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Lime mortar with natural pozzolana: Historical issues and mechanical behavior

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ABSTRACT: This paper deals with the investigation of the mechanical behavior of lime mortar enriched with natural pozzolana, widely used in historical masonry. The study aims to investigate the mechanical behavior of natural pozzolana lime mortars, reproduced in laboratory by using different mix designs, as reported in historical Italian architectural treatises, and natural pozzolana coming from Viterbo (Italy). To this purpose, the works of the most important authors from the Ist century B.C. up to 1864 have been analyzed, in order to collect historical mix designs of pozzolana lime mortars. Flexural and compression tests were performed following EN 1015-11 (2007). Mechanical tests were performed after 28 days and 60 days of curing. Penetration tests, not included in European Standards but very suitable for practical applications, was also performed.

1 AIMS AND RESEARCH METHODOLOGY

Historical evidence on the use of mortars to meet several needs in architecture has existed for millennia. A systematic analysis of ancient mortars dating some hundred or thousand years proved that they have resisted decay by time and other deterioration factors. Difference concerning their durability and the degree of their degradation are attributed to many technological aspects that characterize each specific period, such as the binding methods used, the type and proportion of aggregates, application system as well as the craftsmen skills.

Most of the mortars dated from the historic period are based on the use of lime and many of them are enriched with natural siliceous volcanic sand, commonly called pozzolana. Referring to the Mediterranean building culture and practice, it is known that, since 1500 B.C., Greeks used a highly siliceous volcanic Santorini earth in wall coverings (Moropoulou, 2004). Furthermore, it is widely accepted that the first extensive use of pozzolana was adopted by the Romans who, since the II century B.C. (at least) used a volcanic sand in the *opus caementicium* (i.e. a lime mortar mixed with an aggregate of small stones or rubble), whenever possible. This material, often covered by dressed stones or bricks, represented, from the foundations to the vaulting, the very core of Roman masonry (Lugli, 1957; Lancaster, 2005, also for further references). Pozzolana remained at the base of building practice during the modern ages up to the industrialization, still inspiring, although new materials and systems were developed, the basic concept of contemporary concrete.

In the past, it was empirically clear that pozzolana provides hydraulicity to lime mortar, enabling its setting in wet conditions or underwater. Furthermore, it was known that the addition of pozzolana was useful to increase the durability of lime mortar, allowing it to overcome the need of repeated maintenance and accelerating the carbonation process, as it has been also scientifically proved (Collepardi, 1993). Up to now, however, such favorable properties of natural pozzolana have been subdued to relatively little scientific investigation carried out by means of mechanical tests. Due to the widespread use of pozzolana lime mortars in historic masonry, wider scientific and technical issues about mechanical properties of this kind of mortars would be very useful in repairing and maintenance of historic architectural heritage.

As the aim of this study was to analyze the mechanical behavior of lime mortar enriched with natural pozzolana, a new scientific investigation methodology had to be adopted. It should be observed that it is almost impossible to extract from historical buildings mortar samples without altering the mechanical properties of the material; moreover, the required dimensions of the samples would make this kind of test too invasive. As a consequence, specific samples suitable for mechanical tests had to be prepared. In this respect, the correct reproduction of the historic mix designs, with their different compositions, was a problem of critical importance. On this subject, actually, in absence of convincing primary archival sources, there is no general assessment and several attempts on the characterization of historical mortars were recently made without historical evidence. It was therefore decided to prepare the different mix designs following the indications traced in the architectural treaties, printed documentary sources that can be considered as the official expression of the building culture, generally stating what, in the past, the common sense regarded to as "the rule of the art". Although the craftsmen skills and the circumstances often adapted those guidelines to the case at hand, the mix designs described in the treaties can be taken as a reasonably reliable reference to reproduce historic mortars.

The works of the most important Italian authors from Vitruvius (I cent. B.C.) to Giovanni Curioni (1864) (and also some French authors whose treaties gained great success in the Italian architectural culture) were therefore analyzed in order to collect pozzolana lime mortars historical mix designs. Following the ratios suggested, samples were reproduced in laboratory by using natural components only, and adding Pozzolana originated from the Roman volcanic district of the Monti Sabatini.

