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MORTAR RECIPES THROUGH THE AGES. A BRIEF REVIEW OF DATA FROM PREHISTORY TO LATE ANTIQUITY

ABSTRACT

Mortars, composite materials used to bind together masonry elements and to seal and waterproof architectural surfaces, have been employed by human populations for sheltering purposes since the beginning of technological evolution of mankind, constituting the first attested products of pyrotechnology since the Neolithic period. Over times, selection of raw materials and optimization of recipes and mixing procedures allowed to diversify and optimize their properties, in order to meet the most sophisticated demands of human cultures. In this contribution, a brief excursus of the evolution of mortar technology from the Palaeolithic to the late Roman times is reported, with a particular focus on the selection of functional compounds to push some specific properties of these binding composites within differentiated human societies.

KEYWORDS: ANCIENT MORTARS, LIME, CLAY, GYPSUM, POZZOLANIC AGGREGATES, OPUS CAEMENTICIUM, COCCIOPESTO, NATURAL HYDRAULIC LIME

INTRODUCTION

Mortars are man-made lithoid building materials composed of a combination of inorganic and sometimes organic elements. Two components are necessary for the production of a mortar, namely the binder and the aggregates.

Binders are materials that, when mixed with water, form plastic blends that harden to a solid compound after a certain timeframe. The main binders employed in antiquity are clay, gypsum and, in particular, lime.

Aggregates are all those natural or artificial materials constituting the volumetrically dominant skeleton of the mortars, bound together by binder reaction, and preventing the formation of fractures in the compound after the complete evaporation of water.

Mortars produced in antiquity were not standardised. The physical (porosity, permeability, hydraulicity, etc.) and mechanical (tensile strength, toughness) properties of ancient mortars can vary on the basis of the selection and mixing of the components. Different compositions are influenced by the local availability of raw materials, mixing techniques and the different context of use (i.e. wall joints, foundations, flooring). In this paper, a brief review of the evolution of mortars' making techniques and selection of raw materials from the Palaeolithic to the late Roman times will be presented.

Fig. 1. Palatial Knossos (Crete), wall-paintings (partially restored) (Dilaria in press).

PRE AND PROTOHISTORY

The oldest binder used in antiquity is clay. The earliest cases date back to the Palaeolithic period, with several evidence from African sites. During the Neolithic period, in Mesopotamia, Persia and Anatolia, clay-based mortars developed and were intensively applied for the lining of walls and floors. This kind of mortar was also used as a mild waterproofing agent for wooden roofs and sometimes as a binder for wall joints (Artioli, Secco, Addis 2019; Hobbs, Siddall 2011). Several examples come from the site of Çatal Höyük in Anatolia (Hodder 2006).

The earliest evidence of production of lime-based mortars come from the site of Hayonim in Israel, dated to the 11th millennium BC (Kingery, Vandiver, Prickett 1988). Later evidence are attested in other sites in the Near and Middle East dated to the 8th to 7th millennium BC. In these contexts, mortars were primarily used as revetments of floors and walls (Kingery, Vandiver, Prickett 1988).

In a period chronologically akin to the Near-Middle Eastern cases, lime-making technology developed in the Danubian area, as demonstrated by the findings at the Neolithic (or perhaps even Mesolithic) site of Lepenski Vir in Serbia (Srejović 1972). It is unclear, however, whether this evidence should be regarded as a fortuitous and isolated experience, completely unrelated to eastern examples. However, simple lime-based mortars identified in some Neolithic sites in Thrace and in the Greek's Ionian islands (Kefalonia) (Wright 2005) suggest some type of transmission of the technology from the Levantine area to the Danubian-Balkan territories.

It has also been observed that as early as the Aceramic Neolithic period, the production of hydraulic mortars may have been experimented for the first time. At Aşikli Höyük in Anatolia (Hauptmann, Yalcin 2001), some rudimentary hydraulic mortars were obtained by mixing aerial lime with reactive volcanic tuffs and vegetable ash. However, the case appears isolated so far and probably accidental, as the first mixtures displaying the same properties spread in the Mediterranean several millennia later.

Fig. 2. Plastered water-tank from ancient Paleros, Acarnania, Greece (5th century BC) (Dilaria in press).

