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Ancient mortars and their binder

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ABSTRACT. — Numerous works which have characterised binders in historical buildings (Fratini and Giovannini, 1990; Chiari *et al.*, 1992; 1996a; 1996b; Collepardi, 1993; Fratini *et al.*, 1994; Mannoni and Giannichedda, 1996; Franzini *et al.*, 1999; 2000a; 2000b) reveal that the production of such materials, and of mortars and plasters, was not a simple process. There is increasing evidence that blends of natural materials were studied in ancient building sites and that different technical solutions were adopted to resolve specific problems.

The reported case studies represent a variety of construction contexts and periods, and different issues were addressed in each study.

Each construction site found a unique solution which was obtained by «correcting» the parameters of locally available geomaterials and involved the study of complex analytical strategies suited to each specific case.

The study of hydraulic mortars used to construct the Tower of Pisa is an example of how scientific investigation can help reconstruct ancient production recipes that were never recorded in any manual of the period.

RIASSUNTO. — Il concetto che la produzione di leganti, e di conseguenza la realizzazione di malte ed intonaci, sia stata un'operazione semplice è smentita dai numerosi lavori che hanno trattato la caratterizzazione di tali materiali in edifici storici (Fratini & Giovannini, 1990; Chiari *et al.*, 1992; 1996; Collepardi, 1993; Fratini *et al.*, 1994; Mannoni e Giannichedda, 1996; Franzini *et al.*, 1999; 2000). Sempre più spesso ci si accorge che, negli antichi cantieri, erano studiate e messe in opera miscele di materiali naturali e soluzioni tecniche di volta in volta più adatte al problema specifico.

I casi di studio riportati in questo lavoro appartengono a contesti costruttivi ed epoche molto differenti tra loro. Differenti sono state pure le problematiche di studio affrontate per ciascun caso.

La singolarità di ogni soluzione «di cantiere», risolta attraverso la «correzione» dei parametri di qualità dei geomateriali a disposizione nelle vicinanze del cantiere costruttivo ha comportato lo studio di strategie analitiche complesse, calibrate su ciascun caso specifico.

Lo studio delle malte idrauliche del cantiere costruttivo della Torre di Pisa, rappresenta un esempio di come sia possibile, attraverso le indagini scientifiche, decifrare un'antica ricetta di produzione, mai scritta in alcun manuale dell'epoca.

KEY WORDS: *Ancient mortars, binders, historical buildings, analytical strategy, mineralogical and petrographic features.*

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INTRODUCTION

Mortars are a cultural product whose characteristics and properties depend on raw materials and technological know-how. They are important in conferring resistance, protection and elegance to buildings.

The study of ancient mortars is therefore of twofold interest: a) mortars provide information on raw materials and technologies available at the time of production; b) knowledge of the main physical properties of a mortar and its state of conservation is a prerequisite for the process of restoration and conservation. Earth Science methodologies can be used to measure the main characteristics and properties of ancient mortars and their fractions (binder and aggregate). Since it is not always possible to measure directly all properties of binders and aggregate fractions, some properties are obtained through data processing.

This work reports recent findings in the study of ancient mortars and highlights the contribution of Italian research in this sector by illustrating some case studies.

ANALYTICAL STRATEGIES FOR THE STUDY OF ANCIENT MORTARS

Mortars are usually inhomogeneous because they were produced manually. As a result, in order to obtain significant scientific results, all properties must be measured on the same sample. This is a strong limitation because it is difficult to obtain samples of adequate size. Sample preparation and the analytical sequence must therefore be carefully planned. The following analytical strategy may be adopted:

a) in-situ observation and description of mortars, and possibly measures of mechanical resistance (penetrometers, sclerometers). Collected data are essentially descriptive: colour, homogeneity, application, presence of lumps, pores, extraneous material, etc. Measurements of mechanical resistance provide an indication of the original quality of the mortar and of its subsequent deterioration.

