (c-d) biotite and multiple minute tabular sanidine crystals and phenocrysts in BDX22387; (e) abundant sanidine microlites and phenocrysts in BDX22389; (f) alkali feldspar microlites in parallel arrangement in BDX22391; (g) glassy groundmass with tabular crystals in BDX22397; (h) sanidine phenocrysts and biotite embedded in glassy groundmass in BDX22398; (i) fine-grained matrix with tabular crystals in BDX22399; (j) sanidine microlites and phenocrysts in BDX22408

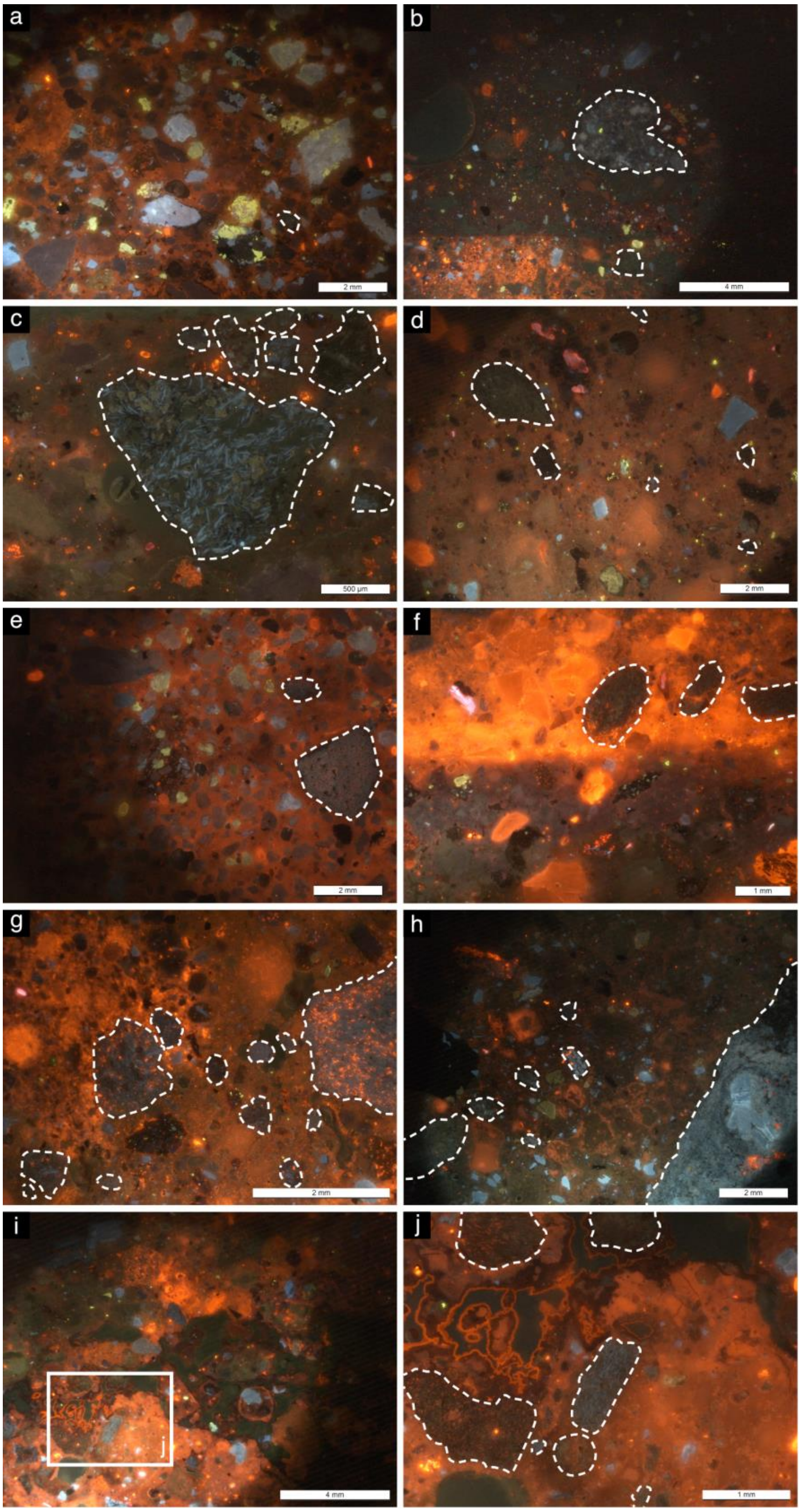

Figure 4. Optical CL images of mortar mineral components highlighting quartz with dark violet-blue luminescence, alkali feldspar and sanidine phenocrysts with light blue luminescence, plagioclase with green-yellow luminescence, and calcite with orange luminescence within samples: (a) BDX22378; (b) BDX22381; (c) BDX22387; (d) BDX22389; (e) BDX22391; (f) BDX22397; (g) BDX22398; (h) BDX22408; (i-j) BDX22399. Tabular crystals of alkali feldspar and sanidine microlites are visible at higher magnifications embedded in volcanic fragments contoured with dashed lines. Orange luminescence reveals lime-based matrices and lumps, crystals of calcite and secondary crystallisation of carbonates within voids