Regarding Egypt, it is generally believed that the use of lime binders was unknown, or extremely limited. A prototype of limestone-based concrete has been found in the pyramids at Giza (Barsoum, Ganguly, Hug 2006). In this territory, until the Ptolemaic period, the production of gypsum-based mortars primarily developed (i.e. Hemeda, Sonbol 2020). Thanks to the local availability and ease of firing of evaporitic stones (~200 °C), gypsum binders got a wide utilization in this territory, mainly as renders, at least since the Predynastic period (c. 5000-3150 BC), or even earlier. Other protohistoric evidence of the production of gypsum-based mortars come from the island of Cyprus (Philokyprou 2019).

During the Bronze Age, lime-based binders started being ordinarily employed by the Aegean societies. Several evidence come from all the major palatial sites in Crete, where these mortars were primarily used for the revetment of floors or walls. Wall plasters were then often painted in wall-paintings (Fig. 1) (Jones 2005). On the other hand, the use of mortars as structural elements is lacking.

From these periods the first "pozzolanic" mortars, started being systematically produced. The pozzolanic reactivity in a mortar-based material occurs when some aggregates chemically interact with the calcium hydroxide $Ca(OH)^2$, namely the "slaked lime" (portlandite) in a liquid solution. The chemical nature and mineralogical structure of the reaction products depend primarily on the content of free silica and aluminum from the aggregate that entered into reaction with the lime (Dilaria et al. 2022a). Some pozzolanic mortars, obtained by mixing aerial lime with *terracotta* fragments, are attested in some sites from Cyprus dated to the Late Bronze Age (late 2nd millennium BC) (Theodoridou, Ioannou, Philokyprou 2013).

CLASSICAL AND HELLENISTIC AGE

From the Minoans, the pyrotechnology for the production of lime binders was transferred to the Mycenaean culture and, in historical times, endorsed by the Greek societies. In these periods, mortars continued being primarily used for the revetment of hydraulic infrastructures (Fig. 2) or

Fig. 3. Mortar bedding of a Hellenistic mosaic in Pella (4th century BC) (photo by Simone Dilaria).

for the production of floor surfaces and floor beddings (Fig. 3). The first waterproofing pozzolanic mortars produced by adding pyroclastic rocks to lime-based mortars are dated to the Late Classic and Hellenistic Age and were primarily used for the revetment of hydraulic infrastructures. Among the earliest cases, dating to the 4th-3rd c. BC, there are the (revetment?) concretes of the cisterns at Kamiros in Rhodes, analysed in the late 1890s (Koui, Ftikos 1998). However, this case needs further analytical in-depth study in light of the advancement in this field of research. Other analyses have detected reactive tuff fragments in the mortar renders of a cistern in Pergamum (Brinker, Garbrecht 2007, 100), while fractured and possibly fired pumices and reactive flints were recently documented in the revetments of some hydraulic infrastructures and floor beddings in Corinth, dated between the Classical and the Hellenistic Ages (Siddall 2019) as well as in many Hellenistic sites in ancient Macedonia (Pachta *et al.* 2014, 847; Stefanidou, Pachta 2015). In certain occasions, volcanic pozzolans were documented also in floor bedding mortars. For example, they were detected in the preparation layers of some pebble mosaics in the Palace of Aigai and in the houses of Pella too (Papayianni, Patcha 2008; Pachta, Stefanidou 2018).

It is not clear if the Greeks were actually the first to use purposely volcanic pozzolans in mortars. By the analysis of some water tanks' revetments in the Punic Pantelleria and Carthage, dated to the 4th and 3rd centuries BC, volcanic pozzolans were observed. According to the hypotheses formulated by the scholars (Schön et al. 2012; Schön 2014, 203-212), volcanic pozzolans in the mortars of Carthage were not locally sourced but were probably imported from Pantelleria. This outcome might testimony one amongst the first trans-regional trades of volcanic pozzolans before the Roman Era (Secco *et al.* 2020, 78-79; Bonetto, Dilaria 2021).

Moreover, in the Phoenician and Punic cultures, from the Levantine area to the territories of North Africa, including Sardinia, Southern Spain and Sicily, hydraulic mortars for cisterns' waterproofing were produced by adding plant and animal ashes to lime-based binders (Lancaster 2015, 201; 2019, 35-38). In these regions, this constructive tradition was maintained also during the Roman period, as attested in Nora in Sardinia (Fig. 4) (Secco et al. 2020, Bonetto, Dilaria 2021).