The different lime mortars were tested according to European Standard EN 1015-11, after 28 days and 60 days of curing. A penetration test, not included in European Standards but very suitable for practical applications, was adopted as well.

2 HISTORIC PERSPECTIVE

Much of what is known about Roman architectural culture derives from Vitruvius, who in his *De Architectura* (I century B.C.) discussed about building knowledge gathering together specific information, construction guidelines and design principles probably widespread in Rome at the birth of the Empire. In the II book of this treaty it can be found the first written reference to pozzolana mortars: "*Est etiam genus pulveris, quod efficit naturaliter res admirandas. Nascitur in regionibus Baianis in agris municipiorum, quae sunt circa Vesuvium montem. Quod commixtum cum calce et caemento non modo ceteris aedificiis praestat firmitates, sed etiam moles cum struuntur in mari, sub aqua solidescunt.*" (Vitruvio, 1997, II, VI: 132). Vitruvius referred to the *pulvis puteolana*, a sand originated from thick deposits of reactive volcanic tuffs present around *Puteoli*, the vesuvian town near Naples (today Pozzuoli) from which the name of this material is derived (Lugli, 1957; Adam, 1989; Lancaster, 2005). This kind of sand, currently used in building Roman practice, was also easily found in the surroundings of Rome, which is situated between two well known volcanic districts. Only two other important Roman authors seem to have focused on pozzolana after Vitruvio. Seneca mentioned pozzolana (62–65 A. D.), spending on it only few words (Seneca, 2004, III, XX, 3: 285). Plinius, instead, in his *Naturalis Historia* (within 78 A. D.), wrote about pozzolana underling its ability of hardening underwater (Plinio, 1987, XXXV, XLVII: 1259); he also seems to be the first author who hinted at pozzolana capacity of increasing mortar strength with the age.

The worth of Vitruvius' work increases if we consider that in his treat the first written indications for obtaining pozzolana lime mortars can be traced, asserting that the ideal proportion (pozzolana:lime) was 2:1 (as volumetric ratio) *"Hae autem structurae quae in aqua sunt futurae, viderunt sic esse faciendae uti portetur pulvis a regionibus quae sunt a Cumis continuatae ad promonturium Minervae, isque misceatur uti in mortario duo ad unum respondeant"* (Vitruvio, 1997, V, II–III: 586–588). Later, many authors referred to pozzolana in their architectural treaties as well, but they didn't give practical and technical information on the use of this volcanic material. In particular, from XV century up to the end of XVIII century, architects substantially referred to Vitruvius' treat adding only accessory comments. Among more than fifty treaties analyzed, there were traced only 24 mix designs pozzolana lime mortars.

During the XV and the XVI centuries, the only Italian author that considered the pozzolana for practical purposes was Francesco di Giorgio Martini (1480–82), who quoted Vitruvius' pozzolana lime mortar mix design (Martini, 1967, I, VIII: 105) but misunderstanding his indications and inverting the pozzolana:lime volumetric ratio (Table 1).

No pozzolana lime mortars mix designs can be traced in the most important Italian Renaissance architectural treaties. Alberti (1450) widely discussed about lime mortar and limestones, but only hinted at pozzolana (Alberti, 1989, II, XII: 86). Filarete (1460–64) hinted to pozzolana too, but sending back to Vitruvio for further information (Filerete, 1972, III, p. 67). Serlio (1537) spent a lot of pages of his work describing important roman buildings, but didn't write about materials and didn't mention the use of pozzolana. Vasari (1550), though not directly referring to volcanic material, praised the building good manufacture in the area around Pozzuoli (Vasari, 1986, XXVII: 74). Palladio (1570) hinted at pozzolana (Palladio, 1980, I, IV, p. 15), focusing his attention on the hydraulic lime used in the Venetian region around the town of Padova (Palladio, 1980, II, IX, p. 77) and indicating, as the first among the Renaissance authors examined, the quick hardening as a peculiar quality of pozzolana mortar. Cataneo (1567) was the first who tried to explain the hardening process of a pozzolana lime mortar (Cataneo, 1985, II, IV: 269–270) and Scamozzi (1615) dedicated an entire chapter of

Table 1. Pozzolana lime mortar mix designs traced in architectural treaties.