b) collection of the largest possible samples (core samples or fragments). Sample collection will be guided by the previous observations and by objective conditions at sample sites. In many cases stereomicroscopy may be used to identify mortars from the same building but from different periods of construction and to manually separate lumps, when present, in sufficient quantities for thermogravimetric, X-ray diffractometry and X-ray fluorescence analyses.

c1) collected samples cannot be used to prepare specimens. The mass, volume (using a He pycnometer or Hg buoyancy method, see Franzini and Lezzerini, 2003) of the dry sample are measured, along with the mass and hydrostatic force of the water-saturated sample.

c2) it is possible to prepare one or more standard specimens (cylinders or parallelepipeds) whose dimensions are determined by the available quantity of sample. Besides the measurements listed in c1, the capillary water absorption coefficient, the water imbibition capacity, load resistance and, possibly, the permeability to water vapour and liquid water are also measured.

d) preparation of polished, uncovered thin sections from residual fragments after completion of step c). Modal analysis of the section, compositional and textural characterisation of the binder, shape and dimension of aggregate granules.

e) metal-coating of thin sections for SEM/EDS analysis. Determination of the chemical composition and compositional inhomogeneity of the binder, of reactions between the binder and aggregate granules, and chemical analysis of aggregate granules.

f) preparation of powders from residual fragments after completing step d). X-ray fluorescence analysis, calcimetry, loss on ignition, thermogravimetric analysis, X-ray powder diffraction, water and He pycnometric measurements.

g) dissolution of residual fragments in HCl after completion of the previous processing steps. Weight fractions and granulometric

analysis through sieving of the non-carbonate aggregate.

When it is impossible to obtain the composition of binders through SEM/EDS analysis (step e), fragments of mortar are disaggregated using ultrasound or other delicate methods. The < 63 μm fraction, assumed to be representative of the binder, is recovered through sieving and then analysed.

h) there are two possible materials on which ways to complete archaeometric dating of mortars: carbon fragments and carbonate binders. When the former are fairly abundant in mortar, they derive from pit firing of limestone alternated with fuel; therefore, the organic synthesis of the piece of wood is dated, not its use nor the use of lime. Given that small pieces of fresh wood were preferred for economic and material culture reasons (Vecchiattini, 1998), chronological differences are no greater than the normal probabilistic oscillations linked to ^{14}C dating (Gallo, 1998).

As for the dating of binders, to avoid contamination by fragments of carbonate rocks in the inert material, lumps of pure binder are preferred when present. The only drawback to this method is that at some point in time the sample may have come into contact with rainwater rich in CO_2, with consequent bicarbonation and partial recarbonation (Gallo, 1998).

In any case, variations tend to overestimate ages while carbons tend to underestimate ages; by using the two methods on the same mortar it is possible to closely determine the actual age. (Fieni, 2002).

Data collected according to the outlined scheme allow an adequate overall description of the mortar: texture, chemical and mineralogical composition, percent volume of aggregate and binder, absolute and apparent density, total porosity, open porosity, saturation index and, when it is possible to prepare specimens of predetermined shape, capillary water absorption coefficient, water imbibition capacity and compressive strength. Although the latter data are nearly always derived from specimens of «non-standard» dimensions, they are still significant.

It is impossible to directly obtain equally detailed knowledge of binder and aggregate characteristics; however, much information can be gained from processing of available data. The weight fractions of aggregate and binder in the mortar are determined first:

$$X_a = V_a \bullet G_a / \gamma_m \; X_b = 1 - X_a$$

where X is the weight fraction, V the volume fraction, G the absolute density, γ the apparent density and the subscripts a, b and m indicate respectively the aggregate, binder and mortar. G_a is determined through the modal analysis of the aggregate, whereas γ_m is measured directly.

