ROMANS’ ESTABLISHED SKILLS: MORTARS FROM D46b MAUSOLEUM, PORTA MEDIANA NECROPOLIS, CUMA (NAPLES)

Claudia Di Benedetto, Sossio Graziano, Vincenza Guarino, Concetta Rispoli, Priscilla Munzi, Vincenzo Morra, Piergiulio Cappelletti

To cite this version:
Claudia Di Benedetto, Sossio Graziano, Vincenza Guarino, Concetta Rispoli, Priscilla Munzi, et al.. ROMANS’ ESTABLISHED SKILLS: MORTARS FROM D46b MAUSOLEUM, PORTA MEDIANA NECROPOLIS, CUMA (NAPLES). Mediterranean Archaeology and Archaeometry, 2018, 18, pp.131 - 146. 10.5281/zenodo.1285895 . hal-01861835

HAL Id: hal-01861835
https://hal.archives-ouvertes.fr/hal-01861835
Submitted on 18 Nov 2018

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L’archive ouverte pluridisciplinaire HAL, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d’enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.
ROMANS’ ESTABLISHED SKILLS: MORTARS FROM D46b MAUSOLEUM, PORTA MEDIANA NECROPOLIS, CUMA (NAPLES)

Claudia Di Benedetto*1, Sossio Fabio Graziano1, Vincenza Guarino1, Concetta Rispoli1, Priscilla Munzi2, Vincenzo Morra1, Piergiulio Cappelletti1

1DiSTAR - Dipartimento di Scienze della Terra, dell'Ambiente e delle Risorse - Università di Napoli Federico II, Via Cintia, 26 - Napoli.
2Centre Jean Bérard, USR 3133 CNRS - École française de Rome, Via Francesco Crispi, 86 - Napoli

Received: 17/12/2017
Accepted: 20/02/2018
*Corresponding author: Claudia Di Benedetto (claudia.dibenedetto@unina.it)

ABSTRACT

Roman mortars from a mausoleum (named D46b) belonging to the archaeological site of Porta Mediana necropolis, in Cuma (Naples, Southern Italy) have been studied by means of petrographic, mineralogical and micro-chemical analyses. The aim of this research is to fill the knowledge gap regarding mortar-based materials used in Roman age within this wide archaeological site.

Two typologies of mortars (bedding and coating) were collected from mausoleum’s masonry. They were lime-based with addition of pozzolanic materials, according to Vitruvius’ recipe.

Raw materials, such as volcanic sand and limestones, mainly from local sources, were preferentially used as aggregate, both for great availability and good properties.

As regard production techniques, the multi-layer feature of the coating mortars, once again shows the great knowledge of the building art. Each layer is the result of a precise choice, as shown by the differences both in texture and petrographic features.

Data from detailed mortars characterization have infer the outstanding skill of Roman craftsmen, as already proved by extraordinary durability of buildings.

The research was very useful not only to increase the knowledge of this ancient culture but also to planning conservative actions, that, through mortar reproduction or the research of suitable materials, can promote the safeguard of this invaluable heritage.

KEYWORDS: roman mortars, bedding, coating, raw materials, volcanic aggregate, technology
1. INTRODUCTION

Mortars represent one of the most important artificial building materials, used since Roman times (Collepardi, 2003; Elsen, 2006; Pecchioni et al., 2008, 2009). They are composite materials, characterized by natural aggregates added in lime binder, often related both to the place and the historical time in which they are made (Pecchioni et al. 2008; Collepardi et al., 2003). Roman mortars were used with different function: plasters (renders), on internal and external walls, supporting substrates for frescoes bedding mortars of masonry supporting materials for pavements or mosaics and watertight lining materials in cisterns, wells, aqueducts etc. (Moropolou et al., 2000).

Outstanding examples of well-preserved Roman mortars can be found in Campania region, with its considerable number of archaeological sites, where interests of researchers were focused.

Several studies mainly involved Vesuvian archaeological sites, such as Pompei (Bonazzi et al., 2007; Castriota et al., 2008; De Luca et al., 2015; Miriello et al., 2010; Piovesan et al., 2013), Ercolano (Leone et al., 2016; De Vita et al., 2010), and Stabia (Izzo et al., 2016). Within Phlegraean Fields area, instead, investigations have been carried out mainly on the nowadays underwater heritage of Baia (Aloise et al., 2013; La Russa et al., 2015; Ricci et al., 2009), while very few on emerged ones, as Piscina Mirabilis and Terme di Baia sites (Rispoli et al., 2015, 2016), although they represent an invaluable archaeological heritage. Relevant archaeometric studies on mortars have been quoted in other cases (Liritzis et al., 2015; Salama et al., 2017).

