



9th International Masonry Conference 2014 in Guimarães

Earthen mortars in Cremona: characterization and first hypothesis of dating

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ABSTRACT: Historic buildings in Cremona are characterized by the almost exclusive use of earthen mortars, also for plasters. The use of earthen mortars was a conscious choice and the masonry structures are of very good quality. The thickness of the walls is considerable, at least four heads of bricks, their tissue is regular from the front to the back of the wall. Their resistance to compression has shown results similar to those of walls of the same period constructed with lime based mortar. Preliminary data of the archaeometric study reveal the compositional and microstructural complexity of the earthen mortars. The properties of some clay minerals to absorb water may be useful in many respects in a climate where the relative humidity is high. The empirical knowledge would allow the exploitation of a country resource.

Keywords: Cremona, earthen mortars, lime

1 INTRODUCTION

In northern Italy it is very easy to find carefully constructed brick walls built using earthen mortars, which co-exist with walls built using lime mortars. The earthen mortars walls are laid in regular courses throughout their thickness. Their depth coincides generally with multiples of a "head" of brick, typically four or five bricks. The regularity of the construction helped limit the thickness of the walls. Despite the fact that earthen mortar's compressive strength and adhesion are much lower than those of lime mortar, it was used for the construction of, at times, complex vaults, and more particularly in basement floors (where the moisture in the masonry and environment greatly accelerates the deterioration of the structure) or finally as a top layer for major mosaic flooring. Generally, the joints are filled with high calcium lime mortar which has often been pressed using a metal instrument. The covering in lime slowly changes in temperature and humidity preventing the progressive powdering of clay mortar that is alternately wet and dry.

The use of earthen mortars is widespread in the Po valley, both in rural buildings [1] and in very important historical building, such as noble residences, churches, monasteries and public institutions' buildings [2]. In the city of Cremona and its surrounding area this technique, together with entirely wooden constructions, was widely used since Roman times [3] [4].

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The analysis of ancient treatise reveals that the use of earthen mortar in Cremona doesn't originate neither from the difficulty in finding lime nor from lime's high cost: hydraulic lime was available from Piacenza [5] and the production costs of earthen mortar were comparable to those of lime mortar [6]. Contrary to previous ideas which consider the use of clay as building material a poor technique, it was the consequence of a conscious choice [6]; it was a building tradition, adapted to the climate and to the local needs.

From these treatises, however, it has not been possible to obtain precise data to understand the nature of the utilized materials, their making process or use. It is also impossible to identify the sites from which the earth was excavated, nor to understand which types of earth were best suited for each various uses. The only indication was offered by Professor Sonsis [7], who asserted that «a red earth which is seen at Cava Tigozzi, a small village near the city of Cremona, if mixed strongly with lime, even though weak, as it comes from the pebbles of the river Adda has a strength equal to the lime from Piacenza».

In recent times some researchers have studied the earthen mortars of Cremona [8] [9] and have [8][8]supposed that these mortars are made by a soil and by a sieved fluvial sand. They affirmed that the terrigenous component is mainly constituted by quartz, muscovite and clinochlore with, more rarely, illite, montmorillonite and biotite, while the sand is mainly constituted by quartz and feldspar [8] [9]. These authors observed that lime, in little amount, was added to these mortars, but they haven't understood if the interaction between the Ca²⁺ and clayey matrix is due to a superficial adsorption process or to some hydrosilicates formation [8] [9]. To better understand the structural properties and the minero-chemical characteristics of these mortars, a preliminary archeometric study on Soldi Palace's earthen mortars has been carried out.

By studying ancient treatise and the critical lecture of different architectural features of the buildings it has been possible to date some construction phases. Particularly in Palazzo Soldi it was possible to identify the 1776 AD as the year of beginning of the construction of some local at the ground floor and at the basement. The construction phases, that cannot be possible to date throughout this method, will be dated with the optically stimulated luminescence (OSL), an experimental technique successfully used in recent times to date mortars.

The final purpose of this study, in addition to the comprehension of the ancient building technique, is to reproduce the mortar for the restoration of the buildings.

2 EXPERIMENTAL SECTION

2.1. Analytical approach

A preliminary study of the earthen mortars was made on samples from Palazzo Soldi in Cremona.

To characterize these mortars, it was used an analytical procedure that combines petrographic, chemical and mineralogical data measured on the mortar bulk samples and chemical and mineralogical data measured on the fine fraction of the mortars.

Microstructural investigations of polished thin and cross sections were accomplished through optical (OM) and scanning electronic (SEM) microscopy coupled with an X-ray energy-dispersive system (EDS). Thin and cross sections carbon coating is required before SEM-EDS analysis. Scanning Electron Microscope is a Tescan FE-SEM Mira 3XMU-series, equipped with the system TRIDENT XM 4 - LEXS System with Hikari, integrating EDS. Operative conditions of the X-ray energy-dispersive system attached to the SEM are: acceleration voltage = 15 kV; excitation current = 20 mA; counting time = 100 s.