Concluding with the pre-Roman evidence of pozzolanic aggregates, the unusual employ of reactive slags (litharge, iron/manganese) in lime-based mortars was documented in the cisterns' renders of the Laurion in Attica, chronologically framed to the 4th century BC (Papadimitriou, Kordatos 1995).

THE ROMAN ERA AND LATE ANTIQUITY

The spread of the Latin culture in the Mediterranean led to a broader use of mortar-based materials that, differently from the previous times, were not uniquely employed as renders. From the 3rd century BC or, more likely, during the 2nd century BC, lime-based mortars began to be used by Roman builders as a structural material for the creation of thick wall cores and foundational casts of monumental buildings. The rapid expansion of Rome in the Mediterranean, along with the deduction of new colonies, greatly influenced this change. In

Fig. 4. (left) Ash and charcoal enriched mortar in a Roman cistern from Nora (Sardinia); (right) Cross-section of a sample of an organic ash-rich sample from a cistern render of Nora (photos by Simone Dilaria).

fact, the need for realizing rapidly new cost-efficient buildings that emulate the magnificence of the full-body Greek monumental architecture represented the essential ground for the spread, from the Middle Republican Age onwards, of the *opus caementicium* (mortar-based structure), a solid, versatile, easy-to-produce and low-cost building material (Fig. 5) (Mogetta 2015; 2021).

The development of the *opus caementicium* was complemented by an extensive use of volcanic pozzolans in the mortars. Vitruvius celebrated the properties of the *pulvis puteolana*, a particular volcanic dust, quarried in the localities between Baia and Pozzuoli in Phlegraean Fields and the Vesuvius, that was recommended for the making of hydraulic concretes of maritime piers (Fig. 6) (Vitr. 2.6.1-2; 5.12.2; Plin. nat. 35.166). This material obtained a broad commercialization in the Empire from the Imperial Age onwards, being extensively used in the making of the *opus caementicium* piers of the main Roman harbours in the Mediterranean (Brandon et al. 2014; Marra et al. 2016a; Sec-