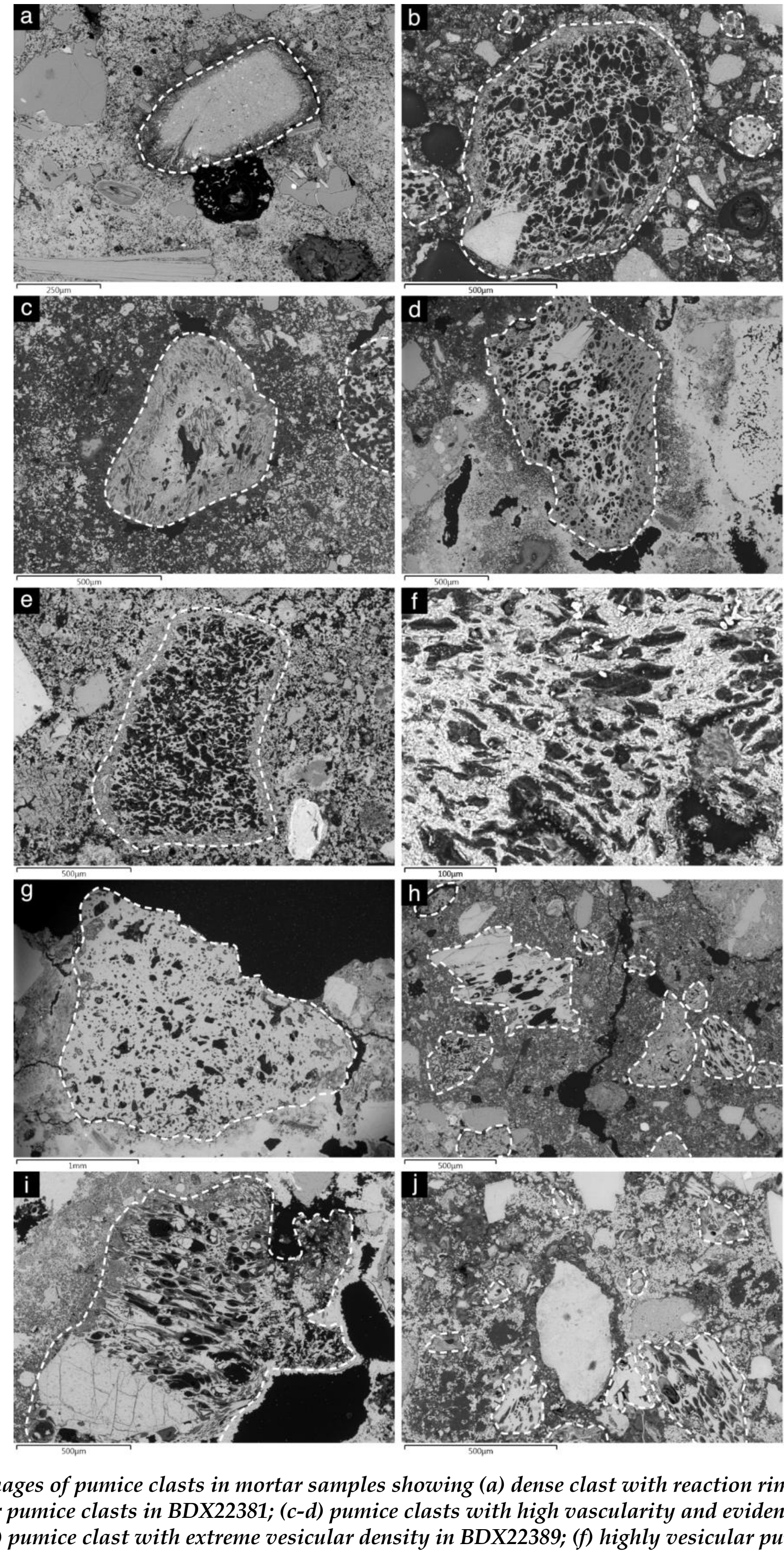

Figure 5. SEM images of pumice clasts in mortar samples showing (a) dense clast with reaction rim in BDX22378; (b) highly vesicular pumice clasts in BDX22381; (c-d) pumice clasts with high vascularity and evident reaction rims in BDX22387; (e) pumice clast with extreme vesicular density in BDX22389; (f) highly vesicular pumice clast with accessory phases (Fe-Ti oxides) in BDX22391; (g) large pumice clast with moderate vesicularity in BDX22397; (h) multiple pumice clasts with heterogeneous vesicularity in BDX22398; (i) large pumice clast with elongated microvesicles in BDX22399; (j) multiple pumice clasts with various vesicle densities and shapes in BDX22408. Glass in groundmass is light grey, vesicles are dark grey or black features

Table 3. Major element concentrations in glassy groundmass of two representative pumice clasts from each mortar sample (measured by EDS, in wt.%)

Sample ID	SiO_2	TiO_2	Al_2O_3	Fe_2O_{3T}	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	Total	$Na_2O + K_2O$
BDX22378	61.44	0.33	17.88	1.99	0.08	0.38	4.86	4.65	7.36	0.29	99.75	12.01
	63.03	0.15	18.67	1.29	0.09	0.33	3.75	6.49	5.76	0.16	99.83	12.25
BDX22381	60.62	0.42	18.45	3.13	0.13	0.59	2.73	4.77	8.16	0.08	99.31	12.93
	60.58	0.41	18.58	2.93	0.16	0.49	4.27	4.10	7.45	0.08	99.26	11.55
BDX22387	62.32	0.43	18.77	2.71	0.15	0.34	2.81	5.11	6.31	0.05	99.20	11.42
	61.68	0.46	17.01	3.22	0.22	0.36	7.61	4.24	4.57	0.00	99.82	8.81
BDX22389	61.43	0.33	18.15	2.51	0.16	0.33	2.74	6.62	6.73	0.00	99.26	13.35
	58.98	0.41	18.04	2.84	0.14	0.42	3.91	6.98	7.22	0.05	99.23	14.20