Reference	Period	Pozzolana	Lime	Sand	Granite	Pumice	Volcanic Breccia
Vitruvio	(I century B.C.)	2	1				
Martini F. di Giorgio	1480–82	1	2				
Viviani Q.	1830	12	9	6			6
” ”	1830	2	1	1			
Valadier G.	1831	5/6	1/6				
Quatremere de Quincy	1832	3	1				
” ”	1832	2	1				
Cavalieri San Bertolo N.	1832	0.85	0.15				
” ”	1832	0.75	0.25				
” ”	1832	0.70	0.30				
” ”	1832	0.55	0.45				
” ”	1832	0.64	0.36				
” ”	1832	0.78	0.22				
de Cesare F.	1855	2	1				
” ”	1855	6	5	3	6		
” ”	1855	4	4	3	9		
” ”	1855	57	11	8			
” ”	1855	3.5	6			3.5	9
Claudel J. & Laroque L.	1863	0.2	0.25	0.94			
” ”	1863	0.45	0.89	1.00			
” ”	1863	0.04	0.36	1			
Curioni G.	1864	2	4	1			
” ”	1864	3	1				
” ”	1864	1	1	2			

his work describing pozzolana, but without giving any practical indication of its use (Scamozzi, 1982, II, VII, XXII: 236–237).

During the end of the XVIII and the first half of the XIX century, up to the Sixties, architects and engineers turned to focus their attention on the practical uses of pozzolana. Some authors, like Milizia (1781) (Milizia, 1991, III, I, IV: 37–38), Rondelet (1802) (Rondelet, 1834, I, I, I, III, II:125; III: 147–151; II, II: 249–250) and Valadier (1831) (Valadier, 1992, I, II, XI: 58–59) discussed about the geological and the chemical characteristic of the volcanic material, its geologic origin and the deposits different location. On the other hand, Viviani (1830) (Viviani, 1830, VI- II: 125), Valadier (1831), Quatremere De Quincy (1832) (Quatremere de Quincy, 1842, II: 99–100), Cavalieri San Bertolo (1832) (Cavalieri San Bertolo, 1845, II, IV: 41), De Cesare (1855) (De Cesare, 1855, I, V: 49), Claudel-Laroque (1863) (in Curioni, 1864, 1, VII: 133) and Curioni (1864) (Curioni, 1864, 1, VIII: 134) proposed different mix designs (Table 1).

The growing concern with faster settings and more durable mortars soon led to the development and to the spread of modern cements. Since the late XIX century, the increasing use of Portland cement displaced the pozzolana lime mortar and led to the end of the interest of the architectural culture and practice toward it.

3 MATERIALS AND EXPERIMENTAL PROGRAM

3.1 *Geological and chemical aspects of pozzolana used in experiments*

To evaluate the natural pozzolana contribution to the increase of the strength in a lime mortar, specific samples of mortar were prepared in laboratory adding the Pozzolana rossa originated from the volcanic complex of the Monti Sabatini (Rome, Italy). As it well known, Rome is situated between two volcanic districts, the Monti Sabatini and the Albano Hills (Colli Albani), which have been active for most of the last millions years (Karner et al., 2001b).

Tephra deposits from the Monti Sabatini crop out extensively to west of the Tiber River and to the north of Aniene River. Three major pyroclastic flow deposits from the Monti Sabatini crop out extensively along the Tiber River Valley. They are, from the oldest to the youngest, the *Tufo Giallo della Via Tiberina*, the *Tufo Rosso a Scorie Nere*, and the *Tufo Giallo di Sacrofano*. A series of ash fall (i.e. pozzolana, which was used in the tests), surge and pyroclastic flow deposits separate these eruptive units (Karner et al., 2001a).

The chemical characterization of the pozzolana used for laboratory tests is shown in Table 2.

