Rendiconti
Accademia Nazionale delle Scienze detta dei XL
Memorie di Scienze Fisiche e Naturali
135° (2017), Vol. XLI, Parte II, Tomo I, pp. 7-18

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Let the walls speak. A brief history of binders in architecture**

Abstract – The use of binders and ligands in architecture played an important role in the development of human societies. Everybody is aware of the importance of Portland cement and concrete in modern construction industry, as a matter of fact there would be no urban or long-range modern infrastructure without the systematic technological use of high performance binders. However the huge volumes of cement and concrete produced at present, making them the materials that are produced the most in terms of weight, are raising serious concerns in terms of emitted carbon dioxide and energy requirements. Active research is ongoing to optimize low-energy and low-carbon footprint alternative materials, such as geopolymers, alkali activated materials, and calcined clays.

In the past, it is argued that the success of the Roman Empire was solidly built on the clever and widespread use of hydraulic mortars. The long history and development of binder technology is reviewed, with an eye on chronological and geographical patterns.

1. *Introduction*

Shelter and repair represent a fundamental need for humans. The use of natural materials for building shelter is not exclusive to humans: termites build resistant mounds from clays, birds build nests, beavers build dams, beehives are wonder structures from the architectural point of view. According to Mike Ashby (Ashby 2013, p. 11) «the difference lies in the *competence* demonstrated by man in his

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** Prolusione tenutasi durante l'inaugurazione del 235° Anno Accademico, Roma 10 maggio 2017, Biblioteca dell'Accademia Nazionale delle Scienze detta dei XL, Scuderie Vecchie di Villa Torlonia.

extraordinary ability to expand and adapt that competence and development». In general terms mankind is the only species that developed well beyond the adaptation to the environment, and now it is extensively adapting the environment to its needs. Although still debated, the term *Anthropocene* (Crutzen and Stoermer 2000, Crutzen 2002, Zalasiewicz *et al*. 2011) has been proposed to designate the recent times in which human activities expanded to such a scale to be detectable in the geological record. The conscious production of binders and their use in architecture is one technical development that allowed man to build more flexible and durable constructions fostering survival and adaptation to the environment, but in the end resulting in large modifications of the natural landscape. Large stone blocks have been employed since prehistory to mark sacred and burial places, still visible in megalithic stone circles and dolmens (such as Stonehenge or Carnac; Burl 2005), and stone buildings quickly became the preferred way to rise monumental constructions, fortified walls, and temples. Evocative examples are the Nuragic towers and villages in Sardinia mostly build out of basaltic blocks, the defense walls of the Hittite capital Hattusa made of carefully shaped limestone units (Fig. 1), the amazing polygonal walls of Inca cities made of granites and andesites, or the great Egyptian pyramids made of carved limestones, despite insistent claims that the blocks were cast using man-made geopolymeric materials. Freestone and Middleton (2007) and Jana (2007) present an extensive account of the decades-old Great Pyramid debate. The use of

Fig. 1. Megalithic fortified walls of Hattusa, the ancient capital of the Hittite empire. They are composed of shaped limestone blocks with no use of binder.

large stone blocks has continued through history for defense and symbolic large buildings, providing robustness and stability despite the requirements for the local availability of suitable stone resources and extensive manpower. On the other hand the architecture of small and vernacular buildings has quickly turned into the use of masonry (that is structures made of small units connected by binder materials), which provides for very flexible, cheap, and durable structures. In fact masonry offers a number of advantages, such as the easy adaptation of the building to landscape or other existing architecture, the practical sub-division of the living space, and functional improvements in terms of fire, thermal, and water protections (Hendry 1998, Artioli and Secco 2016). Masonry architecture is therefore ubiquitously found in residential buildings, showing the availability of local materials and the traditional practices and skills of building.

As a general frame, we will here define artificially produced binders those products that are derived from natural materials through the use of high temperature reactions, including decomposion, sintering, and recrystallization. The reactive powder (binder) obtained through fire is then ground and mixed with water in order to obtain a plastic material that hardens through carbonation (by absorption of CO_2 from the atmosphere; they are called *aerial binders*) or the formation of hydrous products through dissolution and precipitation reactions (*hydraulic binders*). In the course of prehistory and history the main types of binders used in architecture were: gypsum and lime based mortars, pozzolanic cements, natural hydraulic limes, and Portland clinker cements (see Hobbs and Siddal 2011, Blezard 2003, or Artioli 2010, pp. 242-265, for reviews of the technology and chemistry of ancient binders).