BDX22391	63.12	0.32	18.51	1.98	0.12	0.20	2.00	4.93	8.14	0.00	99.52	13.07
	61.62	0.40	17.72	2.84	0.19	0.29	2.01	4.25	9.70	0.00	99.22	13.95
BDX22397	61.56	0.38	18.44	2.86	0.17	0.34	2.77	5.86	6.62	0.06	99.33	12.48
	61.12	0.36	18.39	2.76	0.17	0.33	3.73	5.62	6.46	0.00	99.20	12.08
BDX22398	62.03	0.31	18.94	1.83	0.08	0.30	3.73	5.05	7.07	0.00	99.60	12.12
	60.73	0.39	18.78	2.80	0.12	0.53	2.94	5.11	7.69	0.08	99.32	12.80
BDX22399	61.14	0.40	18.71	2.90	0.12	0.51	2.96	4.66	7.83	0.08	99.49	12.49
	60.01	0.40	18.08	2.99	0.15	0.55	4.12	5.20	7.36	0.08	99.23	12.56
BDX22408	60.79	0.47	17.49	3.72	0.17	0.72	5.58	3.86	6.28	0.00	99.42	10.14
	64.44	0.32	17.94	2.21	0.08	0.52	2.68	6.00	5.51	0.09	99.98	11.51

The chemical composition of the representative juvenile pumice clasts' groundmass is consistent and exhibits a variance of 1.45 wt.% for silica content and 1.53 wt.% for total alkali content, suggesting the trachytic pumice clasts originate from an identical source; whereas calcium oxide content shows a higher variance of 1.78 wt.% that is most likely due to contamination of pumice clasts with secondary crystallisation of calcite that filled relict pores and pumice vesicles (Fig. 4g).

6. DISCUSSION

The multi-analytical approach allowed to identify the components of bedding and coating Roman mortars collected from water-related structures belonging to the city of Hippo Regius. The combination of chemical and mineralogical results and archaeological data provides significant insight about the know-how and technical skills of the artisans, as well as the raw materials used in the making of these hydraulic mortars.