3.2 *Mix chosen and test procedure*

The 24 mortar compositions traced in the architectural treaties analyzed (I cent. B. C. – 1864) were reproduced and tested in a first testing phase. By the light of this first experimental step, a second, more punctual testing phase followed, choosing only five mix designs (Table 3) (due to synthesis requirements, only the second testing phase results are reported and discussed herein, while whole set of results can be found in Sala, 2008).

In reproducing the mortar specimens, both aerial hydrated lime and hydraulic hydrated lime were used.

The new European standard for limes (EN 459 2001) defines three types of limes: natural hydraulic lime (NHL), natural hydraulic lime with added material (NHL-Z), and artificial hydraulic lime (Z). In the present study, the specimens were prepared with natural moderately hydraulic lime NHL 3.5 (resistance after 28days: 3.5–10 MPa; UNI EN 459-1:2001).

No indications about water quantities are given in the mortar mix designs reported in the architectural treaties (Table 1); therefore, the percentage of water added in the mortar specimens to be tested was determined in relation to the workability of the mixes.

Tests were carried out according to European Standard EN 1015-11 procedure in terms of preparation and conditioning of mortar specimens to determine the flexural and compressive strength of mortars.

Table 2. Chemical composition (%) of the pozzolana used for laboratory tests.

SiO_2	Al_2O_3	FeO_2	TiO_2	CaO	MgO	K_2O	Na_2O	p.f.
48.7	17.1	7.7	0.8	9.6	4.2	8.7	2	0.1

The test specimens were prisms 160 × 40 × 40 mm; three specimens were provided for each age of test. Preparation and condition of storing specimens were chosen in compliance with EN 1015-11.

The specimens were cast in metal moulds and eventually placed in polyethylene bags for 2 days; afterwards, the moulds were removed and the specimens remained in the bags for the following 5 days (storage temperature of 20°C ± 2°C; relative-humidity 95% ± 5%). After these 7 days of curing, the samples were placed in a humidity room for 21 days (room temperature = 20°C ± 2°C; relative humidity = 65% ± 5%).

Flexural strength of mortars was determined by three-point-bending tests on the prism specimens. The two halves derived from the flexural strength tests provided, for each age of test, six half prisms to be used for the compressive strength tests.

Moreover, other tests were carried out on different specimens, named "sandwiches" (two bricks and a mortar joint of 20 mm), in order to show a relationship between the compressive strength and a probe penetration rate. The method is based on the measurement of the penetration depth increment of a steel probe by means of repeated blows of a Schmidt rebound hammer (Felicetti & Gattesco, 1998; Giuriani & Gubana, 1993).

Specimens were tested after 28 days and 60 days of curing.

4 EXPERIMENTAL RESULTS

4.1 *Flexural and compressive strength*

Test results, at the ages of 28 days and 60 days, are shown in Table 4.

4.2 *Penetration tests*

Penetration tests results were obtained after 28 and 60 days of curing (Figure 1); results are reported in Table 5.

Table 3. Mortar composition with different volumetric ratio.

Reference	Period	Identification	Pozzolana	A.H. Lime	H.H. Lime	Sand	Water
Vitruvio	(I century B.C.)	V1	52.6%	26.3%			21.1%
Vitruvio	(I century B.C.)	V2	51.6%		25.8%		22.6%
Viviani Q.	1830	Viv1	41.5%	20.8%		20.8%	16.9%
Viviani Q.	1830	Viv2	40.6%		20.3%	20.3%	18.8%
Quatremere de Quincy	1832	Q	61.5%	20.5%			18.0%
Cavalieri San Bertolo N.	1832	Ca1	56.6%	24.5%			18.9%
Cavalieri San Bertolo N.	1832	Ca2	57.5%		24.1%		18.4%
Curioni G.	1864	Cu	60.0%		20.0%		20.0%

Table 4. Evolution of average flexural and compressive strength.