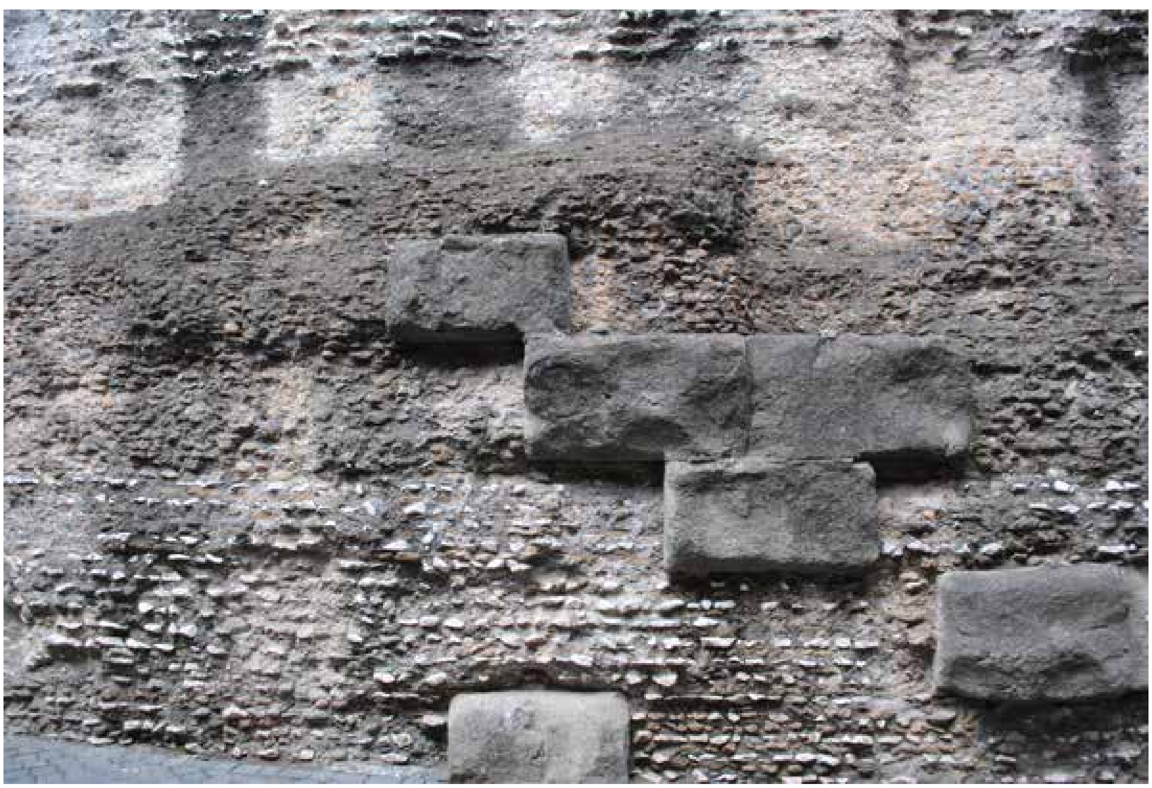

Fig. 5. *Opus caementicium* foundation of the Hadrian Mausoleum in Rome (today's Castel Sant'Angelo) in Rome (mid 2nd century AD) (Dilaria in press).

Fig. 6. Volcanic tuffaceous outcrop in Baia (Phlegraean Fields) (Dilaria in press)

co et al. 2022). The *pulvis puteolana* was also used for the construction of overground buildings, not only in the territory around the gulf of Naples (Miriello et al. 2010; De Luca et al. 2015; Paternoster et al. 2007; Sossio Fabio et al. 2018; Rispoli et al. 2019a; 2019b), but also in far regions, as recent researches are indicating for Sardinia (Nora) (Fig. 7) and Northern Italy (Aquileia) (Fig. 8). In Nora, in fact, the pyroclastic aggregates, probably imported from the gulf of Naples, were extensively used in the 2nd and 3rd c. AD in masonry construction (Ongoing research by S. Dilaria, J. Bonetto, C. Previato and M. Secco) and revetments (Dilaria, Marinello, Zara 2022; Bonetto Dilaria 2021). In Aquileia, this aggregate was detected in the *opus caementicium* vaults of the Late Antique Baths (Dilaria et al. 2022b; Dilaria in press) and in the bedding layers of the *orchestra* of the theatre (ongoing research by S. Dilaria, A.R.Ghiotto, J.Bonetto, M. Secco).

Fig. 7 - Volcanic pozzolans (mainly pumices and fragmented tuffs), probably from the Gulf of Naples, in the bedding of the parietal opus sectile of the Temple of Aesculapius in Nora (3rd century AD) (Dilaria in press).

In their treaties, Latin authors mentioned also other volcanic pozzolans having properties similar to the *pulvis puteolana*. Vitruvius described the *harenae fossiciae* as red (*rubra*), black (*nigra*) and light brown (*cana*) quarry sands (Vitr. 2.4.1; cited by Plin. nat. 36.175; Fav. 8.1; Pall. 1.10.1) to be used to obtain particularly tough and durable mortars (Lancaster 2021). The *harenae fossiciae* were identified by the scholars in the cinerites of the Middle Pleistocene eruptions of the Colli Albani, in the *facies* of the so-called Pozzolane Rosse, Pozzolane Nere and Pozzolanelle (Jackson *et al.* 2007; 2010; Lancaster 2019), although some scholars do not completely agree on this point (D'Ambrosio *et al.* 2015).

Vitruvius finally mentions the *carbunculus*, probably corresponding to the blackish scoria-