Considering the surrounding geological settings, mineral particles and lithic fragments are of a local provenance as Plio-Quaternary deposits are abundant within the area. Microgranite, mica schist, marble and gneiss aggregates are detrital materials derived from the Edough massif, whilst the provenance of ceramic fragments cannot be determined with certainty, as the petrographic differences likely suggest a recycling of ceramic waste materials. Lime lumps and residual calcareous fragments of echinoids and not completely calcinated foraminifera fossil shells suggest that limestone used for on-site production of binders might originate from lower Jurassic to lower Cretaceous bioclastic limestones found in the outcrop near Ain Kerma (Vila, 1980), about 50 km from Hippo Regius.

As regards the natural pozzolana, the mineralogical, petrographic, and compositional data confirm the volcanic origin of the aggregates which mainly consist of highly vesicular glassy matrices along with crystalline phases, such as sanidine, clinopyroxene, rare plagioclase, and brown mica crystals. In the following, we are discussing two hypotheses that might explain the provenance of natural pozzolana.

A first hypothesis is that natural pozzolana and other raw materials were quarried by Romans, from local igneous formations related to Neogene magmatism such as those near Chetaibi, Ain Barbar and Zitouna (Daif and Toubal, 2015), in which rocks are characterised by high concentrations of silica content ranging from 71.6 to 79.8 wt.% and low total alkali content ($Na_2O + K_2O$) with concentrations between 4.83 and 9.43 wt.%. Still in the vicinity of Hippo Regius, compositional data of the igneous formations located in the Cap Bougaroun headland near Ain Sedma (Lakkaichi and Bouabsa, 2017) show SiO_2 content ranges from 74.9 to 76.7 wt.% and total alkali content between 7.26 and 7.81 wt.%. Chemical compositions of these felsic rocks plot within the rhyolitic field (Fig. 6d) of the total alkali silica (TAS) diagram, whereas the scatter plot of the natural pozzolanic clasts is mostly in the trachytic field.

Furthermore, the micromorphological results demonstrated that the pozzolanic fragments exhibit roundness grades varying from subrounded to rounded as well as highly vesicular pyroclastic texture. These features indicate that the fragments were neither quarried nor crushed, but represent tephra fall deposits derived from an explosive volcanic eruption, arguing against a local provenance of the pozzolanic aggregates.

A second hypothesis would be that the natural pozzolana was imported by Romans from a specific volcanic district near *Africa Proconsularis* and characterised by explosive eruptions of intermediate lavas that produced pyroclastic rocks, such as Stromboli in the Aeolian arc and volcanoes of the Campania Province (i.e., Somma-Vesuvius and Phlegraean District volcanics). This hypothesis requires a comparison of compositional data of the analysed pumice aggregates with compositional trends of rocks from Somma-Vesuvius products (Fig. 6b), Stromboli volcanic products (Fig. 6c), Campi Flegrei (Phlegraean Fields) tephra deposits (Fig. 6c) including the Campanian Ignimbrite and Neapolitan Yellow Tuff (Fig. 6d).

