From Vitruvius' ceramic powder additives to modern restoration

Dos aditivos de pó cerâmico de Vitrúvio à restauração moderna

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Abstract
The text aims at giving a general view of the use of lime mortars additivated with ceramic powder taking advantage of its pozzolanic reactions. It emphasizes the main explicit references of this technique in the ancient writers, starting from Vitruvius and going through important theoreticians of the Renaissance, until it reaches the military engineers of the 17th and 18th centuries, particularly the Portuguese engineers who had a strong influence in the overseas constructions techniques. Some mistakes in the interpretation of these texts regarding the properties attributed to the addition of ceramic powder in lime mortars are also mentioned.
The continuation of the work refers to the description and commentaries of the tests and laboratory observations carried out on the additivated mortars in question. Among these are highlighted the hardening time, the mechanical resistance (axial compression and traction by diametral compression), water absorption by capillary uptake, total water porosity, accelerated aging in saturated solution of Na_2SO_4, loss on ignition x-rays fluorescence, permeability to water vapor and other procedures that contribute to the evaluation of the behavior of lime mortars additivated with the "cocciopesto" and of the pozzolanic reactions occurring in the material.
As the theory would have no sense if it is not necessarily put in practice, the work ends with the description of the application of the mortar additivated with ceramic powder in a concrete case of restoration, with the description of the obtained results.

Keywords
Ceramic powder pozzolanas; additivated mortars; mortars for restoration.

Resumo
O texto pretende dar uma visão geral do uso das argamassas de cal aditivadas com pó cerâmico e as vantagens conseguidas através das reações pozolânicas obtidas. Destacam-se as principais referências explícitas desta técnica nos antigos autores, principiando por Vitrúvio, passando através dos mais importantes teóricos do Renascimento até chegar aos engenheiros militares dos séculos XVII e XVIII, particularmente os engenheiros portugueses que tiveram forte influência nas técnicas construtivas do Além-mar. Alguns equívocos na interpretação dos textos antigos, em relação às propriedades do aditivo de pó cerâmico são também, apontados.
O trabalho, em seguida, trata da descrição e comentário dos ensaios e observações de laboratório, levados a efeito nas argamassas aditivadas em questão. Entre estes destacam-se o tempo de endurecimento, a resistência mecânica (compressão axial e tração por compressão diametral), capilaridade ascendente, porosidade total acessível à água, envelhecimento acelerado em solução de Na_2SO_4, perda ao fogo, fluorescência de raios-x, permeabilidade ao vapor d'água e outros procedimentos que contribuem para a avaliação do comportamento das argamassas de cal aditivadas com "cocciopesto" e as reações pozolânicas acorridas no material.
Como a teoria não teria sentido se não tivesse que ser necessariamente materializada na prática, o texto termina com a descrição da aplicação da argamassa aditivada com pó cerâmico, em um caso concreto de restauração, com a descrição dos resultados obtidos.

Palavras-chave
Pozolanas de pó cerâmico; argamassas aditivadas; argamassas para restauração.

Introduction and history

The ceramic powder additivated mortars are not novelty in the study of historic mortars. In the Italian language they became known by the word *cocciopesto*, familiar to every specialist in this matter. What still remains to be studied further are some of the properties of this additive, especially the pozzolanic reactions that the mortar goes through with this mixture, trying to understand the empiric observations done by the ancient authors, from Vitruvius to military engineers of the 18th century. In the case of Brazil, its employment in the constructions is studied very little, even if its application was abundant, as it has been observed in the sampling and material analyses of old buildings, especially fortresses.

What will be mentioned now is in great part known by the history of architecture and archeology professionals, but it is worth mentioning because these are important pieces of information that lead to varied directions. It is not as much a systematic investigation of every source that refers to pulverized ceramic additivated mortars, as it is a collection of some examples throughout different times and construction professionals about the various qualities that were attributed to the additive. To close this text some observations will be presented regarding laboratory tests of old mortar samples containing brick powder and also of new ones, trying to understand their properties.