The chemical composition of a mortar is described by the following system of equations:

$$| \; (C_i)_m = X_a \bullet (C_i)_a + X_b \bullet (C_i)_b \; | \;\; i = 1, n \quad (1)$$

where C_i is the weight percent of the i^{th} chemical component. The n value is normally equal to 11 or 12, since the sum of the H_2O, CO_2, Na_2O, MgO, Al_2O_3, SiO_2, K_2O, CaO, TiO_2, MnO and Fe_2O_3 components, sometimes with the addition of SO_3, is usually very near to 100 and almost completely describes the specimen. In this system X_a, X_b and $(C_i)_m$ are known quantities. Of the $(C_i)_b$ values, only those for H_2O^+ and CO_2 are unknown; the other 9 or 10 are known except for the constant *k*. To solve the system of equations at least some $(C_i)_a$ values must be known in order to rescale measured concentrations of chemical components in the binder (Franzini *et al.*, 2000a).

CaO and CO_2 concentrations in the aggregate are generally particularly suited for this purpose, as they are rather low and can be derived from the modal analysis expressed as weight percent. For example:

$$(C_{CaO})_b = [(C_{CaO})_m - X_a \bullet (C_{CaO})_a \,] / X_b \; ;$$
$$k = (C_{CaO})_b / (C_{CaO})_{SEM} \; ; \; (C_i)_b = k \bullet (C_i)_{SEM}$$

where the «SEM» subscript indicates binder concentrations derived from SEM analysis.

Equation system (1) therefore yields the complete chemical composition of the aggregate and binder, particularly H_2O and CO_2 concentrations in the latter. When it is possible to separate the binder, these data can be compared with direct measurements.

By subtracting the $MgCO_3$, $CaSO_4 \cdot 2H_2O$ and $CaCO_3$ contents (calculated in this order) from the overall composition of the binder, one obtains the content and composition of the residue, which consists of non-crystalline phases similar to the CSH phases of cements. The absolute and apparent densities of the binder may be determined through the following equations (Franzini *et al*., 2000a):

$$\gamma_b = X_b \gamma_m G_a / (G_a - X_a \gamma_m)$$
$$G_b = X_b G_m G_a / (G_a - X_a G_m)$$

where G_a, the absolute density of the aggregate, is derived from the modal analysis of the aggregate.

A suitable strategy for collecting and processing experimental data enables the determination of the main characteristics of the aggregate of the binder and of the mortar as a whole.

CASE STUDIES ON ANCIENT MORTARS

Although they satisfy a wide spectrum of technical requirements, ancient mortars do not meet modern specifications. It should not come as a surprise that materials now considered «inferior» or unsuitable for commerce were once employed in important buildings. This is the case of magnesian limes, considered of poor quality by current industry standards; indeed, with the development of modern industrial furnaces, their production was totally abandoned and substituted by lime rich in CaO (Mannoni, 1988) or by the use of mortars with more or less abundant clay binders, mainly found in some cities of the Po Valley (Bonazzi and Fieni, 1995).

The following case studies are considered significant in terms of the reconstruction of the provenance of geomaterials, production recipes, production technologies and methods of application.

Byzantine and Norman mortars from the Castle of Santa Severina (KR): the provenance of raw materials

Through time the Castle of Santa Severina (Crotone) has been repeatedly reconstructed and enlarged. Such continuous restoration led to the superpositioning of late 18th century masonry on stonework from the Byzantine-Norman period (the earliest phase of construction).

All the mortars from the ancient walled structures (Byzantine-Norman period) may be classified as «non-magnesian, slightly hydraulic lime mortars». The binder contains scarce quantities of hydraulic components (SiO_2, Al_2O_3 and Fe_2O_3) which indicate that slightly impure, local limestones were used to produce the lime.

As in the case of materials from Santa Severina, the hydraulic characteristics of nearly all binding materials adopted in historical buildings of Calabria are correlated with the low purity of materials used to produce lime. In no case was the mortar found to contain additives to improve the hydraulicity index of the binding material. The limestones used to produce binders were quarried near the ancient building sites, even when such materials were not entirely suitable. For example, base limestones of the local evaporite succession were used to build the Abbey of Roccelletta di Borgia (Catanzaro) and the Castle of Roccella Ionica (Reggio Calabria). The salt content adversely affected the stability of limes produced from this particular carbonate material.

