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CHARACTERIZATION OF MORTARS FROM ANCIENT AND TRADITIONAL WATER SUPPLY SYSTEMS IN SICILY

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Long aged mortars from ancient hydraulic constructions of Sicily, i.e. the Roman aqueduct of Thermae and the Punic cisterns and traditional water supply systems in Pantelleria, have been characterised by means of XRD analysis, optical microscopy and simultaneous thermal analysis to correlate the hydraulic properties to the texture and to their different role in the construction, i.e. lining, covering, roofing and joint mortars.

According to a procedure proposed in the literature all of the samples, but two air hardening ones, show high hydraulicity, which somehow can be related to the characteristics of aggregates.

Keywords: *DTA-TG, historic mortars, hydraulicity, punic cisterns, Roman aqueduct, texture*

Introduction

Aim of this paper is to study different types of mortars from historic water supply systems, comparing both the hydraulic properties and the textural characteristics, in relation to their role in the construction. Two hydraulic systems were considered: a) the Roman aqueduct of Thermae (nowadays Termini Imerese), called Aquae Corneliae; b) the Punic cisterns and the traditional rain water storage network of the volcanic island of Pantelleria, 100 km offshore the southern coast of Sicily.

In the Roman aqueducts flowing water was let in slightly sloping channels, that contoured the smallest valleys, while the deep valleys were crossed by bridges or sometimes by means of siphons connected by lead or fictile pressure pipes. The aqueduct that fed the ancient town of Thermae, Fig. 1, is the most interesting Roman aqueduct in Sicily, due to the complexity of its layout with the presence of all such different components. Water tapped from the Brucato spring was let in a system composed by covered channels, galleries, arched bridges and siphons [1].

The traditional water supply system in Pantelleria [2], Fig. 2A, since the Punic era, is designed to optimize the rain water collection from any lined surface and is composed by: 1) the roof, made of rock masonry slightly raised on the ground level and slightly sloping towards an adduction channel; 2) the adduction channel, that conveys the water from the roof of both the cistern and the nearby houses; 3) the overflow channel, at the same level of the cover of the cistern; 4) the settling basin, to purify the water from suspended particles; 5) the storage basin, made of an underground rock masonry or directly carved into the rock body; 6) the drawing hole circular or square,

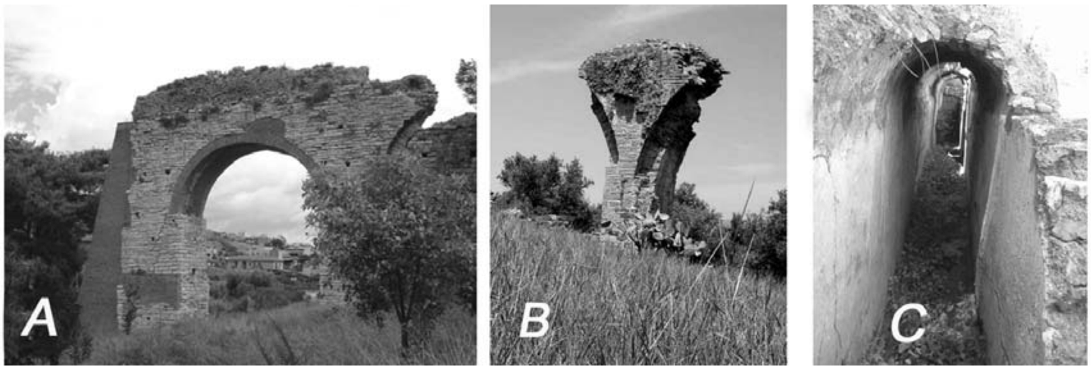

Fig. 1 Aquae Corneliae: A – Barratina siphon, B – Tre Pietre siphon, C – Contrada Tenaglia channel

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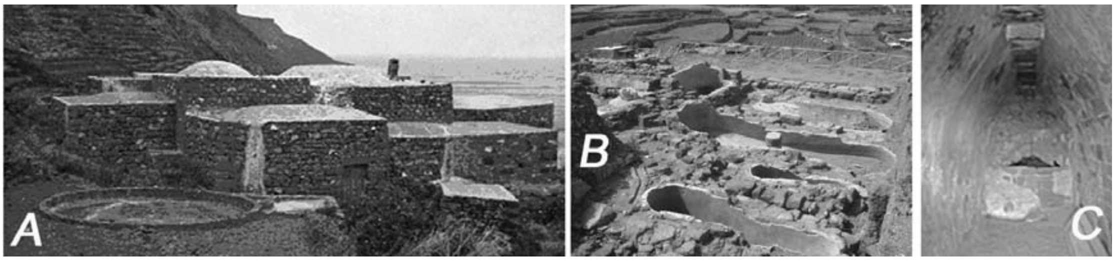