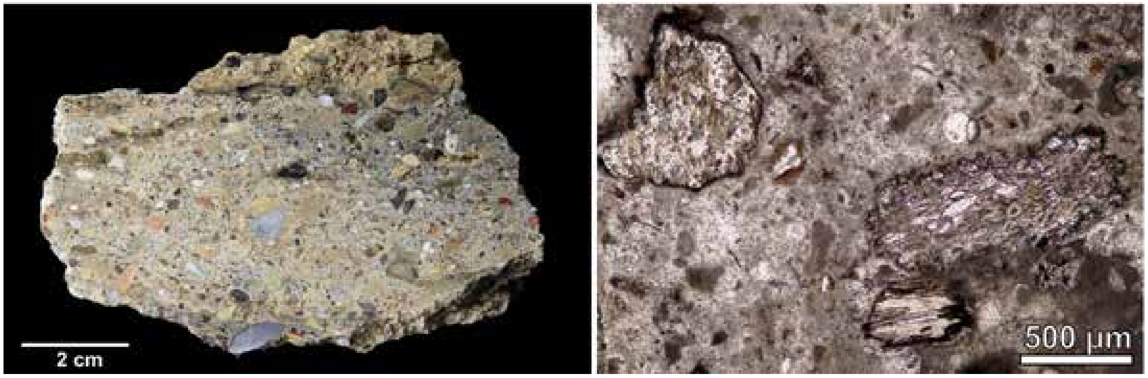

Fig. 8. (left) Cross-section of a sample of volcanic pozzolan enriched mortar from the bedding of the orchestra of the Roman theatre of Aquileia (beginning of 1st century AD); (right) micrograph of transmitted light of pumice aggregates in the mortars of the floor bedding of the orchestra of the Roman theatre of Aquileia (parallel nicols) (Dilaria in press).

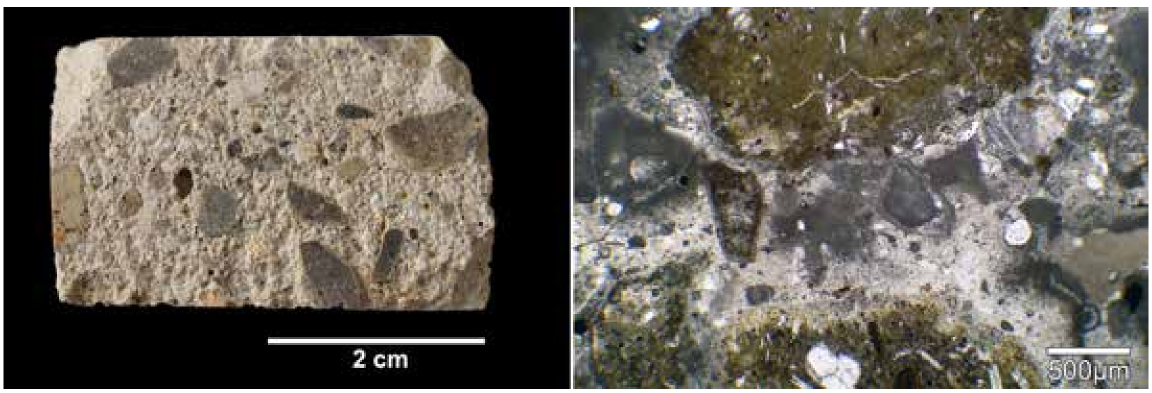

Fig. 9. (left) Cross-section of a mortar sample enriched with volcanic pozzolan from the Euganean hills from the masonry structures of the theatre of Montegrotto; (right) micrograph of transmitted light (parallel nicols) of reacted volcanic aggregates (breccias) (photos by Simone Dilaria).

ceous deposits of the Sabatini Mountains in ancient Etruria (nowadays corresponding to the Northern Latium). The *harenae fossiciae* were extensively exploited since the Middle-Late Republican age to produce mortar-based materials in Rome (Jackson et al. 2011; Marra et al. 2016b; Seymour et al 2021; Schmölder-Veit et al. 2016; Boccalon et al. 2019) and in the sites around the city (Murgatroyd 2016; Botticelli et al. 2021; Boularand, Turci, Bromblet 2022), but, unlike the *pulvis puteolana*, apparently did not obtained a broad distribution out of the territories where they were locally available.

However, the trading of volcanic pozzolans did not involve only the Campanian ones. Recent analyses of sand ballasts from the Wreck B of Pisa (1st century CE) have detected pyroclastic pozzolans whose geochemical fingerprint is compatible with the products of *Vulsinii* volcanic outcrop (northern Latium) (Marra, D'Ambrosio 2013). Pozzolans from these outcrops were detected in the mortars of Vulci and *Vulsinii* in Etruria (D'Ambrosio et al. 2015).