As usual, the oldest reference to this matter comes from the master Vitruvius in at least two segments of his text: *Also in the case of river or sea sand, if anyone adds crushed and sifted potsherds, in the proportion of one to three, he will produce a blending of material which is better for use* (Book II) [1, v. 1, p. 97]. Later on he said: *I have described how plastering is to be done in dry places: I will now explain how stucco is executed in damp places so as to avoid blemishes. First of all, in the chambers situated at ground level, to the height of about three feet* [Roman foot = 0,29 cm] *from the pavement, rough-cast made of powdered earthenware is applied and then the surface is smoothened, instead of a lime and sand mortar with powdered ceramic so that this part of the plaster may not suffer from damp* (Book VII) [1, v. 2, p. 97]. It is clear in the teachings that this is not, as many people say it is, a mortar to avoid the passage of water, but a mortar that avoids damages on the plaster because of its resistance and porosity. What is more, it was with this additivated mortar that the *opus signinum*[1] was fabricated, which was used as finishing without the need for paint and consequently letting the wall breathe freely. Its behavior would be comparable to one of a "sanitation" mortar, as it is known in the present, and not to an impermeable mortar that would not work well. Plinio, likewise, makes references to the ceramic powder mortar when he says: *Constructions exposed to humidity or built up in places where it may be affected by the vicinity of the sea, could, with advantages, be covered with a coat of plaster made with crushed ceramic* [2, p. 139]. Both teachings suggest the property of making mortars endure better the effects of moisture, regarding the durability towards the tensions of crystallizations of soluble salts. In practice it is easy to notice, especially in the archeological remains of Roman baths, the presence of lime plasters additivated with ceramic powder (Fig. 1). It is important to emphasize that, in spite of Vitruvius being the first author whose written references on this subject made it to our times, the use of crushed ceramics as additive was employed before his time by other more ancient civilizations, as the Greeks [3, p. 422].

In medieval times, when Villard de Honnecourt, in his well known sketches and notes book, gives us a recipe for an impermeable recipient, he specifies the use of oil besides the brick powder. The oil produces hydrorepe-

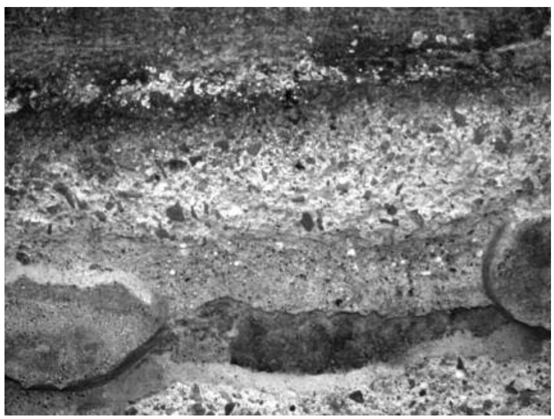

Fig. 1 Ceramic additivated mortars in Stabian baths in Pompey, Italy.

1 Done as in Signia or from this town.

lence when added to the lime mortar[2]: *You take lime and crushed "pagan" tiles in the same proportion then you add a little more "pagan" tiles until its color surpasses the original color. Add to this cement some linseed oil so you can make a recipient to contain water[3]* [4, p. 100].

In the same manner, in the beginning of the Renaissance, the qualified teachings of Leon Battista Alberti came, in his *De re ædificatoria (L'Architettura)*, affirming: *If then you add one third of crushed bricks, it is the general opinion that this mixture will become much more tenacious*. It is important to observe that, once again, the objective was to bring more resistance and not impermeability. In fact, the original text reads *multo tenaciorem* [5, p. 188]. Later on, the experienced Pietro Cataneo, describing a special pavement, which must have been the one named "Venetian", recommends adding to the lime an identical amount of crushed tiles and also iron scoria. It is possible to conclude that these additives must harden the mixture because they bring hydraulic properties, making the pavement more resistant [6, p. 35]. Good references to the ceramic powder mortar were also made by Scamozzi, in some parts of his text, exemplifying their use in the old Roman theater of Vicenza [7, p. 297]. The hydrorepelence was obtained with a treatment of animal fat or vegetable oils, such as linseed oil.