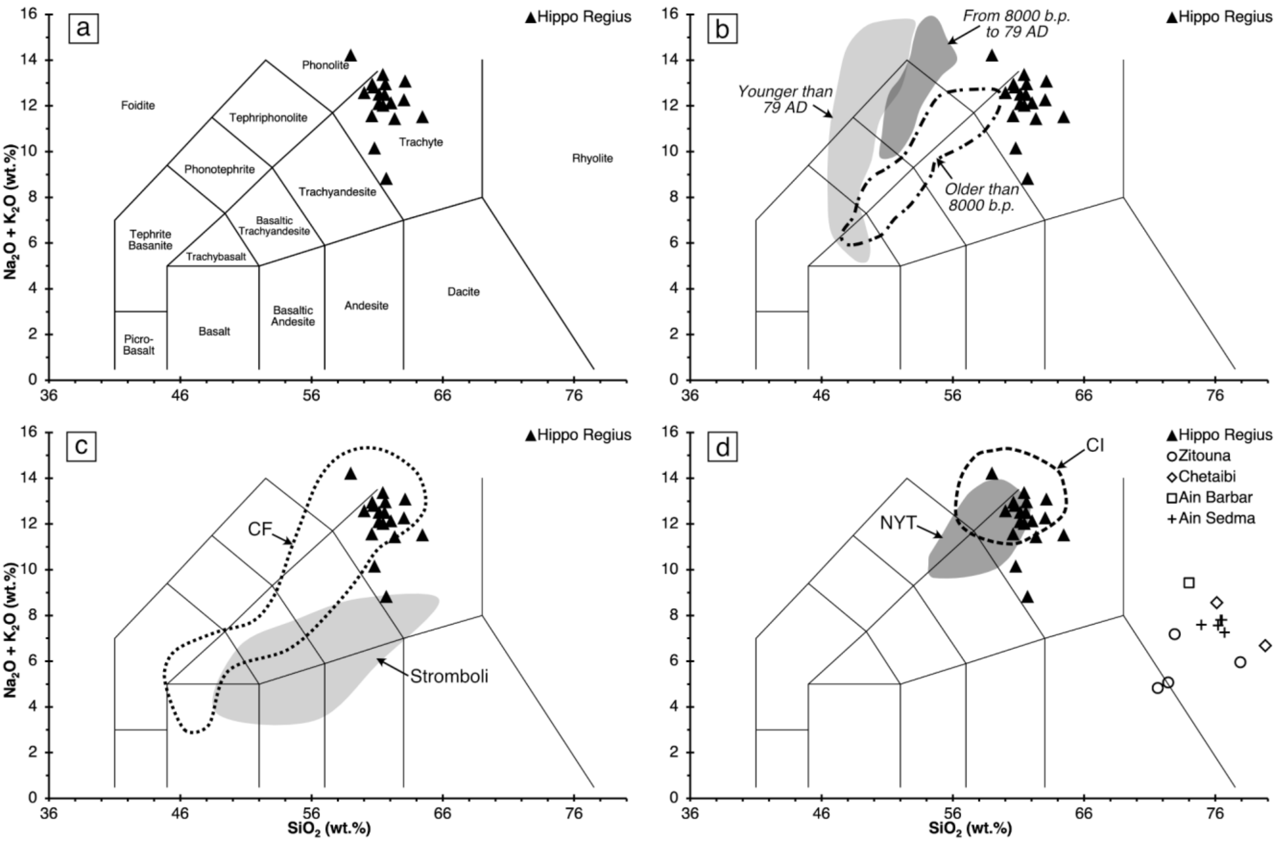

Figure 6. Total alkali silica (TAS) scatter plots of pumice clasts in relation to (a) volcanic rocks' chemistry (modified from Le Bas et al., 1986); (b) fields of the three main eruptive facies of Somma-Vesuvius products (data from Peccerillo, 2005b); (c) fields of Campi Flegrei deposits (CF) and Stromboli volcanic products (data from Peccerillo, 2005a, 2005b; D'Antonio et al., 2007; Peccerillo, 2020); (d) fields of Campanian Ignimbrite (CI) and Neapolitan Yellow Tuff (NYT) (data from Peccerillo, 2005b). Compositions of lithics from Neogene magmatism of NE Algeria (Zitouna, Chetaibi, Ain Barbar and Ain Sedma) are also plotted (data from Daif and Toubal, 2015; Abbassene et al., 2016; Lakkaichi and Bouabsa, 2017)

The geochemical compositions of analysed pumice fragments plot mostly within the trachyte field (Fig. 6a) and away from the compositional fields of the three main rock series of Somma-Vesuvius (Fig. 6b) and the compositional trends of Stromboli volcanic products (Fig. 6c). Stromboli volcanic products range from basaltic andesite and shoshonitic basalt to trachyte lavas and pyroclastic materials with lower Na_2O and K_2O contents (Peccerillo, 2001) and higher MgO content compared to the compositional data of the pozzolanic aggregates (Peccerillo, 2005a). Somma-Vesuvius rock series consist of trachybasalt, shoshonite, latite, trachyte, and trachyphonolite (Peccerillo, 2020) that are moderately silica-undersaturated (SiO_2: 47.3-60.1 wt.%).

On the other hand, the pumice clasts' geochemical results follow the compositional trends of the Phlegraean District volcanics according to TAS diagram: the scatter plot is in the Campi Flegrei field (Fig. 6c) and shows close affinity with the compositional fields of tephra materials related to the Campanian Ignimbrite (39-40 ka b.p.) and the Neapolitan Yellow Tuff (15 ka b.p.). Trachytes and phonolites from Campi Flegrei range from holocrystalline to almost glassy and feature phenocrysts of alkali feldspar, biotite, plagioclase, minor clinopyroxene, Fe-Ti oxides, and rare amphibole and sodalite-group minerals (Peccerillo, 2005b). These mineral components are very similar to the petrographic features identified in the analysed pumice clasts.