2. *A brief history of binders*

Clays are the most widespread natural material with low-level binding properties. Because of the layered nature of the structural units (Brown and Brindley 1980, Bailey 1988) and the strong surface interactions between particles, clay minerals can be transformed into very plastic materials when mixed with water. As such they have been used since pre-history to mold figurines, pottery, ceramics, and clay-based structural products, such as bricks, tiles, or pipes (Brownell 1976, Artioli 2010, pp. 209-242). Pottery and structural clay products were transformed into hard and durable products by the technological use of fire (Rehder 2000). It is surprising that the use of pyrotechnology to produce quicklime (CaO) through the high temperature decarbonation of calcite predates the local use and firing of ceramics. There is in fact ample evidence of the use of lime-based mortars for floor and wall plastering in the Pre-Pottery Neolithic B (PPNB) period in the Near East (8700-7000 BC: Gourdin and Kingery 1975, Kingery *et al.* 1988), following the early use of clays for the production of sun dried bricks (adobe) and for plastering. An excellent example is the original and repeated use of earth structures in the Neolithic early cities, such as Çatalhöyük, in Anatolia (Hodder 2006, Arkun 2003). Earthen structures require continuous maintenance in order to survive. Typically there is annual replastering of the surfaces using a clay-water-straw mixture. Accordingly, in many of the clay-based plasters in Çatalhöyük and also in the lime-plastered floors of the PPNB sites, such as Yiftahel and Jericho in Palestine (Malinowski and Garfinkel 1991, Kingery *et al.* 1988), there is evidence of periodical, maybe yearly, cleaning and renewal of the surfaces. Despite this early start of pyrotechnologically produced lime mortars, there seems to be a puzzling discontinuity in the use of lime technology between the 6th and the 3rd millennium BC. After the widespread use of lime in the Neolithic Near East there are isolated 6th millennium BC reports in the Balkanic site of Lepenski Vir (Radovanović 2000), at Makri, Thrace (Karkanas and Efstratiou 2009) and at the Drakaina Cave, Kefallonia, Ionian Islands (Karkanas and Stratouli 2008). After this scarce evidence, there is in fact no archaeological report of the architectural use of lime mortars until the 3rd millenium BC in the Minoan world, where again lime-based mortars re-appear to be used for wall plastering, mainly for decorative purposes and painting support (Cameron *et al.* 1977). The Aegean area then witnesses a rapid development of the mortar materials, involving the incorporation of pozzolanic sand (Santorini's Earth) and crushed pottery. The replacement of inert sand with reactive materials induces pozzolanic reactions in the binder, which is able to harden under water and yields a much stronger and less porous paste. In Crete, the so-called hydraulic mortar (Elsen *et al.* 2012) starts to be used regularly for waterproofing cisterns and waterways because of the superior properties, although it should be noted that there is no evidence of mortar use for structural purposes, that is among stones in ashlar or among bricks in masonry.

Lime mortars and hydraulic mortars continued to be used in the Middle East and the Mediterranean world through the Bronze Age and the first Iron Age. Phoenicians, Micenaeans and Etruscans made ample use of wall plastering for decorations and cistern waterproofing, but again there is no evidence of the structural use of the binders for strengthening architecture. Only clay is found among masonry units, and the same is true for the early architectural developments in Rome. The Servian walls in Rome (6th century BC) are still made with binder-less ashlar units (*opus quadratum*). The Egyptian world seems to be essentially confined to the use of gypsum plaster. In Asia, there is some limited evidence of the use of lime before the second Iron Age, for example in the proto-Harappan period in the Indus civilization, or in the West Zhou period in China (Carran 2012). In Mesoamerica there is an early use of lime in the Mayan world around 1100-600 BC (Cuello, Belize; Nakbè, Guatemala).

The real technological breakthrough arrived towards the beginning of the 2nd century BC in the Roman world (late Republican Age): the hydraulic mortar technology is optimized to large structures and routinely used for important public and military buildings. Marcus Porcius Cato (*De agri cultura liber*, 160 BC) describes for the first time the systematic use of lime-mortar as a binder (Greco 2011). It is not yet clear whether the development of the Roman cement (*opus caementicium*, Lechtmann and