	Avg flexural strength [MPa]			Avg compressive strength [MPa]		
Reference	28d	60d	Incr.	28d	60d	Incr.
V1	1.39	1.51	8.57%	4.47	5.38	20.5%
V2	2.12	2.66	25.4%	7.16	9.49	32.5%
Viv1	2.04	2.09	2.30%	6.33	7.96	25.7%
Viv2	1.38	1.20	−13.0%	4.26	4.76	11.9%
Q	1.63	1.77	8.61%	5.46	6.78	24.2%
Ca1	1.47	1.52	3.72%	4.85	5.21	7.43%
Ca2	1.59	2.03	28.1%	5.83	6.73	15.5%
Cu	0.81	1.03	26.9%	4.07	4.22	3.79%

Figure 1. Penetration test.

Table 5. Evolution of average penetration tests results.

	Number of hits/cm	
Reference	28d	60d
V1	5.44	6.56
V2	8.00	11.11
Viv1	7.67	11.33
Viv2	5.56	8.56
Q	6.78	10.11
Ca1	5.33	8.67
Ca2	5.89	10.78
Cu	4.00	6.00

5 DISCUSSION

The diagram in Figure 1 shows the influence of aging on the compressive strength of different pozzolana mortars. It can be observed that, in general, mortars with hydraulic hydrated lime (V2, Viv2, Ca2) show higher mechanical behavior and reach higher compressive resistance values in comparison with the same mortars with aerial hydrated lime (V1, Viv1, Ca1).

Referring to results from mechanical tests on mortar with pozzolana and hydraulic hydrated lime, Vitruvius' mix design (V2), with 2:1 pozzolana:lime volumetric ratio, shows the best results.

Vitruvius' mix and, in particular, V2 specimen shows the highest strength increment with time; in fact, the compressive strength of 7.16 MPa after 28 days of curing, reaches 9.49 MPa after 60 days.

Tests on Ca2 (7:3 and no sand) provide lower values of flexural and compressive strength in comparison with V2 specimens, therefore 2:1 seems to be the best volumetric ratio for mortar composed by pozzolana and hydraulic hydrated lime.

Using aerial hydrated lime, results are little different. Q mix (3:1 and no sand) shows the best mechanical performance. The use of a greater pozzolana percentage in the mix is evidently due to the hydrated lime incapacity to increase strength with time. The strength increment is therefore ensured only by pozzolana addition.

Viv1 and Viv2 specimens have the same pozzolana:lime volumetric ratio (2:1) of specimens V1 and V2, but they also have a percentage of sand. The flexural and compressive strength results lower than Vitruvius' one.

During the preparation of the mortar specimens it was observed that the medium value of the volumetric percentage of water added in the mortars, for obtaining the same workability, is 20% and that the water increases for higher quantities of pozzolana. On the other hand, the use of sand in the mix doesn't seem to influence the water quantity.

As mentioned above, in order to verify a non-destructive test method for practical uses, some penetration tests were performed. Experimental results, plotted in Figure 3 for 28 and 60 days of curing, show a good direct correlation between compressive strength and penetration test results. The trend line, obtained from the test results after 28 days of curing (Figure 3), shows a good correlation between the two quantities ($K^2 = 0.8251$). This evidence demonstrates the possibility of using penetration tests to obtain correct information about historical mortars mechanical characteristics with a non-destructive test.

The second diagram in Figure 3 shows the trend line between compressive resistances and penetration tests after 60 days of curing. The average deviation ($K^2 = 0.7066$) is lower than the previous one; however, the direct correlation between the two quantities is clearly evident.

6 CONCLUSIONS

The research deals with the study of historical lime mortars enriched with natural pozzolana. Historic mix designs were traced in the architectural treatises, trough the analysis of more then fifty authors from Vitruvius (I cent. B. C.) to Curioni (1864).