One of the most important archaeological site of Phlegraean Fields is the area of Cuma, which represents the oldest Greek colony of the Occident. In this wide archaeological area, a Necropolis, named Porta Mediana, was revealed by the Centre Jean Berard archeologists, between 2001 and present. The Porta Mediana necropolis consists of about 70 funerary monuments, ranging from 2nd BC and 6th century AD, developed in different building phases (Brun & Munzi, 2008; Brun et al., 2017). This site, in which geology and archeology are deeply related, was involved in a systematic study of natural and artificial geomaterials, carried out by strictly collaboration between DiSTAR and Centre Jean Béard, within the SINAPSIS Project (PON - SistemNAzionale Protezione SIti Sensibili).

Aim of this research is provide, by means of mineralogical and petrographic investigations, a full characterization of mortars from masonry boundaries of specific area, which includes a funerary monument, named D46b (1st century BC) and following commercial buildings (2nd - 3rd century AD). The main goal is fill the gap of knowledge about raw materials, their provenance and availability in the studied area. To this purpose the choice as fell on this area because it represents an important part of a complex stratigraphy sequence identified in this sector of Necropolis. Due to its strategic position along the oldest route of the Necropolis (dating back from archaic era; Brun et al., 2013), this funerary monument can provide useful preliminary information to understand the chronological building evolution of the Porta Mediana site (Brun et al., 2013).

2. GEOLOGICAL SETTING

The archaeological site of Cuma is located in the north-western sector of Phlegraean Fields volcanic district (Campania region, Southern Italy; Morra et al., 2010; Fedele et al., 2011; Fig. 1), that, along with Somma-Vesuvius, represent the two main still active, quaternary volcanoes in the Mediterranean area.

The volcanic activity, mainly explosive, deeply affected the morphology of the area, also providing geomaterials suitable for building purposes. Phlegraean Fields volcanism includes both continental (Campi Flegrei s.s.) and insular (Ischia and Procida Islands) activity. The oldest outcropping products belong to Ischia activity (150 Ka; Poli et al., 1987; Vezzoli, 1988), while as concern continental volcanism, the first activity is dated back to 58 Ka (40Ar/39Ar, Pappalardo et al., 1999). It is also divided by means of two volcanic events, the most important in terms of thickness and areal distribution. The first large eruption is represented by Campanian Ignimbrite deposits (39 Ka; Fedele et al., 2008), the second one is the Neapolitan Yellow Tuff (therefore NYT, 15 Ka; Deino et al., 2004). Both events are the main chronostratigraphic markers for the reconstruction of the Phlegraean volcanic district.
These deposits, linked to high-magnitude explosive eruptions, were accompanied by caldera collapse followed by emplacement of pyroclastic sequences (Orsi et al., 1996; Perrotta et al., 2006; Melluso et al. 2012 and references therein). The last eruption of Phlegraean Fields dates back to 1538 AD (Monte Nuovo) but the volcanic activity still carries on, as testifies by recent bradyseismic episodes and intense fumarolic activity.

The Phlegraean products range from shoshonitic basalt through latite, to trachyte, trachyphonolite and phonolite. The most common mineral phases are clinopyroxene, plagioclase, sanidine, biotite and magnetite with rare olivine. The accessory minerals are zircon, brown amphibole and titanite, whereas nepheline and exotic minerals (e.g. disilicates) can be found in most evolved products (Morra et al. 2010 and references therein; Melluso et al. 2012).

The Somma-Vesuvius central volcanic complex, is located in the southern part of Campanian Plain (Fig. 1). It is a stratovolcano formed by the oldest stratovolcano of Mount Somma, in which Vesuvius cone formed as consequence of 79 AD eruption (“Pompei”; e.g., Cioni et al., 1999; Rolandi et al., 1998, 2004). The Somma-Vesuvius volcanism showed a cyclic mechanism in which periods of quiescence were interrupted by Plinian or subplinian eruptions (Rolandi et al., 1998). The last eruption dates back to 1944. The Somma-Vesuvius volcanic rocks vary from potassic to ultrapotassic, with degree of alkalinity and thus silica undersaturation increasing with time.

These two volcanic complex rest on the Campanian Plain, a structural depression, bordered by Meso-Cenozoic carbonates forming the Southern Apennine chain to the N/E, the Sarno Mountains to the E, and Monti Lattari ridge to the S (Fig. 1).

3. THE ARCHAEOLOGICAL SITE

The archaeological site of Cuma (Campanian Region, southern Italy), includes also the Necropolis area located outside the Porta Mediana, one of the main gates of the ancient city. The funerary site is
divided in several sectors among which D46 represents one of the most interesting (Fig. 2). Excavated in 2012, it includes the D46b funerary complex (1st century BC), and later buildings, ranging in time from 2nd and 3rd century AD. It is set along the Eastern boundary of a square outside the city gate, in a strategic location to understand the chronological evolution of the Porta Mediana site (Fig. 2; Brun et al., 2013). The D46b funerary monument, slightly trapezoidal in shape and build in opus quasi-reticulatum, is not entirely preserved. The only original building is the burial chamber, probably semi-hypogeae (Fig. 3). All the walls are built against earth, so the chamber does not have exterior facings, while its inner part is cover by plaster.

The stratigraphic excavation allowed identifying at least two building phases corresponding to different intended use of the funerary monument.