In the provinces of the Empire, in certain occasions, local builders and engineers experimented volcanic materials in mortars having properties resembling the traditional central-Italian pozzolans. These "alternative" pozzolans were locally sourced and their distribution probably remained mainly intra-regional. These include the so-called Rhineland Trass, a particular tuff attested in the mortars of some Roman buildings in Cologne (ancient *Colonia Ulpia Traiana*) (Lamprecht 1984, 46-49; Wang, Althaus 1994); tuffs from Turkish volcanic outcrops were exploited for the production of mortars employed in some centres of Asia Minor, such as Sagalassos (Callebaut et al. 2000; Degryse, Elsen, Waelkens 2002), Nysa and

Fig. 10. (right) Cocciopesto wall joints (foundation) from the Palace of Galerius (left) in Thessaloniki (late 3rd century-early 4th century AD) (Dilaria in press).

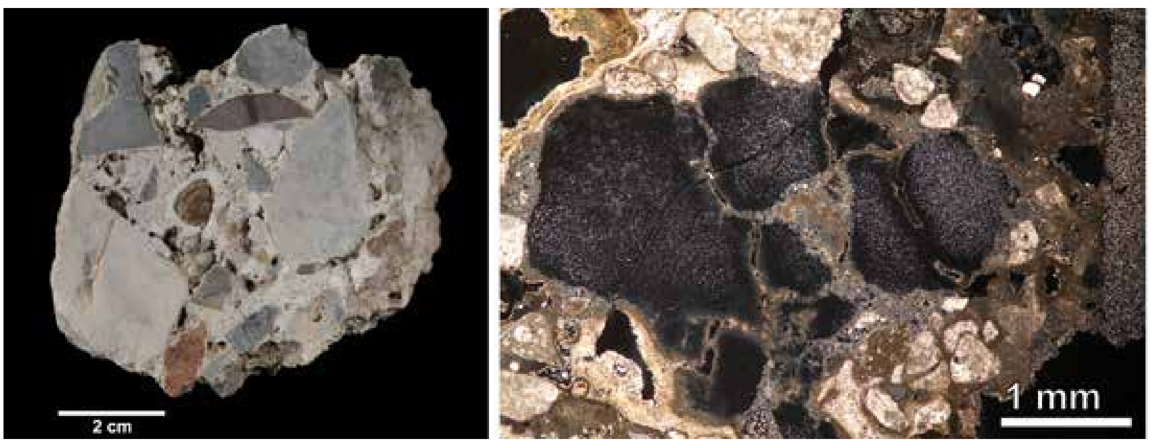

Fig. 11. (left) Cross-section of a sample from the concrete foundation of the Republican Walls of Aquileia (beginning of 2nd century BC); (right) micrograph of transmitted light optical microscopy (crossed nicol) of a lump of the natural hydraulic lime from the concrete sample (Dilaria in press).

Aigai (Uğurlu Sağın, Engin Duran, Böke 2021) and Pergamum (Özkaya, Böke 2009); the use of obsidian and perlites in the structural mortars of the Roman theatre of Nora in Sardinia (Columbu, Garau, Lugliè 2019), with a geochemical characteristics compatible with Monte Arci (southwestern Sardinia), represents another isolated case of exploitation of local alternative pozzolans. Similarly, some latitic and rhyolitic breccias from the Euganean Hills (northeastern Italy), on which the research is still in progress under the coordination of Michele Secco, were used in the *opus caementicium* foundations of the theater and amphitheater of the Roman Patavium as well as in several sites in the hinterland (preliminary report in Bonetto *et al*. 2021, 54-59). For example, the presence of Euganean breccias was recently found in the mortars of some Roman public buildings of Montegrotto (PD), the ancient *Fons Aponi (*Fig. 9*)*. Ongoing research are targeted to the compositional characterization and definition of the exact provenance of these local volcanic pozzolans. Other evidence of "alterative" volcanic pozzolans are unfortunately based only on archaeological observations (Bonetto *et al*. 2019, 464-465; Lancaster 2015, 26-27 with additional data reported in WebCat. 2-C contained online at www.cambridge.org/vaulting).