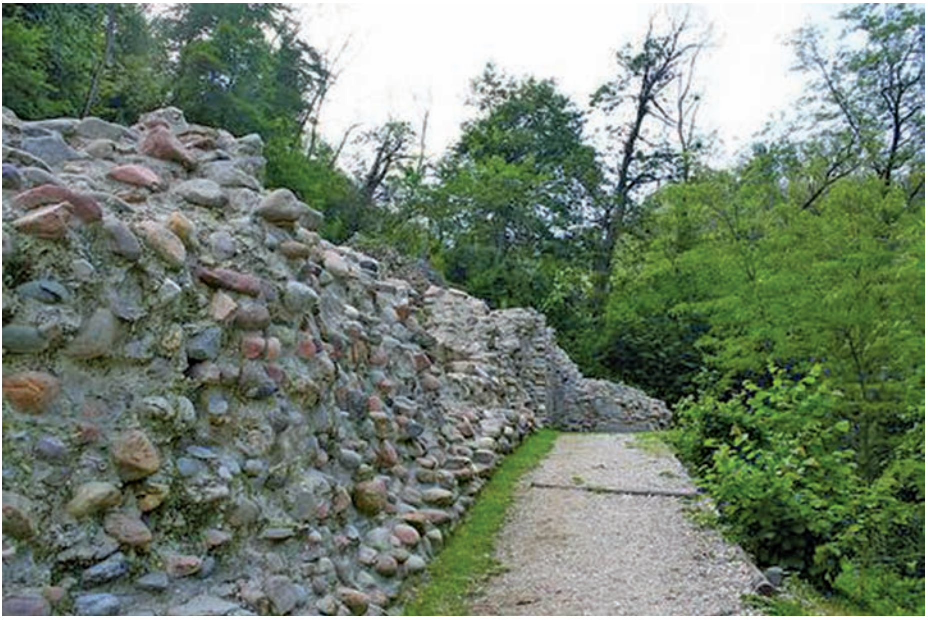

Fig. 2. Typical infilling of the large wall structure of a Roman fort by *opus caementicium*, containing fragments of rocks and broken ceramics embedded in the lime matrix.

Hobbs 1987, Lamprecht 1996) developed in Rome or somewhere else in the Roman world. The hard fact is that within a few decades, in a period just before the third Punic war, the hydraulic mortar recipe previously used in the Hellenistic world for cisterns becomes standardized and massively used in large constructions. The recipes for mortars later encoded by Vitruvius (Marcus Vitruvius Pollio, *De architectura*, 80-15 BC) and Pliny (Gaius Plinius Secundus, *Naturalis Historia*, 23-79 AD) were actually developed in the first half of the 2nd century BC. An optimized formulation encompassing carefully slaked lime, sand, and ground pottery is used for mortars, whereas for the infilling of the walls of large structures (Fig. 2) pieces of rocks and broken ceramics were used. Very often recycled debris from demolished architecture was used (Fig. 3).

The crushed ceramics was quickly replaced with pozzolanic sand and fragments, mostly derived from the «Pozzolane rosse» formation (Marra *et al.* 2016), although the systematic use of crushed pottery was maintained for waterproofing material. The ample use of volcanic and pyroclastic material, imparting hydraulicity to the material and making it the material of excellence for the building of sea structures and harbors (Brandon *et al.* 2014) is possibly due to the availability of suitable material in the volcanic province around Rome, combined to the need of large volumes of hydraulic components impossible to obtain from pottery alone (Mogetta 2015). Whatever the driving force for this critical technological development, the Roman conquest of the ancient world is intimately linked with the development of solid

Fig. 3. Large fragment of a mosaic floor (upside down) embedded in a wall of a Pompeian building as recycled material. The stratigraphy of the preparatory layers of the mosaic, containing abundant ceramics fragments, is on display.

architectural infrastructures. The Roman buildings and aqueducts have certainly shown their solidity, utility and beauty, according to Vitruvius' suggestion «*Haec autem ita fieri debent, ut habeatur ratio firmitatis, utilitatis, venustatis*».

After the long period during which standardized and successful binder recipes were employed throughout the Roman Empire, the political and cultural decline taking place in late antiquity is mapped in the technological character of the architectural binders. In many places, even when there is evidence of continuity in population levels, a certain degree of simplification and approximation of the ancient «Vitruvian» binder recipes is observed (Secco *et al.* 2017). More data are required to understand the social and economic context of this technological decline. The relaxed centralized control during the construction of public buildings maybe one of the causes, together with many of the deficiencies linked to human nature, such as bad planning, corruption, fraudulent behavior, or cost saving (see the interesting account of Oleson 2011).