To the first phase belong semi-circular structures in opus incertum made of yellow tuff bound with grey mortar, in which some niches, probably for cinerary urns, were carved. During the second phase the funerary room has undergone some transformations, including the construction of two funerary bed in replacement of urns along with the addition of partially preserved cocciopesto flooring.

During the 1st century AD at least three others tombs, today obliterated, were built within the sector. Between the end of the 1st century and the first decades of the 2nd century AD, the area underwent several transformations, including the realization of the Domitiana street and the monumentalization of the space in front of the Gate. Moreover, funerary buildings belonging to the previous phases were destroyed and a short time later a new public building is built along the eastern edge of the paving-stones area in front of the Porta Mediana. The latter consists of a monumental facade and some commercial buildings (Brun & Munzi, 2013).

From the middle of the 3rd century AD the area is progressively abandoned, while during the 6th century AD it becomes a source of building materials, in which marbles were used for lime production (Covolan & Lemaire, 2017).
4. MATERIALS AND METHODS

Eleven samples of mortars were collected from boundary masonries belonging to different building times ranging from 1st century BC to 3rd century AD, (Fig. 4; Tab. 1). They can be divided in two typologies: bedding and coating mortars. The first type includes six samples taken from opus quasi-reticolatum masonries; the coating mortars are represented by five samples of multi-layer covering (Tab. 1).

Instrumental methods included: 1) Optical Microscopy (OM) on thin sections; 2) X-Ray powder Diffractometry (XRPD); 3) Scanning Electron Microscopy and Energy Dispersive X-ray spectroscopy (SEM-EDS) analyses. All the analyses were performed at DiSTAR laboratories, University of Naples Federico II.

Optical microscopy was carried out by Leitz Laborlux 12 pol, in order to study composition and texture of the mortar (Elsen, 2005; UNI-EN 11305:2009 and UNI-EN 11176:2006). Images were captured by digital videocamera “Leica DFC 280”.

X-Ray powder diffractometry was carried out by Panalytical X’Pert Pro diffractometer equipped with a RTMS X’Celerator detector (CuKα radiation, 40 kV, 40 mA, 2θ range from 4° to 70°, equivalent step size 0.017° 2θ, 30 s per step counting time) both on binder and aggregate, mechanically separated after powdered sample by hand, in order to preserve soluble
phases, following the UNI 11305 recommendations, in an agate mortar. Identification of mineral phases was performed by Panalytical Highscore Plus 3.0e software with ICSD database.

SEM-EDS analyses were performed on not altered juvenile fragments from thin sections to distinguish the nature of aggregate and determine chemical composition of mortar binder. An Energy Dispersive Spectrometer (JEOL JSM-5310 microscope and an Oxford Instruments Microanalysis Unit, equipped with an INCA X-act detector and operating at 15 kV primary beam voltage, 50-100 mA filament current, variable spot size, from 30,000 to 200,000x magnification, 20 mm WD and 50 s net acquisition real time) was employed referring to Guarino et al. (2017) and references therein. Measurements were made with an INCA X-stream pulse processor and with INCA Energy software. The following standards were used for calibration: diopside (Ca), San Carlos olivine (Mg), anorthoclase (Al, Si), albite (Na), rutile (Ti), fayalite (Fe), Cr₂O₃ (Cr) rhodonite (Mn), orthoclase (K), apatite (P), fluorite (F), barite (Ba), strontianite (Sr) and sodium chloride (Cl). Backscattered electron (BSE) images were obtained with the same instrument.

<table>
<thead>
<tr>
<th>Table 1. Type of sampled mortars.</th>
</tr>
</thead>
<tbody>
<tr>
<td>SAMPLE</td>
</tr>
<tr>
<td>MR1</td>
</tr>
<tr>
<td>MR2</td>
</tr>
<tr>
<td>MR3</td>
</tr>
<tr>
<td>MR4</td>
</tr>
<tr>
<td>MR5</td>
</tr>
<tr>
<td>US</td>
</tr>
<tr>
<td>SP1</td>
</tr>
<tr>
<td>D46</td>
</tr>
<tr>
<td>46BM</td>
</tr>
<tr>
<td>BS1</td>
</tr>
<tr>
<td>BS2</td>
</tr>
</tbody>
</table>

5. RESULTS

5.1 Optical microscopy

Optical microscopy was carried out on all mortar samples to obtain qualitative information about the mix-design of lime-based mortars and their aggregate.

Bedding mortars (MR1, MR2, SP1, BS1, US, 46BM samples) are characterized by a mixture of light olive grey (5Y 6/2 Munsell) lime matrix and volcanic aggregate. The aggregate/binder ratio, observed in all thin sections always exceedes the 50%, according Normal 11176:2006. Aggregate is poorly sorted (Gagliardi et al., 1980) with medium -low rounded clasts (Krumbein & Sloss, 1979), ranging in size from fine (less than 2 mm) to coarse (up to 7 mm in size), consisting of pyroclastic fragments, such as pumice (sometimes with sanidine and clinopyroxene) and obsidian, and subordinate crystal-clasts of sanidine, clinopyroxene, plagioclase and mic (Fig. 5a). Lime lumps occur in almost all samples, except in BS1 and MR1; in this latter, as well as in the MR2 sample carbonate clasts are also identified (Fig. 5b).