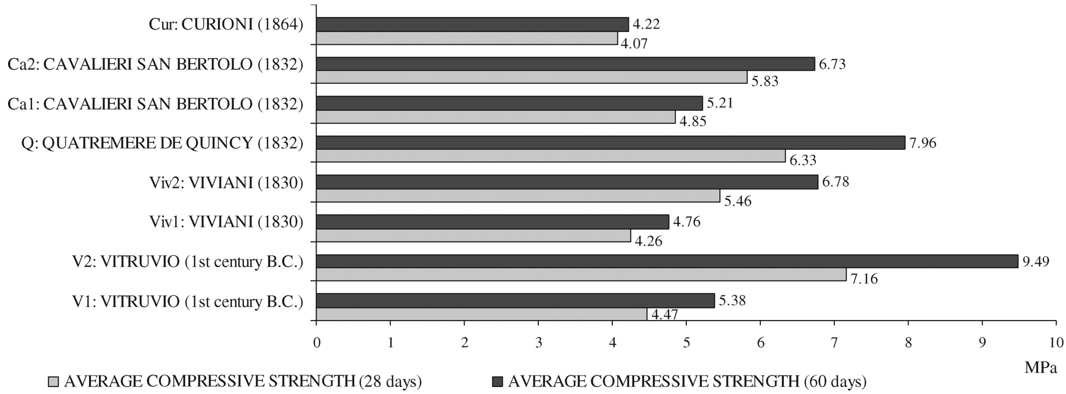

Figure 2. Influence of aging on compressive strength.

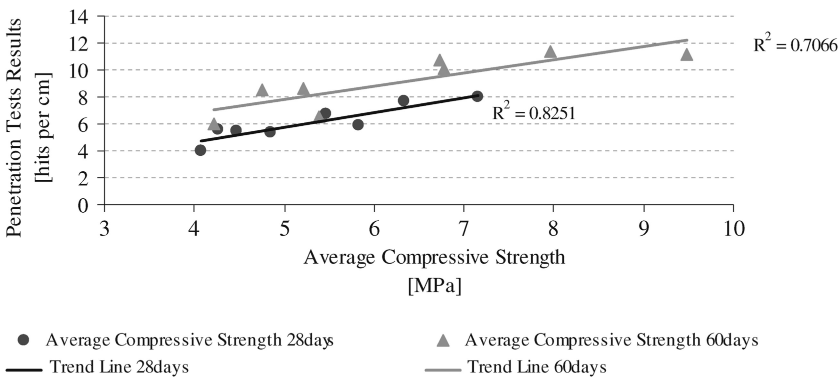

Figure 3. Trend lines. Average Compressive Strength – Penetration tests results (28 days and 60 days).

From an historical point of view, the study of mortars manufacture provides useful information on the building culture and the use of building materials, eventually helping in indicating different building phases and in setting conservative interventions. From a technological point of view, mortar identification is a fundamental step in the design of the repair materials, taking into account the necessary compatibility between the original materials and the new ones.

An exhaustive sampling of natural pozzolana lime mortars was carefully accomplished in order to start off a first study on their mechanical properties which, up to now, were subdued to relatively little scientific investigation. Flexural and compressive tests were carried out, after 28 and 60 days of curing.

The main results of this study can be summarized in the following.

- Historical analysis of architectural treaties shows a continuous reference to Vitruvius' indications. Up to the end of the XVIII century, official architectural culture seemed to be concerned with the good properties of the pozzolana and of volcanic materials but it didn't propose new pozzolana lime mortars compositions. Only at the beginning of the XIX century, many authors turned to examine the practical uses of pozzolana, deepening its technical properties and giving new mix designs.
- An exhaustive number of mortars mix designs were selected and reproduced in laboratory in compliance with UNI EN 1015-11. They were characterized in relation to their mechanical properties. The tests clearly show that the compressive mechanical strength increases with the addition of pozzolana.
- The mortars with hydraulic hydrated lime, proposed by Vitruvio (V2; 2:1 pozzolana:lime volumetric

ratio), showed the best mechanical properties; good performance was also shown by Quatremere de Quincy's mix design (Q) (3:1), using aerial hydrated lime.
- In general, mortars obtained with hydraulic hydrated lime show a better mechanical behavior than those obtained with aerial hydrated lime.
- Some further experiments were also performed in order to verify the reliability of the penetration test, a simple and non-destructive test that is significant for practical use (mortar prisms are not easily obtainable from historic buildings). The comparison between the compressive strength determined from penetration tests and the one determined from compressive tests confirmed that there is a good correlation. Therefore, the penetration test, can be successfully adopted for studying the mechanical response of mortar joints in historical masonry and, moreover, it can be easily used for in-situ tests.

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