In Roman times the manufacturing of hydraulic and water-resistant mortars did not involve volcanic pozzolans only. In this period, the use of *cocciopesto* mortars, produced by mixing finely ground *terracotta* fragments with lime, continued and massified. These mortars were used not only as hydraulic revetments but also for wall joints, in particular from Late antiquity onwards (Fig. 10). Reactive diatoms as pozzolanic material were also mentioned by some scholars (Pecchioni, Fratini, Cantisani 2014, 36). In Magdalengsberg (ancient Noricum), reacted iron slags were documented in lime-based Celtic-Roman (Böttger, Thiedig, Knöfel 2002).

Finally, prototypes of "natural hydraulic lime" in Roman times obtained by the calcination of impure limestones, i.e. marls, (Bonazza *et al.* 2013 ; Lezzerini *et al.* 2017; La Russa *et al.* 2015; Izzo *et al.* 2018; Nikolić, Rogić 2018;) or cherty limestones and breccias, containing microcrystalline silica (Cantisani *et al.* 2002; Drdácký *et al.* 2013), were documented in several circumstances, as recently observed in the concrete foundations of the Republican Walls of Aquileia (Fig. 11) (Dilaria et al. 2019; Dilaria in press; Bonetto in press). Besides the traditional pozzolanic mortars (i.e. cocciopesto, volcanic ash and organic ash rich mortars), it is unclear whether the production of natural hydraulic limes was pursued by ancient crafts or rather depended on the type of limestone suitable for calcination available in the proximity of the site of employment. In fact, the cases of natural hydraulic lime in ancient times always come from sites where impure limestones outcrops were present nearby, thus making the hypothesis of some unawareness about the properties of the final product rather likely.

CONCLUSIONS

This contribution clearly demonstrates the pervasiveness of the application of different types of binding mixtures in diversified human cultures since the beginning of the technological development of mankind. Such a class of functional materials represents a key for the interpretation and parametrization of social advancement within societies, in relation to the optimization of sheltering techniques in primitive human groups, and to complex architectural development in more advanced cultures. The study of mortar recipes over times and in different geographical contexts, often reciprocally disconnected, indicates a thorough knowledge of local resources and a significant awareness of material properties and mutual interaction among them, even though they were simple empirical technological processes. In this perspective, the scientific study of such anthropogenic materials constitutes a promising research field, not only for the archaeometric characterisation of archaeological and historical tangible and intangible contexts, but also for the development of novel building materials, less environmentally impacting and more respectful of the anthroposphere.

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REZIME

RECEPTURE MALTERA KROZ VEKOVE. KRATAK PREGLED PODATAKA OD PRAISTORIJE DO KASNE ANTIKE

KLJUČNE REČI: ANTIČKI MALTERI, KREČ, GLINA, GIPS, PUCOLANSKI AGREGATI, OPUS CAEMENTICIUM, COCCIOPESTO, PRIRODNI HIDRAULIČNI KREČ.

Maltere, kompozitne materijale koji se upotrebljavaju za spajanje zidnih elemenata, za zaptivanje i davanje vodootpornosti arhitektonskim površinama, ljudska populacija je koristila u svrhu izrade skloništa od početka tehnološke evolucije čovečanstva, stvarajući prve posvedočene proizvode pirotehnologije od neolita. Vremenom su izbor sirovina i optimizacija receptura i postupaka mešanja omogućili da se diverzifikuju i optimizuju njihova svojstva, kako bi se odgovorilo na najsofisticiranije zahteve ljudskih kultura. U ovom prilogu je dat kratak pregled evolucije tehnologije maltera od paleolita do kasnog rimskog doba, sa posebnim fokusom na odabiru funkcionalnih jedinjenja kako bi se neke specifične osobine ovih vezivnih kompozita podstakle u različitim ljudskim društvima. Detaljnije će se baviti prvim primenama jednostavnih veziva na bazi gline u praistorijskim i protoistorijskim društvima, uz rane eksperimente sa pirotehnologijom maltera na bazi kreča. Zatim će biti opisana evolucija tehnologija veziva u grčkom i helenističkom dobu, sa posebnim osvrtom na razvoj prvih pucolanskih smeša, nakon čega će uslediti detaljan opis izuzetnog tehnološkog razvoja hidrauličnih i pucolanskih strukturalnih kompozita u rimskom društvu.

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