Fig. 2 A – Traditional building of Pantelleria, called dammuso, showing the rain water supply system, B – bathtub Punic cisterns, C – trapezoidal Punic cistern

carved in a rock ashlar. The Punic cisterns have elliptical plane (bathtub shaped, Fig. 2B) or a rectangular plane and a section with a trapezoidal upper part, Fig. 2C.

The samples, taken from both sites, were covering, lining and joint mortars, according to their role in the construction. The kind of aggregates characterizes each mortar, i.e. crushed brick, red and white tuffs, sedimentary and crystalline sands.

Materials were analyzed by means of X ray diffraction (XRD), optical microscopy (OM) and simultaneous thermal analysis (STA). The latter proved to be the most powerful tool to evaluate the degree of hydraulicity of a mortar, which is strictly related to its permeability. The results were processed with the aim of finding out some correlation between the hydraulicity of mortars and their textural characteristics.

Experimental

Materials and methods

Samples from each element of the water systems were taken to acquire a collection of the different typologies of mortars employed for both structural and lining purposes. In the following letter ‘C’ in the sample labels refers to Aquae Corneliae and letter ‘P’ to Pantelleria. Furthermore, each sample is identified by a number followed by the specification of the mortar type in relation to the aggregates:

cp=crushed bricks
rt=red tuff
wt=white tuff
pz=pozzolana
wl=carbonate-quartz sands

and a symbol that indicates the role in the construction:

^external covering or roof
floor
* lining
° joint

The crushed brick mortars are suitable lining materials for ancient cisterns and channels, sometimes overlapping layers of different mortars. The lining of roofs of dammusi is made of red tuff mortar, sometimes replaced by white tuff, covered by a thin layer of air hardening mortar, like samples P11Awl$_1$ and P11Awl$_2$, the first lacking in aggregates, the latter containing crushed calcareous rock aggregates. Their role is smoothing of the roof surface and preserving the inner layers, according to the building tradition of the island. In the joint mortars of Aquae Corneliae the aggregates are carbonate-quartz sands, the binder is slaked lime, sometimes mixed with charcoal slack and/or pozzolana, in order to improve their strength.

Materials characterization was performed by means of:

- XRD: X ray diffraction, for the analysis of powdered samples to identify the crystalline phases;

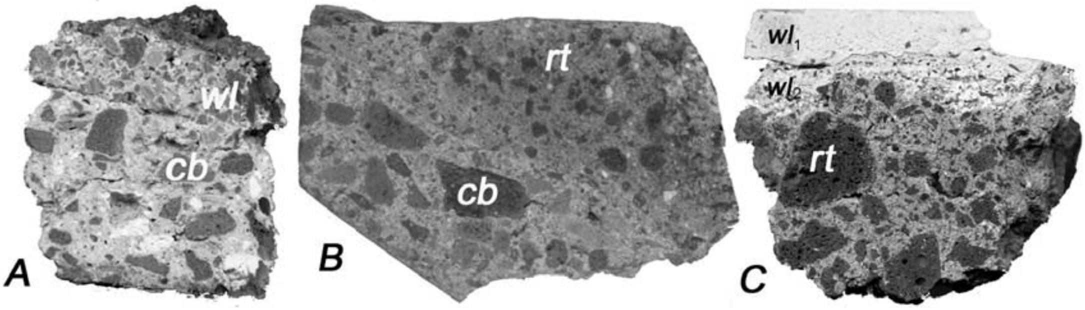

Fig. 3 Samples of different types of mortars: A – Aquae Corneliae, sample C6 from the lining of the channel: wl – slaked lime mortar and cp – crushed bricks mortar; B – Pantelleria, sample P9 from a Punic cistern: cp – crushed brick mortar and rt – red tuff mortar; C – Pantelleria, sample P11A from a roof: wl$_1$ – and wl$_2$ – layers of air hardening mortars on the rt – red tuff mortar

- OM: optical microscopy, both in reflected and in transmitted light on thin-sections, to analyze composition and texture;
- STA: simultaneous thermal analysis, TG/DTA, to evaluate the rate of hydraulicity.