Therefore, the natural pozzolana used in the hydraulic mortars of Hippo Regius was imported from the Phlegraean District volcanics in the Gulf of Naples, as advised by Vitruvius (*De Architectura*, 2.6.1).

The Phlegraean source of the natural pozzolana is further investigated by comparing compositional variations in pumices' glassy groundmass with the geochemical trends of the three magma batches that erupted during the Baia–Fondi di Baia explosive events (Voloschina et al., 2018).

Variation diagrams of K_2O vs. SiO_2 (Fig. 7a) and Na_2O vs. MgO (Fig. 7b) show that the pumice aggregates follow the geochemical trends of batch B magma that has a trachytic composition with SiO_2 content between 60.3 and 64.7 wt.% and which represents the main volume of the products erupted by the Baia-Fondi di Baia explosive events.

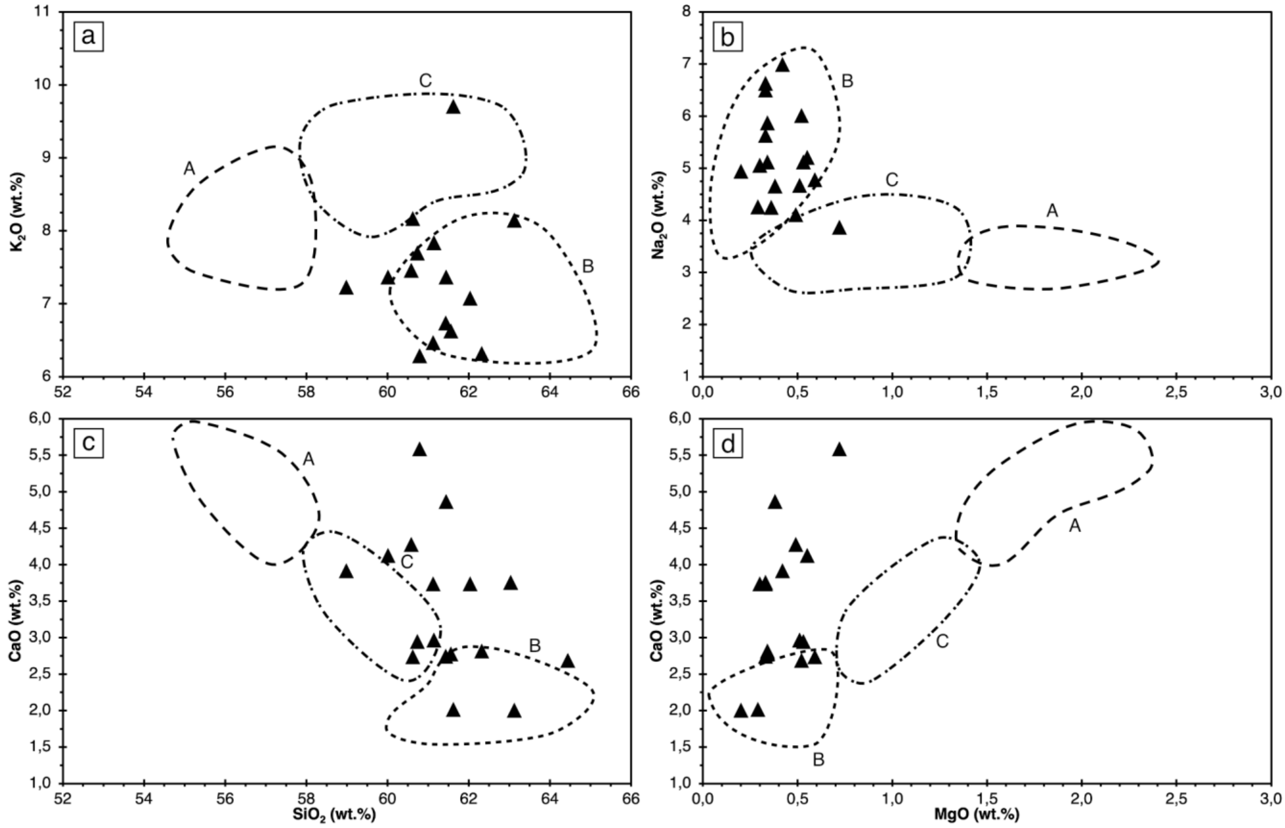

Figure 7. Variation diagrams of (a) K_2O vs. SiO_2; (b) Na_2O vs. MgO; (c) CaO vs. SiO_2; (d) CaO vs. MgO for matrix glasses of pumice clasts present in the Roman mortars of Hippo Regius. The letters A, B and C indicate the three distinct compositional batches of Baia-Fondi di Baia matrix glasses and rocks representative of Campi Flegrei (CF) magma bodies (data from Voloschina et al., 2018)