The European military engineers of the 17th and 18th century, that many times had to deal in the fortresses with the fury of the cannons, with walls leaning against embankments or in the vicinity of water, made frequent use of this additivated mortar, as reported by Knight de Ville: *In Palmanova there is a mixture with a cement that, when dry, creates a body that resists well to bad weather and when it is hit by the cannon discharge it is not ruined[4]: This mortar could be made of crushed bricks, of lime and of crushed marble* [8, p. 89]. The teachings of the French, Italian and Dutch military engineers were source of knowledge for the Portuguese engineers; among them was Azevedo Fortes, the most well known Portuguese writer in the art of Fortification of the 18th century, who taught us: (…) *however, if it is near a river or for a water ditch it is necessary to protect the facing with good regular masonry in its beds and joints, and settled with lime and sieved tile powder, and also with bitumen if necessary* [9, p. 285]. So, his disciples and followers who worked in Brazil or taught in our military classes certainly applied these techniques of production of mortars, as observed in some of the documents and in several examples found in monuments through the investigation of their mortars (Fig. 2).

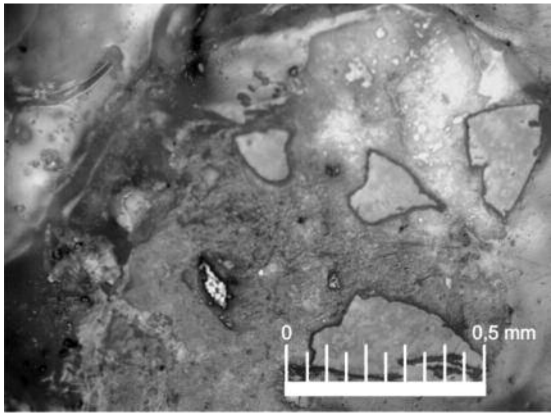

Fig. 2 Microphotography of a polished section of mortar with ceramic powder from the S. Alberto Fortress in Salvador.

Besides the European texts about construction, that were practically unanimous in recommending the good properties of lime mortar additivated with ceramic powder a "common sense", as Alberti affirmed, the following paragraphs will now address what was written and observed about this matter in Brazil, while colony of Portugal and even afterwards. This technical production was restricted among us to the military engineering profession, which was very important in the construction of the Portuguese America, since the architects only appeared with the French Mission brought by D. João VI, after 1808. The first known reference is from the Head Engineer of Brazil, Frias da Mesquita, who worked here the first 30 years of the 17th century. This document is a report about the fortress of the Three Wise Kings in the city of Natal [10]. In two segments of this document, he talks about a material called *tetim* or *tettim* that may be the ceramic powder. He says: (…) *and also the brick must*

[2] In Portugal and in Brazil it was common the employment of oils in mortars to bring hydrorepelence (and not to increase its resistance, as it is popularly affirmed).

[3] This information is found in the image 21, where the "Fortune Wheel" is represented.

[4] For this reason, the Field Master Engineer Miguel Pereira da Costa, who was born in Portugal but worked in Brazil, always used the "cocciopesto" in the parapets of the fortresses that he built..

be of good clay and well burned, and with no trace of salt water, and the "tettim" very well crushed, the mortars thoroughly rested for several days (…)[5] and also: *protected on the outside with good mortar and "tettim" for its better defense against humidity* (...) [10]. In this case, besides a better resistance, the mixture was hydrorepelent due to the presence of oil, a recipe similar to those left by Villard de Honnecourt and other writers.

The specific texts about construction are very rare in Colonial Brazil. One of them is a military engineering treatise written in Pernambuco by the Field Master Diogo da Silveira Velloso [11], a manuscript inedited until recently[6], in which the following reference related to the Vitruvian teachings is found*: It helps a lot the strength of the work (says Vitruvius) mainly in the foundations, and in damp places, the mixing of lime with crushed ceramics, pieces of tiles and bricks, which because of its dryness and roughness dries the work quickly and makes it harder.* It is interesting to observe the curious explanation for a pozzolanic reaction!