Tests were carried out in a simultaneous thermal analyzer Netzsch STA 409, heating rate 10°C min^{-1} in the range 30–1000°C. The samples of mortars were mildly pounded in order to separate binder from aggregates, then sieved at 53 μm to enrich them into the binder. Before STA tests the powders were dried at 70°C for at least 24 h.

Results were processed measuring the mass loss in four consecutive temperature ranges:

- 30–120°C hygroscopic water;
- 120–200°C water from hydrated salts;
- 200–600°C water structurally bound to hydraulic components;
- >600°C carbon dioxide from carbonates decomposition.

It is appropriate to underline that such a sharp distinction of temperature ranges is just a working assumption, even though widely used and validated in the literature [3–6].

As a final consideration, the size of the samples was limited by the fact that they were collected from archaeological artefacts. After separating a suitable portion for thin sections, the residual sample was further reduced by sieving to increase the binder fraction for thermal analysis. For this reason it was not possible to duplicate the tests, in order to evaluate

Table 1 Composition of the samples, evaluated by means of XRD and OM. C=Aquae Corneliae; P=Pantelleria

Sample ^=roof, *=hydraulic artifact, °= joint mortar, # =Floor	XRD: Crystalline components	O.M.: Aggregates composition	
		Temper in the fictiles (cp)	Sands or crushed rocks
C3* cp	Cal, Qtz, Fs	Cal, Qtz, Fs, Hem,Di	pz, qza, ca, ar
C3* wl	Cal, Qtz, Fs, Dol		qza, ca, Qtz, Fs, pz, chsl
C4* wl	Cal, Fs		Qtz, ca, pz, Px, chsl
C5^ Cp	Cal, Qtz	Qtz, Fs, Hem, Di,Mi	Qtz, ca
C6* wl	Cal, Qtz, Fs		qza, ca, Qtz, eir
C6* cp	Cal, Qtz, Dol	Qtz, Fs, Hem, Di, Mi	qza, Qtz, vegetal fibres, pz
C7^ wl	Cal, Qtz		qza, ca, Qtz, chsl Aug, Ms
C10° wl	Cal, Qtz, Fs		qza, ca, eir, iir, Qtz,Fs, Cal, pisolithi
C11° wl	Cal, Qtz		eir, iir, qza, ca,Qtz, chsl
C12*cp	Cal, Qtz	Qtz, Mi, Fs,Hem,Di, Cal, Ch	Qtz, ca,chert,
C13°wl	Cal, Qtz		ca, sb,Qtz, pz,carb
C14*wl	Cal, Qtz		
C14*cp	Cal, Qtz, Fs	Qtz, Cal, Fs, Di, Hem, Mi, Ch	Qtz, ca
C15° wl	Cal, Qtz, Fs		Qtz, ca,pz, chsl
P3^ wt	Cal, Dol (tr),Mgs(tr),An		pz, O, Pl, Ol, Px, E
P5*cp	Cal, Dol, Qtz, Fd, Hl,Di	Qtz, Cal, Di, Fs, Mi, Hem	
P8^rt	Cal, Dol, Aor		vt
P9*cp	Cal, Qtz, Dol(tr), Aor	Qtz, Cal, Fs, Cpx, Di, met, cb	
P9*rt	Cal, Qtz, Dol (tr), Aor		vt
P11A^wl$_1$	Cal, Mgs, Dol		ca
P11A^wl$_2$	Cal, Mgs, Dol, An		Qtz, Cal, vt, Fs, px
P11A^rt	Cal, Dol, Mgs (tr), An		vt
P11B#cp	Cal,Qtz, An	Qtz, Cal, Px, Di, Fs, Mi, Hem	
P11B# rt	Cal, Dol (tr), Aor		vt
P11C*cp	Cal, Qtz, Fs	Qtz, Cal, Hem, Di, Px, Mi, Fs	
P11D* cp	Cal,Qtz, An	Qtz, Cal, Hem, Di, Px, Fs	