The mineralogical composition of the mortars' fine fraction was analyzed by X-ray powder diffraction (XRDP) and Fourier transform infrared spectroscopy (FTIR) while the chemical composition of both the fine fraction and the mortar bulk sample was analyzed by SEM-EDS.

2.2. Results

Earthen mortars from Soldi Palace are very complex and heterogeneous materials (Figure 1). They show a substantial variance in particle grain size and ratio between the fine fraction ($\leq 0.063 \ \mu$ m) and the aggregate (>0.063 $\ \mu$ m). Furthermore, they can contain sand levels and/or pockets, brick fragments and charcoal (Figure 1).

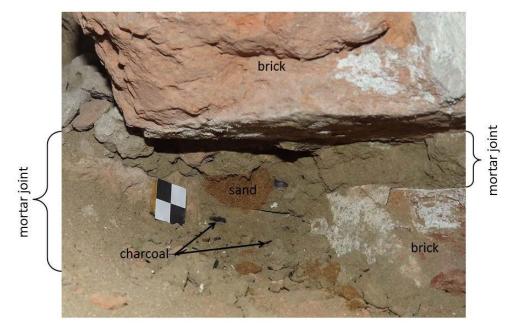


Figure 1. macroscopic view of the mortar joint in which are visible the fragments of charcoal and the pockets of sand.

The mortars have matrix support texture and are very rich in framework (50 - 70%). The framework is medium to coarse grained and moderately sorted. Grains have angular shape, rarely sub-rounded. The framework is composed mainly by quartz grains (60-70%) and less feldspars, phyllosilicates, amphiboles and, in some cases, brick fragments and charcoal (Figure 2.a).

Optical investigation shows that grains in sand level are similar, both for mineralogy, shape and dimension, to those that constitute the framework of the mortar.

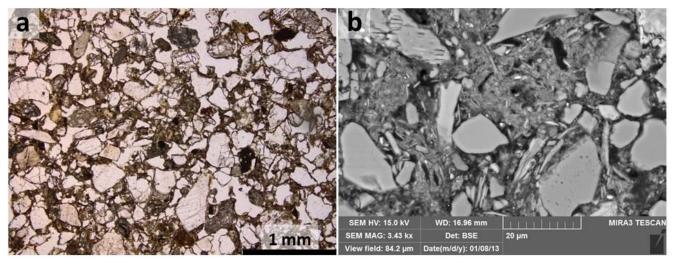


Figure 2. a) microscopic photograph of the earthen mortar. b) BSE image of the mortar in which are visible the clay minerals and the aggregate.

The fine fraction is composed by very small minerals, which aren't recognizable under the optical microscope. Consequently, it has been investigated by SEM (Figure 2.b). Nevertheless, the spot of the EDS isn't small enough to make punctual analysis of these minerals, so chemical analyses of the fine fraction are always areal analysis.

In Table 1 the chemical composition of the earthen mortars and of the fine fraction are reported. The comparison between SiO_2 and Al_2O_3 values of the mortar and of the fine fraction indicates that the framework is mainly constituted by quartz grains, as already revealed by optical microscopy. The variation of the CaO content, which is higher in the fine fraction than those of the bulk mortar, revealed that in the fine fraction of the mortars exists a carbonatic component associated to clay minerals.

Description	mortar	mortar	mortar's fine fraction	mortar's fine fraction	brick fragment in mortar	brick fragment in mortar	brick	brick
SiO ₂	70.85	70.67	58.40	59.21	61.32	53.87	52.70	59.15
TiO ₂	0.00	0.35	0.82	0.63	0.00	0.80	0.00	0.00
Al ₂ O ₃	13.33	12.87	20.95	19.82	20.24	21.23	15.52	15.86
FeO	5.50	3.90	7.28	7.12	7.76	6.97	6.10	7.27
MgO	2.27	1.59	2.80	2.88	2.40	4.76	6.27	4.91
CaO	2.77	6.04	3.02	5.36	2.36	6.70	14.17	8.05
Na ₂ O	1.34	1.99	1.07	0.60	1.15	1.47	1.81	1.49
K ₂ O	2.89	2.17	4.67	3.51	4.61	3.85	2.57	2.78
Cl	0.77	0.41	0.77	0.79	0.00	0.35	0.00	0.00
S			0.21	0.08			0.85	0.49
Total	99.72	99.99	100.00	100.00	99.84	100.00	99.99	100.00

 Table 1.
 Chemical analyses of the main constituent of mortars and bricks

The presence of CI and S can be explained by degradation phenomena already in action.