The examples in which the cocciopesto mortar is found are abundant and some samples from monuments of the city of Salvador and from other parts of Brazil have been known and analyzed. It is possible to find this material, for example, in the parapet of the walls of the fortresses of Barbalho and of São Pedro, or in the cannons platform of the little fortress of Santo Alberto (Fig. 2), when the excavations of its rampart were carried out. The material was found in very good conditions and it was extremely resistant. So, it is possible to arrive to the conclusion that this knowledge would be very important in modern restoration works, namely knowing more about the properties and better proportions of the mixture.

About recipes of ancient mortars in general, including mortars additivated with ceramic powder, a very interesting text of historical investigation by Carla Arcolao [12] was produced and deserves a special reference.

Testing and analysis

This way, the NTPR (Núcleo de Tecnologia da Preservação e da Restauração) has been trying, through systematic investigation, to better understand the behavior of the pozzolanic properties resulting from the addition of ceramic powder to mortars. About this matter, some observations will be made, many of which are already known by those who study the subject, but they may be useful as confirmation and eventually to bring some doubts to be discussed. The information obtained through tests and the observations made will be summarized so as to be compatible with the space available for this text.

Initially it is necessary to declare that the motivation of these studies is not merely academic or cultural; in this case, searching to rescue the memory of the construction science, it goes further. The constant proximity to the project and the execution of the monument restoration has demonstrated the need to find alternative technologies for its operational problems. The vastness of the Brazilian territory and, in most cases, the little resources available for intervention procedures to be carried on monuments, make necessary, to avoid the use of industrialized products, some of them imported, to face the restoration problems with solutions which are simpler or identified more closely to cultural traditions. Certainly in the field of "sanitation" mortars there are already a lot of commercialized products which are efficient. Some of them are imported, others are made in Brazil. However the final price of these plasters is around five times more than that of traditional mortars, as it has been calculated. This leads us to search for alternatives, especially in the covering of large areas.

The first question to be presented is the presumed impermeability of the mortar with ceramic powder. Really, none of the cited authors makes any reference to this impermeability – when desired they recommended the addition of oil. The tests that will be commented later on show that the mortars additivated with ceramic powder are in fact more porous, more permeable to water vapor, with a better capability of absorption of water under low pressure and present other indicatives of a "sanitation" mortar and not those of an impermeable mortar.

By definition a pozzolana (in this case the ceramic powder) must be finely pulverized to become more reactive. On the other hand, this is not recommended textually in many ancient writings and the workers did

[5] This waiting period could not be long because after 5 or 6 days the mixture could have hardened, as it was verified.

[6] Velloso's treatise was transcribed and edited with the authorization of Ajuda's Library and commented by the author of this text.

not always notice the problem, considering it is possible to find, in many Roman monuments and even in Brazil, mortars with ceramic coarsely pulverized in the mixture, with fragments superior to 4 mm (Fig. 1). Vitruvius and Azevedo Fortes, however, suggest the sieving of the material, which presumes reduced grain sizes. Frias da Mesquita talks about "tettim" very well crushed which leads us to the same conclusion. In Brazil, the theme of the size of the grain has already been explored by Nappi and Meyer [13], but other observations were performed, arriving to numbers compared among several forms of mixture. Notice, in Fig. 3, that the samples intended by CVN additivated with coarse pulverized ceramic (retained in the sieve # 12 ASTM, 1.68 mm or 10 mesh) do not increase the resistance, as expected, but on the contrary, may induce a slight reduction of the latter.

It is important to clarify that the ceramic material employed in the observations had the same origin. Initially it was used industrially burned material, but due to the knowledge of the local reality in which unfortunately there is no burning temperature control, it was decided to obtain pre-burned industrial material, to be burned in the lab under controlled temperature. It is equally important to emphasize that, even though harder to obtain, the crushing of the ceramic powder was made in a porcelain pounder, when the samples that would be chemically analyzed were made. This procedure was done to avoid iron contamination originated from the iron balls grinder available for the operation.