The same type of material was used as an aggregate in Byzantine and Norman mortars from the Castle of Santa Severina. Most samples are characterised by an aggregate of prevalently siliciclastic composition and

consisting of elements of different origin: fragments of granitic or metamorphic rocks and/or monomineralic grains generally consisting of quartz or, subordinately, of feldspar. The aggregate of some mortars contains fragments of calcarenitic rocks which were disaggregated and used in the mix as a unique constituent or associated with siliciclastic material. Comparative studies based on chemical and petrographic data have shown that the siliciclastic component was collected from the bed of the Tacina River, near the village of Rocca Bernarda (about 20 km from Santa Severina). In contrast, the calcarenitic aggregate was collected from the lower levels of the sedimentary sequence forming the terrace on which the Castle of Santa Severina was built. The local calcarenite was rarely used as an aggregate, probably due to the poor resistance of the resulting mortar with respect to that of mortar made from sand from the Tacina River. It seems that the calcarenite was used as an aggregate only when it was impossible to reach the Tacina River. Indeed, the Castle of Santa Severina was besieged on more than one occasion, sometimes for long periods of time. The identification of the Tacina River as the source area for the aggregate raises another issue. The Neto River runs near Santa Severina, and like the Tacina River it originates from the Silano Massif. Consequently, the composition of sand from the Neto River is quite similar (though not identical) to that of sand from the Tacina River. From a historical perspective it would be interesting to understand why Byzantine and Norman masons, as confirmed by other studies on subsequent periods (Angioini), preferred to use sand from the Tacina River (20 km distant) and not the very similar one from the nearer Neto River.

Although no significant differences were found among materials used to produce artefacts in the two studied periods, construction techniques were found to vary. Representative samples of the Norman and Byzantine periods were plotted in the CaO/MgO vs. TiO_2/Fe_2O_3 diagram (Fig. 1).

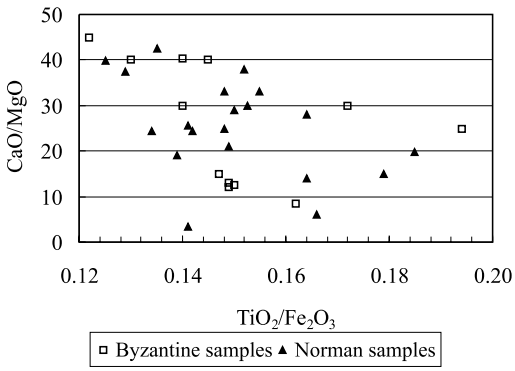

Fig. 1 – Representative samples of the Byzantine and Norman mortars (Santa Severina Castel, KR): TiO_2/Fe_2O_3 vs CaO/MgO diagram.

Norman artefacts clearly fall into three distinct groups: about 60% of samples lie in the central portion of the diagram, three samples have a low TiO_2/Fe_2O_3 ratio, and four have low CaO/MgO and high TiO_2/Fe_2O_3 ratios. The good compositional homogeneity of Norman specimens contrasts with the distribution of Byzantine specimens: the latter seem to have a casual distribution and no apparent grouping.

These differences are linked to the more careful preparation of Norman mixes. The aggregate/binder ratio in Norman samples is quite constant; calcarenite alone was used as an aggregate in only a few cases out of necessity rather than choice.

Petrographic analysis has highlighted how from the standpoint of granulometry, aggregate fragments in Norman specimens are better sorted. The fragments are often fractured, clearly indicating pre-processing by Norman masons prior to preparation of the mix. Data in the Mean Porosity vs. % Aggregate diagram (Fig. 2) provides further evidence that Normans took greater care than Byzantines in the preparation of mortars: the mean porosity is much lower in Norman specimens than in Byzantine ones. This difference is certainly due to the fact that Normans carefully dosed water when preparing the mix. The typical physical and/or chemical characteristics of each typology can be used to discriminate the