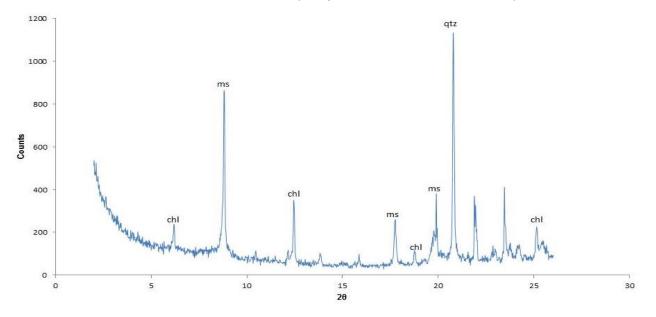


Figure 3. XRPD diffractogram of the fine fraction of the mortars. The abbreviations above the peaks indicate the minerals they refer to. ms: muscovite; chl: clinochlore; qtz: quartz.

The bricks of the masonry were made with a Ca-rich mixture, while the brick fragments found in the mortar have lower CaO value. Also the K_2O content is different between the two kinds of brick; particularly it is higher in the brick fragments.

The comparison between XRPD data (Figure 3) and FTIR data (Figure 4) indicates the presence in large amounts of quartz (XRPD: 20.78 20; Figure 3), as already detected by the optical investigation. It shows also that the fine fraction of the mortar is composed mainly by clinochlore (Figure 3; XRPD: 6.2, 12.44, 18.74, 25.14 20. Figure 4; FTIR: 958, 1434 cm⁻¹) and muscovite (Figure 3; XRPD: 8.8, 17.74, 19.9 20. Figure 4; FTIR: 744, 815, 904, 958, 3619 cm⁻¹). However, this is only a preliminary investigation and needs further analyses to better investigate the fine fraction of the mortar.

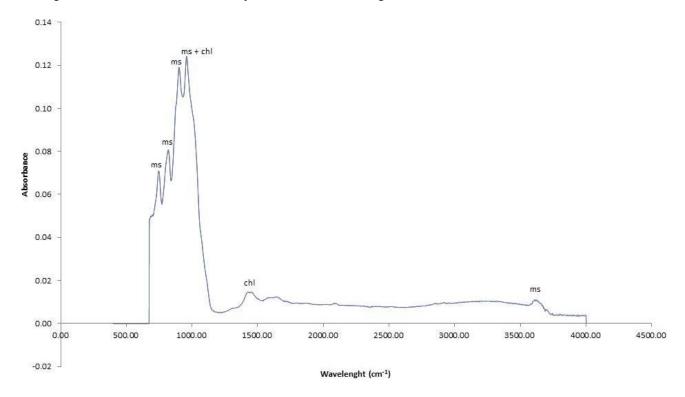


Figure 4. FTIR spectra of the fine fraction of the mortars. The abbreviations above the picks indicate the minerals they refer to. ms: muscovite; chl: clinochlore; qtz: quartz

Chemical data of the main constituents of the mortars and of the bricks were plotted in a ternary diagram CaO-Al₂O₃-SiO₂ (Figure 5).

The average composition of the bricks, of the mortar and of the brick fragments contained in the mortar plots close to the silica vertex, but it forms different data sets. Moreover, it can be observed that the average composition of the mortars is very different from the composition of the fine fraction of the mortar.

In the CAS ternary diagram (Figure 5) were also plotted some chemical analyses of clays current utilized for brick production carried out by Plastes laboratory on clayey soil placed near Cremona. These latest analyses were found during an archival research focused to found any information about the earthen mortars, the architecture and the clay quarries of Cremona. The chemical composition of these clayey soils plots very close to the composition of the fine fraction of the mortars and the composition of the brick fragments found in the mortar, whereas it deviates a little from the composition of the bricks. Moreover, the chemical composition of the brick fragments, of the mortars' fine fraction and of the clays analyzed by Plastes laboratory plots on the CaO-clay minerals join in the ternary diagram (Figure 5), that is the typical field of the composition of the bricks, while the composition of bricks of the masonry plots little away from it.

Raw and building materials were also plotted in a sand-silt-clay diagram [10] (Figure 6), and that allows to make some considerations. The mixture of the mortars, in terms of aggregate and binder amount, doesn't correspond to the natural soils that outcrop in the Cremona's area [9] nor to the raw materials utilized for current brick production [11] [12]. The high amount of framework in the mortars leads to suppose that soils naturally enriched in coarse grain fraction (clayey sand soils and silty sand soils in Figure 6) were selected or that, in construction site, it was realized a mixture by adding a coarse grain material to the soil.