Coating mortars are composed by different layers, shown in table 2, along with relative thicknesses.

Coating mortars (MR3, MR4, MR5, BS2, D46 samples) are composed at least by two layers: the innermost (layer 0), representing the anchorage layer, also named scratch layer, followed by layer 1 (arriccio) (Izzo et al., 2016 and references therein).

| Table 2. Stratigraphy of mortar sample with thickness of identified layers |
|-----------------------------|----------------|--------------|
| Anchorage | Arriccio | Plaster | Painting |
| MR3       | 6 mm      | 1.6 mm      |
| MR4       | 5 mm      | 3 mm        |
| MR5       | 9 mm      | 3 mm        |
| BS2       | 8 mm      | 2 mm        |
| D46       | 5 mm      | 0.8 mm      | 39 μm and 105 μm |

Anchorage layers show 2.5Y 7/3 (Munsell) colored matrix, mixed mainly with volcanic aggregate. Matrix shows from cryptocrystalline to micritic texture, generally deriving from pozzolanic reaction between binder and aggregate. The aggregate/binder ratio ranging from 40% (D46) to 50% (MR4, MR5, BS2). The aggregate is ranging in size from fine (2μm) to coarse grained (up to 13 mm), except for D46 sample, showing predominant fine grain size. Shape of the anchorage layer aggregate mostly varies from sub-angular to sub-rounded (Krumbein & Sloss, 1979) with poorly sorted size distribution (Gagliardi et al., 1980). The thickness of this layer ranges from 5 mm (MR4 and D46) to 9 mm (MR5).
Volcanic aggregates are characterized, in order of abundance, by pumice (sometimes with alkali feldspar and clinopyroxene) and obsidian (fragments), along with sanidine, clinopyroxene, plagioclase and brown mica grain. Crystal fragments are particularly abundant in MR5 and MR4 samples (Fig. 5c) comparing to the anchorage layer of MR3, D46 and BS2 samples (Fig. 4d). Subspherical or elongated lime lumps, ranging from 200 μm to 4 mm, with well-defined edges, are often observed in the matrix. Shrinkage cracks (in the middle area), are also observed (Fig. 5e). Calcareous clasts occurrence was detected in MR3, D46 and BS2 samples.

The transition to arriccio (layer 1) is well marked in all coating mortars (except in BS2 samples, in which well-defined layers cannot be identified, Fig. 5d), also for the occurrence of few ceramic fragments and volcanic aggregates in subordinate amount with respect to layer 0.

The arriccio layer can be recognizable in BS2, MR5 and MR4 samples. It can be distinguished from anchorage layer mainly for the features of aggregate. In this layer, the aggregate is less abundant compared to layer 0 (about 40%), ranging in size from fine grained (less than 2 mm) to a maximum of 3 mm (MR5 sample). It appears moderately sorted (Krumbein & Sloss, 1979) with medium roundness of grains (Gagliardi et al., 1980), also revealing occurrence of ceramic fragments (Fig. 5f). The thickness of this layer range from 3 mm (MR4 and MR5) to 2 mm (BS2).

The complete stratigraphic sections, which should include both preparation (plaster) and finishing (painting) layers, are not preserved in all samples. Among investigated coating mortars only two samples show this kind of layers: D46 and MR3 (Fig. 5g, h). In both samples arriccio layer is lacking, while a thick layer (0.8-1.6 mm for D46 and MR3, respectively) of plaster can be easily distinguished. The main differences between these two samples regard:

a) occurrence of calcareous aggregate (maximum grain size 2 μm); b) presence of shells, identified only in MR3 sample.

As concern outermost layers, D46 sample shows two preparations (layers 2 and 3), very similar from petrographic point of view and characterized by carbonate dense binder, with thickness of 39 μm and 105 μm, respectively. Instead, the MR3 sample has only a very thin paint finishing layer.

Figure 5. Representative images of: a) volcanic fragments, US, (PPL); b) carbonate fragments, US (CPL); c) abundant crystal fragments in anchorage layer, MR3 sample (thin section scan); d) low amount of crystal fragments in anchorage layer, MR3 sample (thin section scan); e) CaCO3 lime lump showing shrinkage cracks, D46 (CPL); f) ceramic fragments in arriccio layer, MR3 (CPL); g) undefined layers in the BS2 sample (thin section scan); h) anchorage, plaster and two preparation layers, D46 (PPL) i) anchorage, plaster anchorage, plaster with calcareous aggregates and paint finishing layer MR3 (PPL).

PPL: Parallel Polarized Light; CPL: Cross Polarized Light.