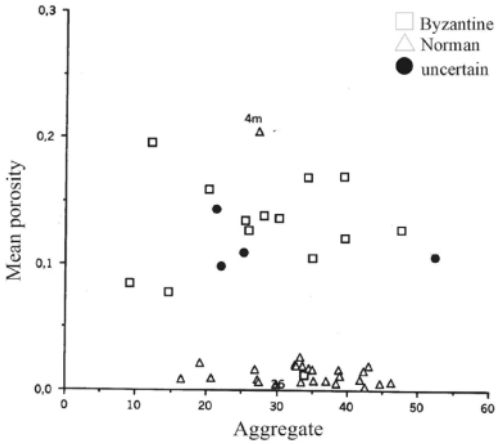

Fig. 2 – Representative samples of the Byzantine and Norman mortars (Santa Severina Castel, KR): Mean porosity vs Aggregate

different mortars. In this case, the diagram in Figure 2 confirms that samples 4 and 26 were erroneously classified as Byzantine and Norman respectively during the phase of architectonic investigation. Furthermore, four samples of uncertain attribution were classed as Byzantine materials on the basis of mean porosity data.

The structural mortars of San Lorenzo in Milan: a particular aggregate rich in technological information

The structural mortars used in the Late-Antique phase of construction of the San Lorenzo complex in Milan have a rather peculiar aggregate. These mortars contain fragments of vitreous slag and of mosaic tiles. The percentage of fragments is low in mortars from this phase of construction, and their presence is only sporadic in subsequent phases (Riccardi *et al*., 2004).

The slag have some common characteristics: small sample dimensions, a dark, translucent outer surface, rounded shapes, fine granulometry, and abundant macro- and micro-porosity. The extremely variable quantity of crystalline grains (degree of crystallinity), best observed on the fracture surface, was used to distinguish the different vitreous materials (Riccardi *et al*., 2004).

Observation of the macroscopic characteristics of mortars did not reveal whether the addition of slag to binders was deliberate or not. The low percentage of slag in aggregates suggests that its presence was unintentional and can probably be attributed to contamination by structural components of the lime firing kiln. Nevertheless, the vitreous slag in the aggregates was not inert but reacted with the carbonate binder during setting.

Reaction margins are some hundred microns thick (Fig. 3) and develop between two chemically reactive materials: a silicate system

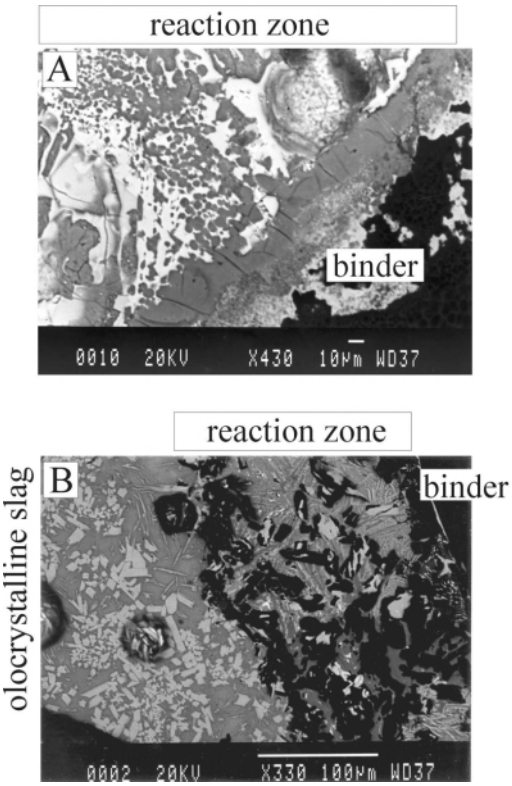

Fig. 3 – Structural mortars of San Lorenzo (Milano): the reaction microstructures (some hundred microns thick), between slag and binder, developed into coronitic structures.

(the slag) and a carbonate system (the binder). The formation of new crystalline or amorphous (mainly Ca-silicates) phases depends on the texture and composition of the vitreous slag. The reaction microstructures are two-phase symplectites which develop into coronitic structures.