During the Middle Age a widespread use of medium to poor-quality lime mortars is observed, although for important buildings contractors do systematically improve the hydraulic character of the binder with the addition of reactive silica sources (Franzini *et al.* 1999, Franzini *et al.* 2000). Substantial compositional differences are observed in the binders in different places, denoting the use of local materials and the lack of standardized formulations (Blezard 2003). The use of crushed ceramics in the lime mortar continued with different traditions after the collapse of the Roman Empire, it is called *Cocciopesto* in Italy, *Horasan* in Turkey, *Homra* in the Middle East, *Surkhi* in India.

Occasionally, the firing of impure limestones containing clays or marls induces the production of mortars containing reactive silicate phases. These materials, still produced today especially for conservation purposes, are called *natural hydraulic limes* (NHL). The *Karlův most* (Charles Bridge, 14th-15th centruy AD) in Prague is one example of the early use of NHL in a large durable construction (Frankeová 2012). However, the systematic experimentation of firing limestone-clay mixtures or impure limestones and the scientific investigation of the products obtained started in the mid 18th century AD: the attempts of Smeaton in the UK, Bergmann in Sweden, Saussure in Switzerland, Descotils in France and many others rapidly lead to the developments of modern hydraulic binders (Blezard 2003, Elsen 2010).

Louis Vicat first introduced the hydraulicity index (HI) to classify and quantify the reactivity of the silica and alumina content of the binder (Vicat 1818). The possibility to perform analytical measurements and assess the reactivity character of the material opened the way to the large scale production of standard binders with controlled properties.

3. *Modern investigation of ancient binders*

The modern multidisciplinary investigation of ancient mortars is based on a variety of techniques, providing for the detailed understanding of (a) the nature of the components of the binder composite; (b) the technology employed for the production; (c) the engineering and conservation history of the material.

Several analytical methods are routinely employed to investigate ancient binders: optical microscopy, scanning electron microscopy with elemental imaging, X-ray diffraction, thermal analysis, Infra-red spectroscopy, nuclear magnetic resonance, and many others (Riccardi *et al.* 1998, Moropoulou *et al.* 2000, Middendorf *et al.* 2005, Elsen 2006, Pecchioni *et al.* 2014). Since binders are very complex composite materials, each technique alone is hardly sufficient to reach a satisfactory comprehension of their nature and history. Several complementary techniques are therefore needed to obtain an adequate knowledge of the material. Figs 4 and 5 show the reaction rims formed by pozzolanic reactions around a pottery fragment and a cluster of volcanic glass embedded in ancient mortars.

Here it is important to remark that the detailed mineralogical, petrographic, and physico-chemical characterization of the mortar is not only providing important archaeological, historical and technological insights. In the case of rather heterogeneous materials such as mortars, the information obtained is also fundamental to appropriately select the samples for further analysis.

The issue is critical when preparing the mortar sample for dating by modern

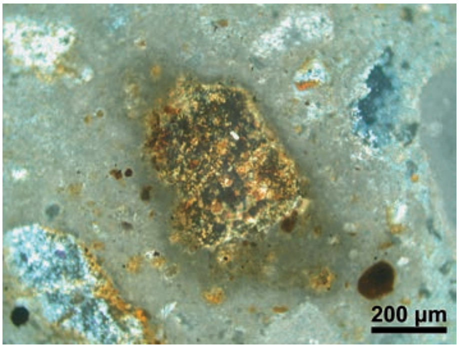

Fig. 4. Reaction rim formed by pozzolanic reaction around a crushed pottery fragment. Optical microscopy, plane polarized light.

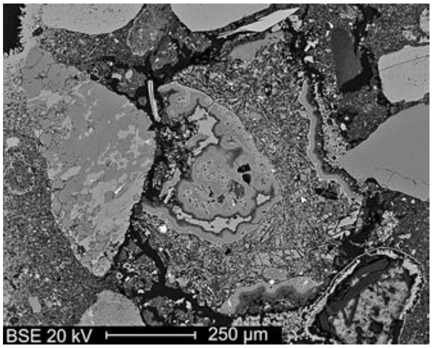

Fig. 5. Reaction rim formed by pozzolanic reaction around a fragment of volcanic glass. Electron microscopy, backscattered electrons image.

radiocarbon techniques (Heinemeier *et al.* 1997, Van Strydonck 2016). To date, sequential dissolution techniques are largely employed to separate the different dateable fractions. However, it has been shown that the different components of ancient mortars cannot be safely separated by acid dissolution reactions. Depending on the nature of the mortar and its history, the components of the material may include geological carbonates, incompletely burned lime, carbonated lime binder, products of the hydraulic reactions, lime lumps, and secondary calcite from subsequent reactions. Alternative preparation techniques exploiting the full experimental information from each separated binder fraction have been proposed (Addis *et al.* 2016, Hajdas *et al.* 2017). Each component of the mortar is adequately identified by microscopy, diffraction, optical luminescence, and C,O stable isotope ratios, so that the separated fractions are processed accordingly. Fig. 6 shows the detection of fine particles of geological limestone mixed with the carbonate of the binder.