Minerals Cal=calcite, Dol=dolomite, Mgs=magnesite, An=anorthite, Aor=anorthoclase, Aug=augite, Qtz=quartz, Hl=halite, Hem=hematite, Di=diopside, Fs=feldspar, Px=pyroxene, Cpx=clinopyroxene, O=orthoclase, Ol=olivine, E=enigmatite, Pl=plagioclase, Mi=mica. Crushed rocks: qza=quartzarenite, ca=carbonate rocks, eir=effusive igneous rocks, iir=intrusive igneous rocks, ar=arenite, pz=pozzolana, vt=vitrophyrous, sb=siliceous bioclasts, cb=calcareous bioclasts, met=metamorphites, chsl=charcoal slack; Ch=chamotte. Mortars: cp=crushed bricks mortar, rt/wt=red/white tuff, wl=slaked lime mortar their reproducibility, as usual in most of papers related to historical mortars.

Results and discussion

The whole set of samples is characterized by high heterogeneity, due to the fact that they were prepared for different purposes, in different ages, according to different building traditions. Thus, the mortars have been classified, on the basis of aggregates observed by OM, in the following groups: a) crushed brick mortars; b) tuff and pozzolanic mortars; c) slacked lime mortars. It is worth specifying that the pozzolanic mortars are very poor in pozzolana fragments and that both in pozzolanic and in slaked lime mortars the prevailing aggregates are clasts of sedimentary or crystalline origin.

The mineralogical-petrographical characteristics of samples are summarized in Table 1. Thirteen kinds of different fictiles have been recognised in the crushed brick mortars from Aquae Corneliae, nine kinds in the samples from Pantelleria, on the basis of the following characteristics: a) the bulk colour; b) the ratio temper/matrix/pores; c) the minerals present in the temper. Such compositional heterogeneity is mainly related to the different aggregates used in the mortars.

The results of thermal analysis are summarized in Table 2. It is worth reminding that, according to the approach used in the literature [3, 4], the hydraulicity of mortars by thermal analysis is evaluated on the binder fraction, as already reported in the experimental. This reduces the effect of the grain size distribution of the aggregates and stresses the influence of their reactivity *vs.* lime. This is a promising starting point for a clustering procedure of mortars according to the typology of aggregates. According to [3–5, 7] in Table 2 the ratio between the mass loss percentage CO_2 above 600°C and the mass loss percentage H_2O in the range 200–600°C has the meaning of the inverse of the rate of hydraulicity. In Fig. 4 the ratio CO_2/H_2O is plotted *vs.* CO_2. The representation of data according to such coordinates places all of the samples, but two, in the field of high hydraulicity,

Table 2 Results of STA on samples sieved at 53 μm and dried at 70°C

Sample	mass/mg	<120°C/mass%	120–200°C/mass%	H_2O/mass% 200–600°C	CO_2/mass% >600°C	CO_2/H_2O
C3* cp	100.4	1.2	2.0	7.6	19.5	2.6
C3* wl	100.5	1.0	1.8	6.4	15.7	2.5
C4* wl	101.4	1.8	3.1	6.1	16.2	2.6
C5^ Cp	102.3	0.6	1.2	4.7	31.0	6.6
C6* wl	101.5	0.6	0.6	4.7	26.0	5.5
C6* cp	103.2	1.2	1.9	7.7	14.1	1.8
C7^ wl	104.1	0.8	1.3	6.1	26.5	4.3
C10° wl	100.2	0.2	0.8	5.2	21.7	4.2
C11° wl	100.3	0.4	0.6	5.0	25.3	5.0
C12*cp	101.5	1.2	1.8	7.1	18.1	2.5
C13°wl	101.7	1.0	1.2	4.7	19.8	4.2
C14*wl	100.9	1.4	1.0	3.4	20.6	6.1
C14*cp	100.9	1.2	1.8	4.8	22.4	4.7
C15° wl	104.6	1.3	2.1	6.3	16.2	2.6
P3^ wt	99.5	0.4	0.6	5.2	11.6	2.2
P5*cp	99.6	1.2	2.0	8.4	11.0	1.3
P8^rt	100.0	0.4	0.8	5.4	15.2	2.8
P9*cp	101.6	0.8	1.4	6.7	17.1	2.6
P9*rt	101.2	0.8	1.6	5.9	14.8	2.5
P11A^wl1	101.4	0.4	0.4	0.8	40.2	50.9
P11A^wl2	101.0	0.8	0.8	1.6	37.2	23.5
P11A^rt	101.8	0.5	0.1	3.0	15.9	5.4
P11B#cp	99.3	0.4	0.6	3.8	17.3	4.5
P11B# rt	104.0	0.4	0.4	3.1	16.3	5.3
P11C*cp	101.1	1.4	1.8	6.3	16.2	2.6
P11D* cp	97.0	0.4	1.4	4.5	13.2	2.9