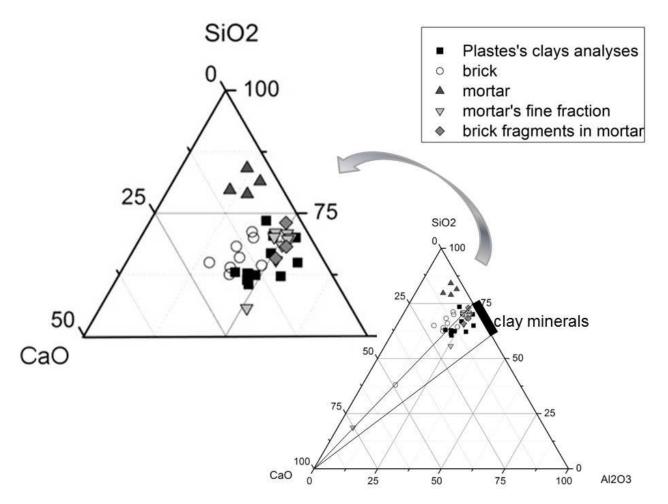


Figure 5. ternary diagram CaO-Al₂O₃-SiO₂ in which are plotted the chemical composition of the principal constituents of the mortar, of the bricks and the chemical analyses collected by Plastes Laboratory. In the diagram is highlighted the typical composition of the clay minerals.

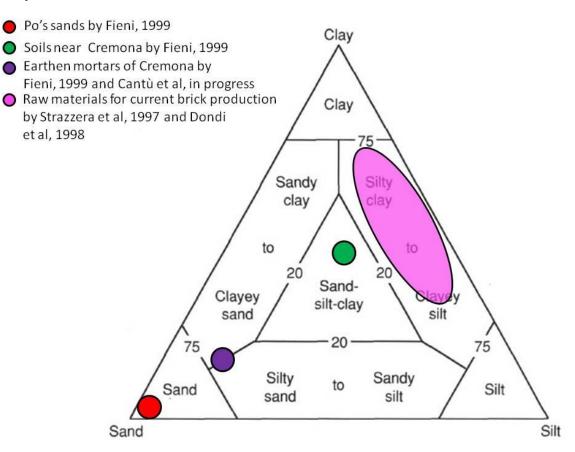
Finally, it must be said that sand-rich soils have low cohesion. These materials require the addiction of a binder and this support the hypothesis that lime were added to Cremona's earthen mortars, though in little amount.

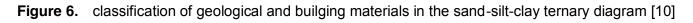
3 DISCUSSION AND CONCLUSIONS

The earthen mortars of Cremona are constituted mainly by terrigenous materials, but at the moment it's impossible to establish if their composition is natural or made by mixing two different kind of soil.

In accordance to what found in an historical treatise [7] and in the more recent literature [8] [9], this material was added with lime, probably to increase its strength, and it caused the development of reactions between clay and lime, with the formation of new C-S-H and C-S-A-H phases.

By plotting the analytical data of the mortars and the bricks in a ternary diagram CaO-Al₂O₃-SiO₂, some considerations can be made about the materials utilized in the architecture of Cremona. Since the chemical data of every component of the mortar and of the bricks plot in well-defined groups, this probably indicates that the raw materials utilized were different. This difference is important especially for bricks and brick fragments, because it indicates that ancient materials were recycled and added intentionally to the mortars.





Furthermore, by comparing the mortars' chemical data and the Plastes's analyses it can be possible to hypothesize that the same clayey soils were first utilized to produce bricks and then to produce the mortars of Soldi Palace, while a different clayey soil were utilized to produce the bricks.

At the moment, there aren't hypothesis to explain the presence of the charcoal in the mortar, also because they are in very small amount and may not have any function in the mortar.

The packaging of these materials is, however, linked to a complex procedure, both if it is hypothesized the use of natural heterogeneous row material and if it is hypothesized the use of an artificial mixture; that means two or three different materials were employed (soil, sand and lime).

In the end, another important indication is that the preparation of this material required competence and time, as indicated in some ancient treatise [13] [14].

The minero-chemical analyses showed also the presence of calcium carbonate, C-S-H and C-S-A-H phases, principally localized at the contact between the joint and the brick. Where the C-S-H phases

are sited there are some interesting microstructures, such as dissolution rims, reaction rims and the formation of new phases, which indicate that clay minerals have reacted with a Ca-phase [6].

Finally, some constructive phases of Palazzo Soldi are dated at about 1770 thanks to the critical lecture of its architectural features and to information reported in ancient treatises. The phases of uncertain age will be dated with the optically stimulated luminescence technique (OSL), because the occurrence of large amounts of quartz in earthen mortars makes them suitable for this approach.

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