Mediterranean Archaeology and Archaeometry, Vol. 18, No 5, (2018), pp. 131-146
5.2 X-ray powder diffraction

XRPD results confirmed occurrence of lime-based mortar with volcanic aggregate, as shown by semi-quantitative analyses reported in Table 3, in which aggregate and binder are identified by the A and B at the end of the abbreviation, respectively.

Calcite is the most abundant binder phase, even if a subordinate amount could be derived also from calcareous clasts not well separated during the mechanical grinding. Sanidine, mica-group minerals and subordinate analcime were detected in all samples. Phillipsite was recognized, in small amounts, in many samples (except in BSP1B and MR2B). Plagioclase only occurs in three samples: BSP1B, MR2B and USB. Small amount of quartz was also detected in BS2B sample. Finally, vaterite, a calcium carbonate polymorph, metastable under ambient conditions, was observed in MR2B and MR4B samples. Its occurrence is probably related to a) alteration of carbonate binder, which stabilizes in the presence of organic compound or b) formed as an alteration product of C-A-S-H (hydraulic binder) in both subaerial and submarine environments (Brandon et al., 2014).

XRPD analyses also highlight calcite as the main component of the aggregate (Table 3). However, its presence might be related to the binder fraction remained in contact with aggregate fragments, as consequence of mechanical separation.

Table 3. Semi-quantitative mineralogical analyses (XRPD) of binder and aggregate samples: ++++ predominant, +++ abundant, ++ moderate, + scarce. Mineral abbreviations from (Siivola & Schmid, 2007; Whitney & Evans, 2010).

<table>
<thead>
<tr>
<th>BINDER</th>
<th>cal</th>
<th>sa</th>
<th>pl</th>
<th>mca</th>
<th>php</th>
<th>cbz</th>
<th>anl</th>
<th>vtr</th>
<th>qtz</th>
<th>px</th>
<th>hem</th>
</tr>
</thead>
<tbody>
<tr>
<td>BS1B</td>
<td>+++</td>
<td>+</td>
<td></td>
<td>+</td>
<td></td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>BS2B</td>
<td>+++</td>
<td>+</td>
<td>+</td>
<td></td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>BSP1B</td>
<td>+++</td>
<td>+</td>
<td>+</td>
<td></td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>BMB</td>
<td>+++</td>
<td>+</td>
<td>+</td>
<td></td>
<td></td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>MR1B</td>
<td>+++</td>
<td>+</td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>MR2B</td>
<td>++</td>
<td>+</td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td>++</td>
</tr>
<tr>
<td>MR3B</td>
<td>+</td>
<td>++</td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>MR4B</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td>++</td>
</tr>
<tr>
<td>MR5B</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>46BB</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>USB</td>
<td>+++</td>
<td>++</td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>AGGREGATE</th>
<th>cal</th>
<th>sa</th>
<th>pl</th>
<th>mca</th>
<th>php</th>
<th>cbz</th>
<th>anl</th>
<th>vtr</th>
<th>qtz</th>
<th>px</th>
<th>hem</th>
</tr>
</thead>
<tbody>
<tr>
<td>BS1A</td>
<td>+++</td>
<td>+</td>
<td>+</td>
<td></td>
<td></td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>BS2A</td>
<td>+++</td>
<td>+</td>
<td>+</td>
<td></td>
<td></td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>BSP1A</td>
<td>+++</td>
<td>+</td>
<td>+</td>
<td></td>
<td></td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>BMA</td>
<td>+++</td>
<td>+</td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>MR1A</td>
<td>+++</td>
<td>++</td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>MR2A</td>
<td>+</td>
<td>+</td>
<td>++</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td>+++</td>
</tr>
<tr>
<td>MR3A</td>
<td>+++</td>
<td>++</td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>MR4A</td>
<td>++</td>
<td>++</td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td>++</td>
</tr>
<tr>
<td>MR5A</td>
<td>+++</td>
<td>++</td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>46BA</td>
<td>++</td>
<td>+++</td>
<td>++</td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>USA</td>
<td>+++</td>
<td>++</td>
<td>+</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>BS2C</td>
<td>++</td>
<td>+</td>
<td>++</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>+</td>
<td></td>
</tr>
</tbody>
</table>

Only for MR2 and MR4 mortars, both in aggregate and binder samples (MR2A, MR2B, MR4A and MR4B), XRPD results highlight the presence of vaterite, along with calcite, sanidine, chabazite, phillipsite (MR4A), plagioclase (MR4) and mica.
Concerning ceramic fragments from BS2C sample, they are mainly composed by calcite, quartz and, subordinately, by plagioclase, sanidine, pyroxene, mica and hematite. X-ray patterns also suggest presence of a Low Ordered or Amorphous Phase (LO-AP), that may be related to volcanic glass components (juvenile fragments) and hydraulic phases (C-A-S-H; Jackson et al., 2010) formed after pozzolanic reactions between lime and volcanic glasses or cocciopesto grains (obtained crushing shattered tiles, brick and pottery; Fernandez et al., 2010; Middendorf et al., 2005; Izzo et al., 2016).