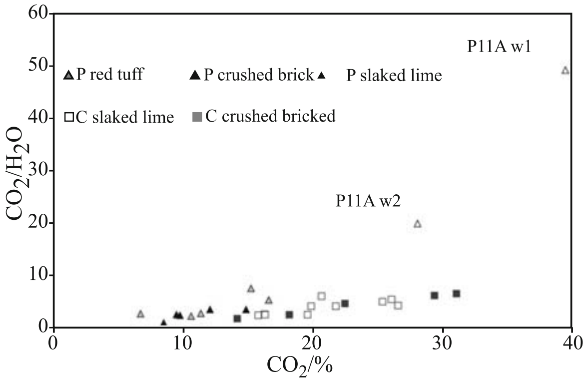

Fig. 4 STA data represented according to [3]

i.e. in the lower part of the plot. It is worth observing that the samples from Pantelleria are grouped in the left part of the diagram, corresponding to low content of carbonates, whereas the samples from Aquae Corneliae are definitely shifted to the right. This is consistent with the different nature of the aggregates, identified by OM and XRD, which were available in the two sites according to the different geology. As for the two mortars represented by hollow triangles, they fall correctly in the upper part of the diagram, as they are air hardening systems.

In order to find out some correlation between the hydraulicity of mortars and their textural characteristics, the thin sections were observed in polarized light to evaluate the aggregate/binder/pores ratio, according to Italian Standards on historic mortars [8], which suggest the use of visual comparators [9, 10] for areal evaluation of such textural components. It is worth specifying that, according to this procedure, only the macroporosity is taken into account.

On the basis of the texture of mortars, as observed in thin section, the ternary diagrams shown in Fig. 5 were drawn. It can be noted that the crushed brick mortars from Pantelleria, Fig. 5A, have a porosity lower than 15% and an almost constant value of the aggregate/binder ratio, close to 1.5 in 80% of the cases. The lowest value is 0.7 and refers to a fragment of the lining of a cistern.

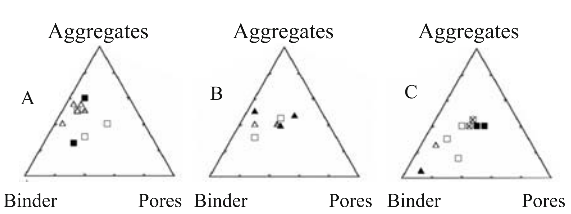

Fig. 5 Representation of mortars on triangular diagrams in terms of values of the main textural parameters, i.e. the abundance of aggregate, binder and pores: A – crushed brick mortars; B – tuff and slacked lime mortars with minor presence of pozzolana; C – slacked lime mortars. Samples C4 and C15 are present both in B and C diagrams because of their low content of pozzolana. ■ – C roof, × – C joint mortar, □ – C hydraulic artifact, ▲ – P proof, ▲ – P floor, △ – P hydraulic artifact

The values of aggregate/binder ratio for crushed brick mortars of Aquae Corneliae are more dispersed, varying from 1.25 to 1.5. In particular C3 and C5 are very similar to the Pantelleria materials, while the samples from the inclined plane and from the internal lining of the channel, C12 and C6, are richer in binder.

Figure 5B shows the textural characteristics of tuff mortars from Pantelleria and pozzolanic mortars from Aquae Corneliae. Red and white tuff mortars have a texture similar to the crushed brick ones, but sometimes are more porous.

The texture of slacked lime mortars is represented in Fig. 5C. The two samples from Pantelleria have a low porosity and are rich in binder. In fact, their role in the roofing of dammusi was to give a smooth finishing, without any problem of permeability. The mortars of Aquae Corneliae are very similar each other, most of them clustered in the central area of the diagram. Different properties are shown by two samples: C4, the lining of a tank reused continuously since the mediaeval time, has a porosity comparable to the cluster but is richer in binder; C6, a mortar layer overlapping a crushed brick mortar, has comparable porosity associated with a low content of aggregates.