Furthermore, variation diagrams of CaO vs. SiO_2 (Fig. 7c) and CaO vs. MgO (Fig. 7d) confirm the relationship of the pumice aggregates with the magma body of group B. The outliers in scatter plots displayed in CaO vs. SiO_2 and MgO variation diagrams are ascribed to the secondary crystallisation of carbonates within the vesicular groundmass of pumice clasts.

This robust evidence indicates the link between the pozzolanic aggregates used in the mix of lime mortars in Hippo Regius, and their relationship with the geochemistry of the Baia-Fondi di Baia explosive events suggesting that the pozzolana may have been imported from the vicinity of *Baiae* (Fig. 8). Indeed, the volcanic products of this district were widely used by Romans as a fundamental component of mortars and plasters (Morra et al., 2010) and *Baiae* was mentioned twice by Vitruvius as a location from where natural pozzolana could be found (De Architectura, 2.6.1-2).

The trade of Phlegraean pyroclastic aggregates from southern Roman Italy and its shipments to other cities of the Empire across the Mediterranean is corroborated by previous studies related to the Roman Maritime Concrete Project (ROMACONS), where natural pozzolana was used in the concrete structures of *Chersonesus*, *Soli-Pompeiopolis* and *Caesarea Palestinae* harbours (Brandon et al., 2005; Stanislao et al., 2011; Vola et al., 2011), hence it is clear that the transport of pozzolana was part of a wider system of trade and transport, even to the eastern Mediterranean (Gianfrotta, 2011).

The maritime connection of Hippo Regius to the Empire is proven as well by at least eight imported lava millstones, four of which were made of red ignimbrite, probably from the *Mulargia* area (Antonelli et al., 2014) which is at a halfway point between the Phlegraean volcanic district and Hippo Regius (Fig.

8), and could be connected to the maritime cabotage between the two harbours.

This, combined with the data reported here, strongly support the hypothesis of the use of natural pozzolana in the provincial city of Hippo Regius that was imported by Romans from the Phlegraean District volcanics in the Gulf of Naples and probably from Baia-Fondi di Baia (in the Gulf of Pozzuoli, the modern *Puteoli*).