5.3 **Electronic microscope observation and chemical analyses of minerals**

SEM-EDS observations performed on the binder allow to identify some phases characterized by ‘spongy’ morphology (Garcia et al., 2009; Fernandez et al., 2010) that highlight, as suggest by XRPD results, the formation of a newly hydraulic phases (C-A-S-H; Fig. 6), attesting the reaction between lime and pozzolanic materials.

![Figure 6. SEM image and relative EDS spectrum of neo-formed hydraulic phases (C-A-S-H), BS1 sample.](image)

As regards aggregate, SEM-EDS analyses, according to XRPD data, once again evidence the presence of phillipsite with well-defined prismatic crystal habit and of pseudocubic crystals of chabazite, often associated with acicular crystals of phillipsite (Fig. 7; de’ Gennaro et al., 1999).

![Figure 7. SEM image of phillipsite (on left) and chabazite (on right) crystals from MR1 sample.](image)

In order to infer provenance of volcanic aggregate, SEM-EDS microanalysis was carried out on fresh juvenile fragments (Fig. 7, Table 4). Investigations were performed on three points for each pumice. The occurrence of many altered pumice required a careful selection of analyses closing between 98% and 100%, to exclude altered ones. Chemical classification (TAS diagram, according to Le Maitre et al., 1989) of investigated pumice shows a trachytic/phonolitic composition (SiO$_2$ = 59.6-63.9 wt.%; Na$_2$O+K$_2$O = 12.4-13.7 wt.%; Fig. 9).
Table 4. Major element concentrations (wt.%, by EDS), pumice.

<table>
<thead>
<tr>
<th>Element</th>
<th>MR1a</th>
<th>MR1b</th>
<th>MR2</th>
<th>MR3</th>
<th>MR4</th>
<th>MR5</th>
<th>SP1</th>
<th>BS1</th>
<th>BS2</th>
<th>US</th>
<th>46BM</th>
<th>D46</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>61.6</td>
<td>60.3</td>
<td>60.2</td>
<td>63.2</td>
<td>62.0</td>
<td>60.0</td>
<td>60.0</td>
<td>59.6</td>
<td>63.9</td>
<td>63.9</td>
<td>63.2</td>
<td>61.6</td>
</tr>
<tr>
<td>TiO₂</td>
<td>0.43</td>
<td>0.82</td>
<td>0.33</td>
<td>0.50</td>
<td>0.12</td>
<td>0.82</td>
<td>0.70</td>
<td>0.55</td>
<td>0.49</td>
<td>0.11</td>
<td>0.36</td>
<td></td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>18.3</td>
<td>18.0</td>
<td>18.0</td>
<td>18.2</td>
<td>17.9</td>
<td>18.5</td>
<td>18.2</td>
<td>18.1</td>
<td>17.2</td>
<td>17.7</td>
<td>18.2</td>
<td></td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>2.45</td>
<td>3.06</td>
<td>2.90</td>
<td>2.34</td>
<td>3.18</td>
<td>2.60</td>
<td>3.50</td>
<td>3.20</td>
<td>2.31</td>
<td>2.61</td>
<td>2.70</td>
<td></td>
</tr>
<tr>
<td>MnO</td>
<td>0.13</td>
<td>0.07</td>
<td>0.20</td>
<td>0.12</td>
<td>0.00</td>
<td>0.05</td>
<td>0.21</td>
<td>0.35</td>
<td>0.09</td>
<td>0.26</td>
<td>0.35</td>
<td></td>
</tr>
<tr>
<td>MgO</td>
<td>0.37</td>
<td>0.45</td>
<td>0.25</td>
<td>0.24</td>
<td>0.49</td>
<td>0.51</td>
<td>0.57</td>
<td>0.47</td>
<td>0.31</td>
<td>0.27</td>
<td>0.19</td>
<td></td>
</tr>
<tr>
<td>CaO</td>
<td>2.21</td>
<td>2.20</td>
<td>2.60</td>
<td>1.72</td>
<td>2.28</td>
<td>2.24</td>
<td>2.53</td>
<td>2.31</td>
<td>1.50</td>
<td>1.39</td>
<td>2.00</td>
<td></td>
</tr>
<tr>
<td>Na₂O</td>
<td>4.40</td>
<td>4.50</td>
<td>4.40</td>
<td>4.54</td>
<td>3.89</td>
<td>4.29</td>
<td>4.35</td>
<td>3.67</td>
<td>5.96</td>
<td>5.74</td>
<td>4.86</td>
<td></td>
</tr>
<tr>
<td>K₂O</td>
<td>8.72</td>
<td>8.72</td>
<td>9.30</td>
<td>8.57</td>
<td>8.99</td>
<td>9.32</td>
<td>8.77</td>
<td>8.73</td>
<td>6.97</td>
<td>7.07</td>
<td>8.45</td>
<td></td>
</tr>
<tr>
<td>P₂O₅</td>
<td>0.08</td>
<td>0.23</td>
<td>0.30</td>
<td>bdl</td>
<td>bdl</td>
<td>bdl</td>
<td>0.36</td>
<td>0.22</td>
<td>0.26</td>
<td>bdl</td>
<td>0.07</td>
<td>0.12</td>
</tr>
<tr>
<td>SO₃</td>
<td>0.07</td>
<td>0.27</td>
<td>0.10</td>
<td>0.17</td>
<td>0.02</td>
<td>bdl</td>
<td>0.30</td>
<td>0.18</td>
<td>0.18</td>
<td>0.22</td>
<td>0.04</td>
<td></td>
</tr>
<tr>
<td>BaO</td>
<td>0.21</td>
<td>bdl</td>
<td>0.30</td>
<td>bdl</td>
<td>0.28</td>
<td>bdl</td>
<td>bdl</td>
<td>bdl</td>
<td>0.20</td>
<td>0.19</td>
<td>bdl</td>
<td></td>
</tr>
<tr>
<td>SrO</td>
<td>0.18</td>
<td>1.09</td>
<td>0.60</td>
<td>bdl</td>
<td>bdl</td>
<td>0.21</td>
<td>0.82</td>
<td>0.48</td>
<td>0.67</td>
<td>0.83</td>
<td>bdl</td>
<td>0.46</td>
</tr>
<tr>
<td>Cl⁻</td>
<td>0.81</td>
<td>0.51</td>
<td>0.70</td>
<td>0.53</td>
<td>0.70</td>
<td>0.53</td>
<td>0.62</td>
<td>0.57</td>
<td>bdl</td>
<td>0.83</td>
<td>0.76</td>
<td></td>
</tr>
<tr>
<td>F</td>
<td>bdl</td>
<td>bdl</td>
<td>bdl</td>
<td>bdl</td>
<td>bdl</td>
<td>bdl</td>
<td>bdl</td>
<td>bdl</td>
<td>bdl</td>
<td>bdl</td>
<td>0.29</td>
<td>bdl</td>
</tr>
<tr>
<td>Na₂O+K₂O</td>
<td>13.1</td>
<td>13.2</td>
<td>13.7</td>
<td>13.1</td>
<td>12.9</td>
<td>13.6</td>
<td>13.1</td>
<td>12.4</td>
<td>12.9</td>
<td>12.8</td>
<td>13.3</td>
<td></td>
</tr>
</tbody>
</table>