In relation to the role of each kind of mortar in the construction, correlations between textural characteristics and hydraulic properties have been investigated. In the following a portion of the diagram in Fig. 4, containing all points relative to each specific role, is associated with the corresponding plot of CO_2/H_2O versus porosity as evaluated by optical microscopy.

As for the lining mortars designed for being in contact with water, the plot, Fig. 6B, shows good clustering of data relative to the mortars of Pantelleria, that are the less porous ones; more dispersed are the values of Aquae Corneliae samples. All of the mortars from both sites are made of crushed brick mortars, with the exception of P9, C14, C6 and C4, which are, but the latter, made of two layers. Sample P9 is made of a red tuff mortar, applied on the cistern walls, overlapped by a crushed brick mortar, having the same hydraulicity and porosity. Samples C6 and C14 are slacked lime mortars with a medium high hydraulicity, associated with a crushed brick mortar, having the same porosity but higher hydraulicity. Sample C4 is the lining of a cistern, probably introduced in the aqueduct in the medieval age.

As for roofs and coverings, Fig. 7, the differences between samples seem to be related to the functional requirements of the mortar. The impermeability of roofs of dammusi plays a major role both for the comfort of the house and for the efficiency of rain water supply system, this corresponds to the high

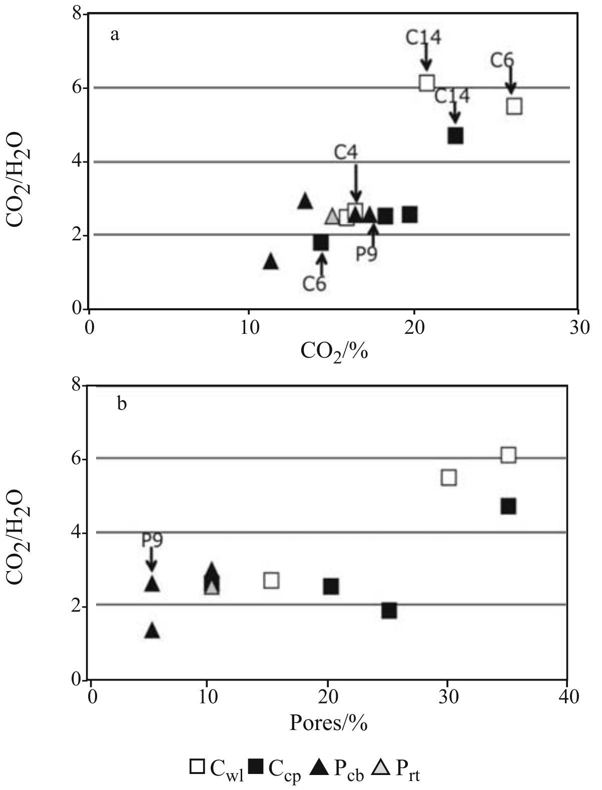

Fig. 6 Porosity of lining mortars as related to hydraulicity

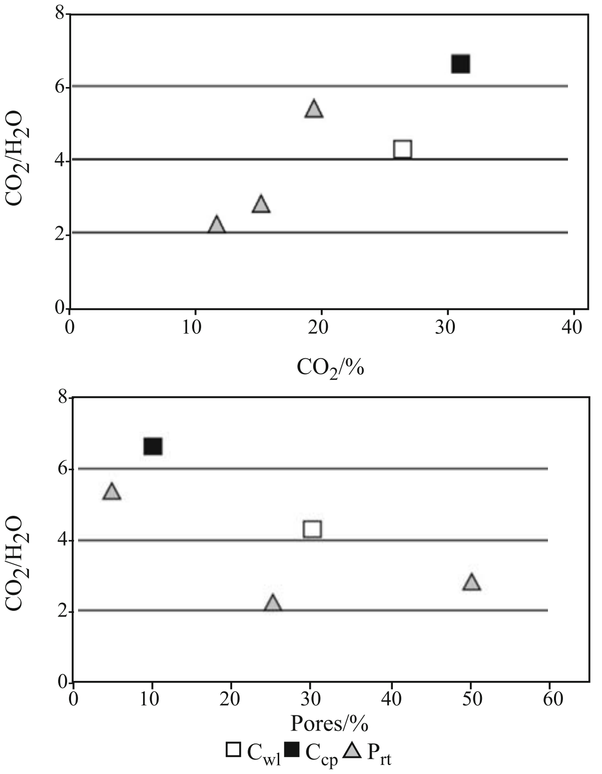

Fig. 7 Porosity of roof and covering mortars as related to hydraulicity