Plasters from the cellars of the Visconti Castle in Pavia: the use of a «clayey» binder

The older plasters from the cellars of the Visconti Castle in Pavia consist of three main layers. The more ancient, coarse layer (20-30 mm thick) is directly sustained by the masonry and has a yellowish colour; the second technical layer is also a coarse plaster (3-12 mm thick) of whitish colour; the third technical layer is of limited thickness (50-70 μm) and has a homogeneous texture with extremely fine grain size. A bluish-black film consisting of thin levels (80-100 μm) is separated from the underlying technical layers by a surface of discontinuity. The coarse, black material is opaque to transmitted light; the binder has a very fine grain size and represents about 50% volume (Tomasi *et al*., 2001).

The first technical layer is a coarse aggregate with the granulometry of a medium-fine sand. It consists of quartz, feldspar (K-feldspar and plagioclase) and micas (biotite and muscovite). Mafic minerals are also present (i.e. green amphibole, garnets and subordinate pyroxenes). Lithic fragments mainly consist of metamorphic rocks and fine-grained sedimentary rocks (Tomasi *et al*., 2001).

The binder is not homogeneous and is opaque under the optical microscope. A fine-grained aggregate of pure calcite alternates with more complex areas where at least two different compositional phases are present (Fig. 4a). The identification of such phases is important to define the technical characteristics of the binder. The coexistence of carbonates and silicates raises the question of whether the binder had hydraulic properties or whether it was a mixture of an air-setting binder with clay. In order to determine the chemical composition of heterogeneous phases in the binder, areas displaying different properties under the optical microscope were analysed by electron microprobe. Results are reported in the ternary diagram $(CaO+MgO)+SiO_2+Al_2O_3$ (Fig. 4b). The phases in the binder are divided into two compositional groups: the first close to the (CaO+MgO) apex, the second containing high concentrations of SiO_2 (about 50%) and variable $(CaO+MgO)/Al_2O_3$ ratios, with only some compositions similar to those of clay minerals. Non-stoichiometric compositions

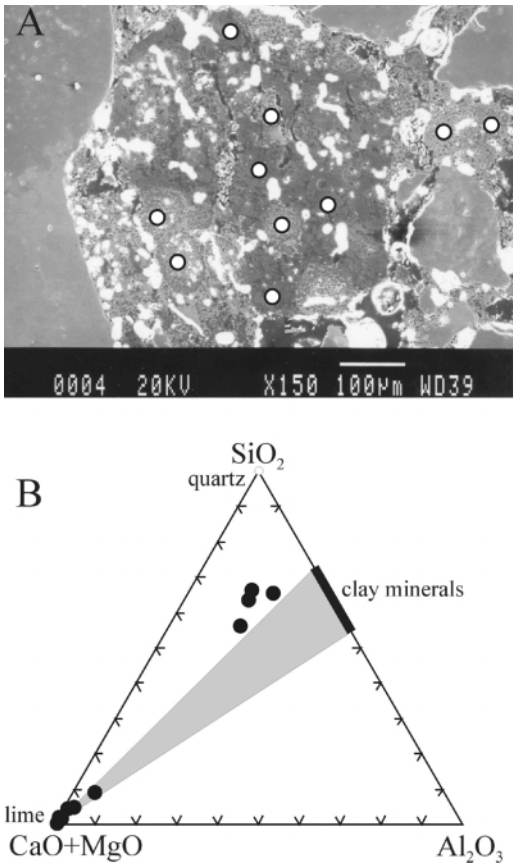

Fig. 4 – Plaster of the cellars of the Visconti Castle (Pavia): A) BSE imagery of binder inhomogenity; B) Compositional data of the binder obtained with Electron Probe Micro-analisys (EPMA). The open circles in Figure A correspond to full circles in Figure B.

such as those shown in Figure 4b may be justified taking into account the extremely small size of the phases, which is lower than the spot of the electron beam (~ 5 μm^2). In such cases microanalyses must be interpreted as «microbulk» analyses which cannot yield the definite composition of the binder.