bdl: below detection limit

Figure 8. Back Scattered-SEM images of pumice from a) MR1 and b) BS1 samples.

Figure 9. Classification of pumice fragments from D46b funerary monument samples according to the TAS diagram (Le Maitre et al., 1989). Compositional fields of Phlegraean Fields and Somma-Vesuvius products and volcanic glasses are also reported (data from Morra et al., 2010 and references therein; figure modified after De Bonis et al., 2016).
6. DISCUSSION

The study allowed to characterize several mortars belonging to D46 sector.

The obtained chemical and mineralogical results, combined with archaeological data, provide significant information about the D46 bedding (MR1, MR2, US, SP1, 46BM, BS1) and coating (MR3, MR4, MR5, D46, BS2) mortars samples. Although the existence of different typologies already testifies the high knowledge and skill of Roman craftsmen, only a detailed investigation allowed to point out the provenance of raw materials and used technology.

6.2 Provenance

Coating and bedding mortars were made by a mixture of lime with three different aggregate types: limestone, volcanic sand, ceramic fragments, these latter used only for coating purposes.

As regard limestone, mainly used for binder and aggregate, is not possible precisely to assign this material to specific formation. However, it is reasonable to hypothesize that this geomaterial comes from Mesozoic limestone outcrops that border Campania Plain (Fig. 1).

Aggregate has predominantly a volcanic origin and it mainly consists of juvenile products (i.e.: pumice, scoriae and obsidian fragments), along with crystalline phases, such as sanidine, clinopyroxene, plagioclase and brown mica crystal clasts. Local source of volcanic aggregate is firstly suggested by the location of Necropolis within Phlegrean Fields area (Figg. 1; 10), in which volcanic material (tuffs) have been widely exploited as aggregates mixed with pozzolana and lime to obtain mortars since Roman times (Vitruvio, De Architectura). Moreover, the result of mineralogical analyses carried out on volcanic aggregate highlighted the presence of phillipsite, chabazite and analcime, the typical zeolitic association of Phleagrean tuffs, namely Neapolitan Yellow Tuff (NYT) and Campanian Ignimbrite-Yellow facies (de Gennaro et al., 2000; Langella et al., 2013). This evidence is also confirmed by the chemical analysis of pumice fragments that, according to the TAS diagram, follow the compositional trend of rocks from Phleagrean Fields (Fig. 9). A further and more specific indication might come from previous studies performed on Piscina Mirabilis and Thermal Complex of Baia, two Phlegraean archaeological sites (Rispoli et al., 2015; 2016), pointing out that in these site mortars were obtained by mixing lime with volcanic aggregate from NYT deposit. Moreover, such hypothesis could be consistent with the occurrence of NYT outcrops close to the city of Cuma (Fig. 10).