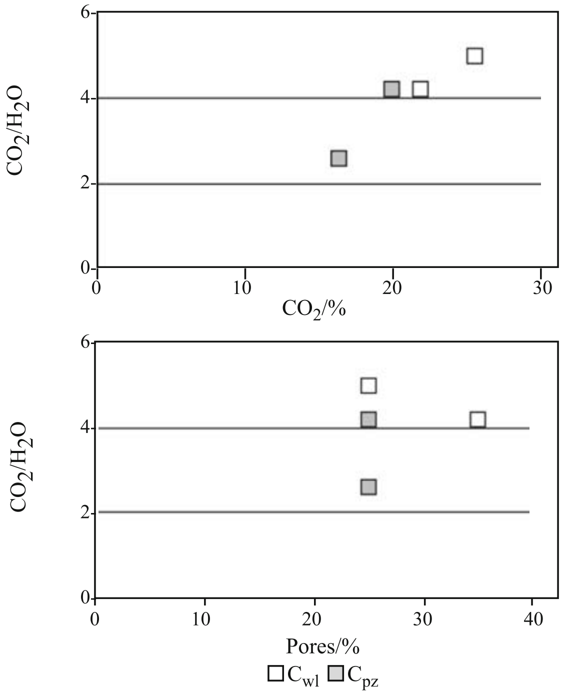

Fig. 8 Porosity of joint mortars as related to hydraulicity

hydraulicity of the red tuff mortars of Pantelleria. The Aquae Cornaliae samples, two made of slaked lime and one of crushed brick mortar, had the simple role of protecting the walls of the aqueduct from environmental attack and this corresponds to lower hydraulicity index. The correlation of hydraulicity with porosity is complicated by the presence of red tuff aggregate, which introduces a considerable amount of bubbles. This gives the advantage of reducing the weight of the roof and, on the other hand, produces a higher specific surface of the reactive aggregate, thus contributing to increase the hydraulicity of the mortar.

As for the joint mortars from Aquae Corneliae, Fig. 8, some show higher hydraulicity because of the presence of a small amount of pozzolana, whereas the porosity varies in a limited range between 25 and 35%. The use of pozzolanic aggregate can be related to the requirement of both strength and durability due to the structural function of such mortars in the aqueduct.

In Fig. 9 the ratio CO_2/H_2O is represented for different kinds of mortars by the height of truncate cones, so that the size of the top section is proportional to the hydraulicity of the mortar. The lining mortars have approximately the same hydraulic properties both in Aquae Corneliae and in Pantelleria, the external coverings of Aquae Corneliae have a lower hydraulicity than the roofs of Pantelleria, the hydraulicity of joint mortars of Aquae Corneliae is similar or slightly higher than the external coverings.

Some interesting trends can be observed for crushed brick mortars by plotting the ratio CO_2/H_2O