The diffraction pattern shows that the fraction corresponding to the binder (< 63 μm) consists of carbonate and clay minerals in an approximately 1:1 ratio. The high percentage of clay minerals cannot simply be ascribed to the use of unwashed sand, but suggests the deliberate addition of clay material to the carbonate binder.

The mortars of the Leaning Tower of Pisa: a good medieval recipe for producing hydraulic binders

This example illustrates a special case of eminently hydraulic mortars (compressive strength of about 16 N/mm^2; mean SiO_2 content in the binder of about 29%) in which pozzolanic material is not visible to the naked eye, nor can it be detected through optical or electron microscopy (Franzini *et al.*, 1999; 2000a; 2000b).

The extremely high hydraulicity of the binder excludes its production through firing of marly limestones. Indeed, the intergranular binder and numerous lumps in the mix are snow-white with extremely variable silica contents (13-41%); their Al_2O_3/SiO_2 and K_2O/Al_2O_3 ratios are not compatible with those of clay minerals.

Collected data derived from whole samples and directly from the binder material indicate that the binder consists of a mix of lime putty with an extremely reactive and highly siliceous material. Of pozzolanic materials commonly used in the past to produce mortar, diatomaceous earth has the required characteristics (colour, high silica content, extremely high reactivity in an alkaline environment, etc.). In particular, although a provenance from other Mediterranean localities cannot be excluded, the ratio between major chemical components of diatomaceous earth quarried in the Mt. Amiata area is similar to that determined in mortars from the Tower of Pisa.

The presence of poorly crystalline or nearly completely amorphous CSH phases is confirmed by X-ray diffraction patterns, which reveal the weak characteristic peaks of these phases, and by the low calculated absolute density of the binder (2.565 g/cm^3).

As for the aggregate, its chemical and mineralogical composition is similar to that of local sands currently also found in the bed of the Arno River.

On the whole, mortars from the Leaning Tower of Pisa show rather constant characteristics throughout different construction periods and within individual periods. Only small differences were observed between mortars in the foundations and mortars in more recent portions of the monument: on average the granulometry of the aggregate and the hydraulic characteristics of the binder decrease, whereas the percentage of binder increases.

These findings indicate that the construction technique was well-established and did not vary significantly throughout the construction of the tower, which lasted about two hundred years from the 12^{th} to 14^{th} century. In western Tuscany at the time, the technique was apparently only known to masons in Pisa.

Magnesian lime at the medieval port of Genova

After the first studies on mortar in the medieval port of Genova (Mannoni, 1988), the ancient mortars in all investigated regions were found to be produced with magnesian lime.

Studies on lime production areas have demonstrated that this choice of raw material was deliberate and not casual (Vecchiattini, 1998; Fieni, 2000). This would be only a historical issue if the quality of ancient mortar through time had not proved to be even superior to that of cement mortars.

Research undertaken by the Department of Building, Urban Studies and Materials Engineering of Genova University, which takes

into account reactivity due to microstructural characteristics and chemical composition, is beginning to explain this phenomena. Historical research suggests that the quality of magnesian limes from the same source worsened after the introduction of industrial kilns; conditions in former kilns were therefore tentatively reconstructed in the laboratory on the basis of collected oral and written data (Vecchiattini, 1998). Results show that the low temperatures intentionally used to burn poor, humid wood created an atmosphere with water vapour and little CO_2; this atmosphere affected the size of CaO crystals (Beruto *et al*. 2003a; Beruto *et al*. 2003b).

Even if scientific research had been undertaken at the start of the 20th century, when carbon fossil fuel furnaces were introduced to reduce the firing time, it would not have been possible to obtain such data. How could such good choices, difficult even for modern materials science, be made empirically in roman and medieval times? One might reply that empirical knowledge was rational because it was experimental (trial-and-error); even now, when investigating complex phenomena, it is faster to conduct experiments on effects, i.e. empirically, rather than on causes as in modern science (Mannoni and Giannichedda, 1996). A similar situation exists for hydraulic mortars: according to modern technological know-how, ancient ones should be much less hydraulic than cement, whereas in actual fact they have proved to be more resistant (Giordani and Mannoni, 1999).

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