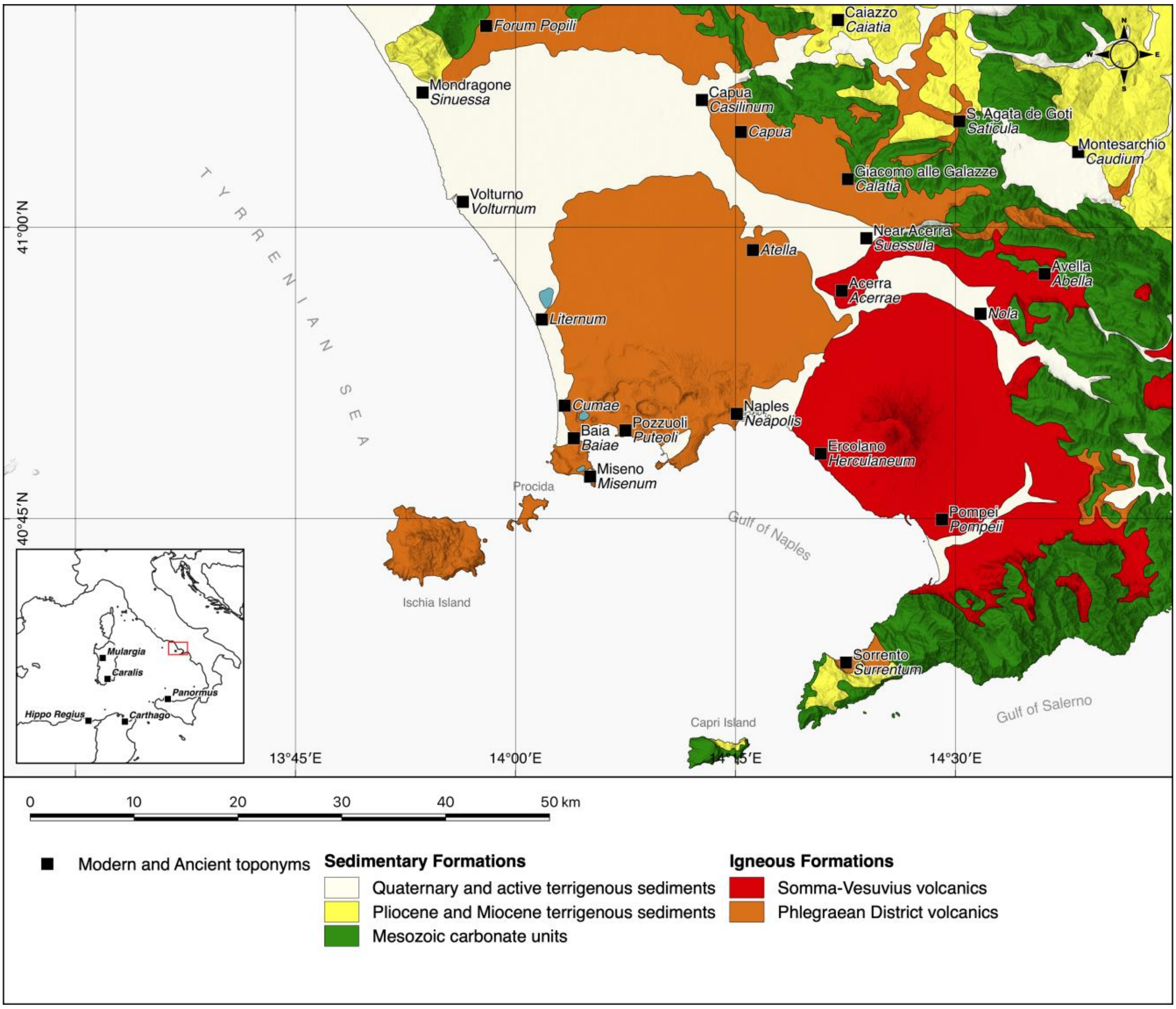

Figure 8. Geological sketch map of the Campania Magmatic Province showing sedimentary and igneous formations of Phlegraean District volcanics (Campi Flegrei) and Somma–Vesuvius volcanics (adapted from Orsi et al., 1999; Tomlinson et al., 2012; Peccerillo, 2020)

7. CONCLUSIONS

The investigation performed on samples of hydraulic mortars collected from the archeological site of Hippo Regius highlights the continuous use of volcanic aggregates as natural pozzolana in mortars of water-related structures built between the late 1st century AD and the first half of 4th century AD. The pozzolana was added to the mix of lime-based binders in order to improve the strength and hydraulic properties of mortars by producing pozzolanic reactions, confirmed by the presence of reaction rims around the pumice clasts.

Results of the multi-analytical characterisation indicate the use of local geomaterials such as limestones and quarry sands, which are well consistent with the surrounding geological settings. Crushed terracotta fragments obtained from the recycling of ceramic waste materials were added to provide additional pozzolanic effect.

Comparison of the chemical composition of representative pumice clasts that constitute the natural pozzolana with geochemical data of known pyroclastic deposits indicates that it was imported from the Phlegraean District volcanics and probably from Baia-Fondi di Baia in the Gulf of Pozzuoli.

As regards its shipping, it is known that Hippo Regius was an important harbour for grain export, and freighters that carried grain shipments to Roman Italy wouldn't return empty. It is hypothesized that they normally sailed back with sand and volcanic stones as a convenient ballast (Hohlfelder, 1999).

The new data deriving from this study represent a significant contribution to the knowledge of ancient building techniques in Hippo Regius which is crucial in the perspective of future restoration works. They also testify to the transmission of Roman know-how to local artisans of the city and their good knowledge of the Vitruvian recipes.

Future research should investigate the use of natural pozzolana in hydraulic mortars from other cities in both *Africa Proconsularis* and *Mauretania Caesariensis* and especially settlements in the Numidian hinterland to assess the breadth of Roman technologies and raw building materials exports in places linked to the Empire via terrestrial connections.

AUTHOR CONTRIBUTIONS

All co-authors contributed equally to conceptualization, methodology, investigation, resources, data curation, writing—original draft preparation, writing—review and editing, visualization, and funding acquisition. All authors have read and agreed to the published version of the manuscript.

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