The possibility of radiocarbon dating ancient mortars represents a powerful aid for the interpretation of archaeological and architectural structures, and in turn an important support for conservation strategies.

4. *The future of binders: binders for the future*

The scientific study of ancient binders greatly helps to understand the technological evolution behind the impressive architectural achievements of the past. Further, the quantification and the modelling of the kinetics governing the hydraulic reactions is mandatory to develop novel binders bound to replace modern Portland clinker (Shi *et al.* 2011, Gartner and Hirao 2015, Liew *et al.* 2017). The rationale is that modern society cannot efficiently build infrastructures without clinker-based cement and concrete. However, the environmental impact and carbon footprint of

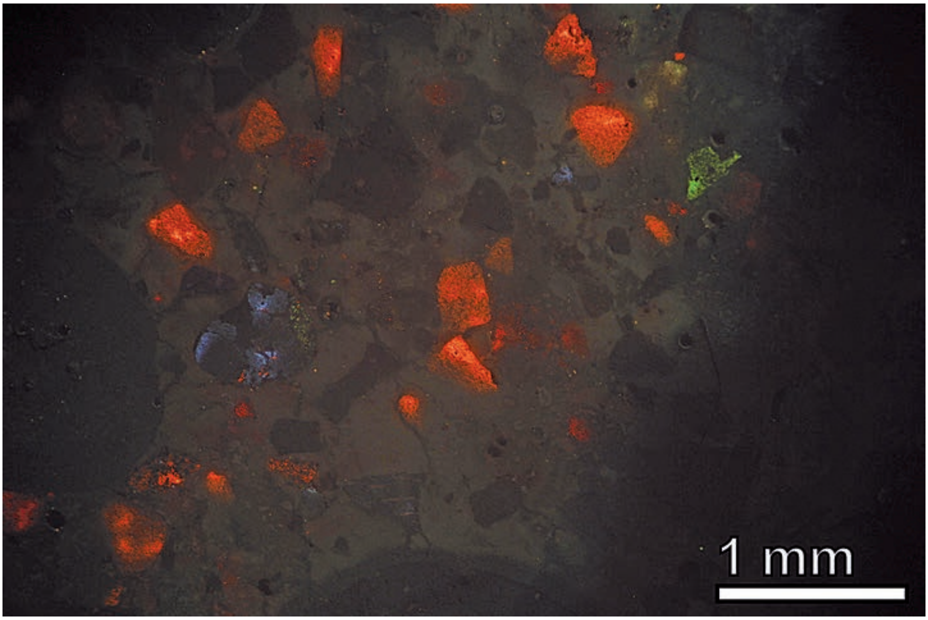

Fig. 6. Fine particles of geological limestone mixed with the carbonate of the binder. These particles are perturbing the signal for the radiocarbon dating of the mortar and must be eliminated before measurement.

commonly used binders are not compatible with the prospected growth of developing countries and the needs of global economy (Gartner 2004, Barcelo *et al.* 2014). Hence, substantial research efforts are under way to find viable substitutions to clinker-based binders, which are universally used at present.

Three solutions are being pursued: binders based on alkali activated materials (Shi *et al.* 2006), binders based on geopolymers (Provis and Van Deventer 2009) and binders based on calcined clays (Scrivener and Favier, 2015).

Each one of these alternative systems needs development and optimization in terms of performance, sustainability and impact. Besides active research on model industrial materials and binder systems, the investigation of ancient mortars yields fundamental information on the type and structure of the reaction products formed in presence of hydraulic reactions, and their long-term stability and behavior. It is interesting that ancient materials may provide some of the knowledge needed for the development and optimization of the binders of the future.

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Acknowledgements

I take the opportunity of this inaugural address to the Accademia to acknowledge the hard work and enthusiasm of the young collaborators working on ancient and modern cements at the CIRCe Centre: Anna Addis, Enrico Garbin, Giulia Ricci, Luca Valentini, Marco Favero, Maria Chiara Dalconi, Michele Secco, Simone Dilaria, Silvia Contessi, Yotam Asscher.