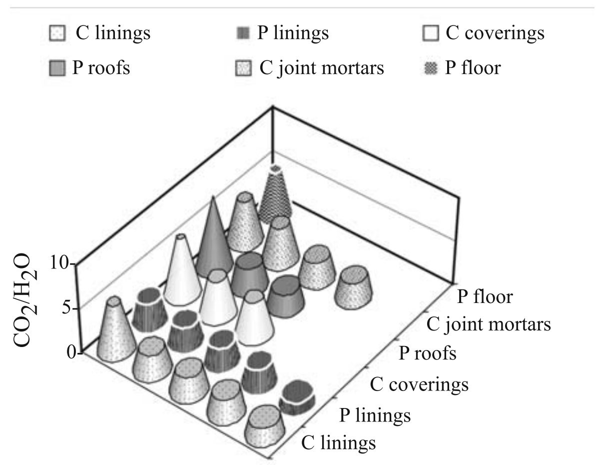

Fig. 9 Hydraulicity of mortars as related to their role in the construction

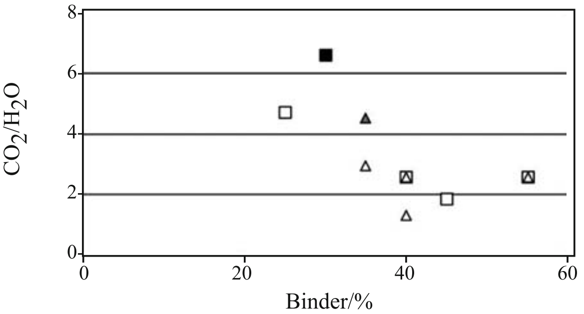

Fig. 10 Hydraulicity of crushed brick mortars as related to the abundance of binder. ■ – C roof, □ – C hydraulic artifact, ▲ – P floor, △ – P hydraulic artifact

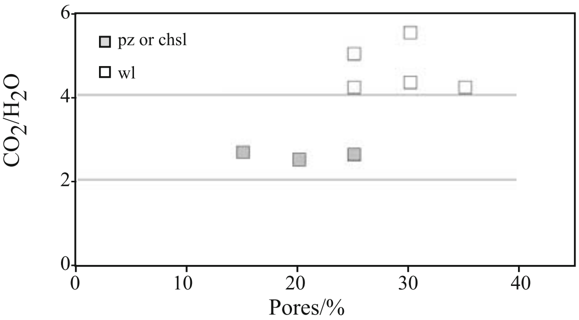

Fig. 11 Porosity of slaked lime mortars as related to hydraulicity (legend as in Table 1)

vs. the percentage of binder, as evaluated by optical microscopy, Fig. 10. On increasing the binder content the hydraulicity of the mortar increases.

For the samples of slaked lime mortars from Aquae Corneliae, plotted in Fig. 11 as the ratio CO_2/H_2O *vs*. porosity, the presence of pozzolana or charcoal slack produces higher hydraulicity and lower porosity.

Conclusions

Simultaneous thermal analysis proved to be a powerful tool to evaluate the degree of hydraulicity of a mortar, which is strictly related to its permeability and strength, both characteristics being critical for the correct running of hydraulic constructions, such as aqueducts or water storage systems.

In the past hydraulic mortars were prepared by mixing slaked lime with reactive aggregates, mainly pozzolana or crushed brick. The expected correlation with textural characteristics is that on increasing the degree of hydraulicity the porosity decreases whereas the strength increases, but, according to the results presented in this paper, sometimes the effect of the aggregates addition is not so plain. In particular, the role of total porosity, as evaluated by OM, needs further investigation, using mercury intrusion porosimetry and helium picnometry. For the tuff mortars it is necessary to distinguish between the binder porosity and the aggregate porosity, as the latter plays opposite roles: a) on one side it increases the total porosity; b) on the other side, it increases the specific surface and, thus, the reactivity of the aggregate with the slaked lime, inducing higher hydraulic properties in the mortar.

The improvement in clustering of hydraulic mortars, according to the modified coordinates proposed in [11] probably could produce better correlation between the textural properties and the hydraulicity of the mortars. Unfortunately such procedure for processing the results of simultaneous thermal analysis can be applied only after conditioning all of the samples at the same temperature in equilibrium with air at 99±1% relative humidity, and this was not the case. Therefore, more samples of hydraulic mortars will be collected from archaeological sites and submitted to the analytical procedure proposed in [11], both to validate the method for evaluating the degree of hydraulicity and to verify the correlations between texture and hydraulicity outlined in this work.

As a final comment, the combination of simultaneous thermal analysis with mineralogical and textural characterization of samples gives interesting information on the use of different mortars in relation to their role in the construction. For example, the samples collected in the two archaeological sites show that the Punic and Roman techniques of building hydraulic systems had marked similarities. Two overlapping lining levels are present: i) a crushed brick mortar in contact with water; ii) beneath it, a mortar with aggregates of the same composition of rocks outcropping in the area, i.e. tuff in the volcanic island of Pantelleria and sedimentary and crystalline rock clasts in the northern Sicily. The results give evidence of the thorough knowledge of materials science, even if based on empirical observation, that allowed the 'aquarius', i.e. the hydraulic engineer, to build more than 2000 years ago such durable and effective water systems.

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