All the results highlight that no significant differences occurred in terms of composition between bedding and coating mortars, except for the presence, in the latter, of ceramic fragments, probably used in addition to pumice to improve pozzolanic reactions.

6.3 Technology

As regards production techniques, the bedding and coating mortars are lime-based with addition of pozzolanic materials, according to Vitruvius’ recipe (De Architectura), sometimes along with cocciopesto.
Lime lumps are common within binder. They represent unreacted lime related to dry-slaking process. Along with lime lumps, carbonate aggregates were found and linked to incomplete decarbonation processes during lime production, probably due to low temperature or insufficient calcination time (Bakolas et al. 1995; Barba et al., 2009; Yaseen et al., 2012). The occurrence of both lime lumps and carbonate aggregates testifies for a not accurate mortars manufacturing technology.

Further information about used technology come from the identification, by means of EDS-SEM analyses, of newly formed hydraulic phase, the gel-C-A-S-H. These hydrated calcium and aluminium silicates could derive from reaction between silica and alumina contained in the “pozzolanic” material (volcanic and ceramic fragments) commonly added to the mix design.

However, not many information about these manufacturing techniques has been so far reported in literature data (Drdacky et al., 2013).

Bedding mortars result from a mixture of slaked lime, fine to coarse-grained volcanic fragments and carbonate rocks. Coating mortars are result from a mixture of slaked lime, fine grained volcanic, ceramic and carbonate aggregates.

Moreover, as concern the coating mortars, they have been using a multi-layered technique, which, from the inner to the outer, has provided: anchorage, arriccio, plaster and preparation layer. Each layer shows different features, depending on its purpose.

The anchorage layer, the innermost, is made by poorly sorted and rounded volcanic aggregate (mainly pumice and obsidian) embedded in a carbonate matrix. The scratchy fabric of the anchorage layer ensures the good adhesion to the wall, making easy the ‘arriccio’ application. The arriccio layer shows less abundant aggregate with respect to the anchorage layer. The aggregate, moderately sorted with medium roundness, is characterized by ceramic fragments that, as pozzolana, also provide further strength to the mortars-based too. This layer anyway is not present in all investigated samples.

The plaster and preparation layers, not always well preserved, are characterized by lower amount of aggregate (mostly with carbonate composition). The preparation layer shows a finest grain size and a reduced porosity more suitable for following painting layers.

7. CONCLUSIONS

The research pointed out new insights about technological performances of Roman craftsmen, producing different mortars from D46 funerary monument.

Results were obtained combining chemical, mineralogical and archaeological data with the aim of: 1) characterized the used mix-design, 2) identify raw materials, 3) improve the knowledge on manufacturing techniques in Roman time.

The location of the Necropolis in the Phlegrean Fields granted craftsmen a large amount of geomaterials, widely used for mortar preparation. Indeed, in both types of mortars (bedding and coating), the use of volcanic materials of local origin has been recognized.

Investigated mortars, though produced over a wide period of time, show continuity in the choice of materials and used technology.

All lime-based mortars, with addition of volcanic aggregate (pumice, obsidian, lithic, and crystal fragments of alkali feldspar, plagioclase, pyroxene and mica), suggesting that mix design was obtained by mixing lime and volcanic phlegrean products. The use of the Neapolitan Yellow Tuff is strongly suggested by the characteristic mineral assemblage (phillipsite, cabasite and analcime), and also by the presence of a tuff cone relict (Monte Grillo) very close to Necropolis.

However, no sufficient elements allow to exclude also the use of Campanian Ignimbrite yellow facies, characterized by a mineralogical composition very similar to the Neapolitan Yellow Tuff one.

The limestone, used for both binder production and as main aggregate (coating mortars and preparation layers), may have been quarried from carbonate rocks that surround the Campanian Plain.

As far as production techniques are concerned, detailed information arises from coating mortars. In fact, although the innermost anchoring mortars (scratch layers) show presence of lime lumps, associated with defects in production technique, multiple layering allows to hypothesize a good knowledge by craftsmen of geomaterials and their properties.

Such good knowledge is further confirmed by the choice of a) coarse grain size for the aggregate of the anchoring layer and b) addition of ceramic fragments, which provide additional pozzolanic effect.

New data deriving from the study of these Roman mortars may represent a significant contribution to the knowledge of the ancient, but surprisingly actual, constructive techniques.

This research is also the starting point for comparing mortars from numerous funerary monuments of the same site with the aim of reproducing mortars of high durability and/or finding suitable restoration materials.
ACKNOWLEDGEMENTS

The authors wish to thank Dott. Roberto de Gennaro for his kind support in electron-microprobe determinations and backscattered electron images and Dott. Sergio Bravi for his technical ability in thin sections preparation. This research was granted by national research project PON “SINAPSIS” (Sistema Protezione Siti Sensibili – PON01_01063).

REFERENCES