Another subject that deserved observation was the predominant composition of the clay mineral of the argillaceous material contained in the ceramic. It is obvious that the pozzolanic reactivity depends on its mineral components. It was observed, for example, that the white ceramic in which the clay mineral kaolinite was abundant has a much differentiated effect from that of the red ceramic where the iron oxide is more abundant. This phenomenon was emphasized equally by Silva et al. [14, p. 10]. Apparently the iron works as a material that facilitates the pozzolanic reactions. This is a matter to be discussed by chemists. It is also important to clarify that with the ceramic powder employed it was not possible to reach the level of 6 MPa,

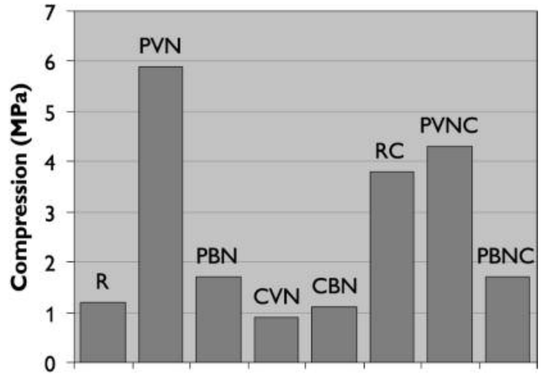

Fig. 3 Comparison of the mechanical strength. Preliminary tests with 1: 2: 1 (lime, sand and ceramic powder) in mass. Samples' codes: R= reference samples; P=powder; V=red; B=white; C= artificially carbonated; N= burned in lab.

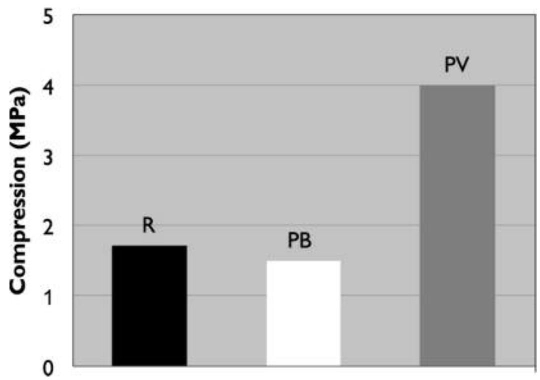

Fig. 4 Comparison of the mechanical strength. Proportions in volume, 1: 2.5: 0.5 (lime, sand and ceramic powder). Samples' codes as in Fig. 3.

(NBR-5751) suggested in the text of Silva et al. [14, p.8].

In Fig. 3, it is observed, for example, that the increase of the resistance to compression of the additivated with red ceramic powder samples (PVN) rose significantly comparing to the samples of sand and lime (R), while the white clay raised this resistance relatively less. In the case of the employment of the rough granulation (CVN and CBN), nothing was added, but perhaps lessened. On the other hand, when the samples are submitted to accelerated carbonation[7] a very interesting phenomenon happens.

[7] The objective need to develop studies, within a time limit, when working with lime mortars, in which carbonation is very slow, led to the need of creating a process of induced carbonation, which does not reproduce exactly the result given by a mortar naturally hardened, but it gives a good idea of the tendency of resistance increase. The process consists of submitting the samples to vacuum, in a special container, and after that letting in pure CO_2 until the regular pressure is stabilized.

The lime mortar (RC) naturally increases its resistance, but the samples additivated with red ceramic powder (PVNC) have a lesser increment of their resistance, compared to the ones cured naturally. This is certainly due to the fact that a part of the Ca, that would originate the calcium silicates along time, is directed to form the $CaCO_3$, which leads us to think that the result of the pozzolanic reactions increases over time. The samples employed in this experiment were tested after 6 months[8] and the carbonated ones received, after removed from the mold, 15 days of carbonation chamber.

After this preliminary evaluation, the subsequent observations were focused on the behavior of the mortars exclusively additivated with the red ceramic powder, with a larger percentage of iron (Fe) in its composition, and fine grain size (< # 20 ASTM, 0.84 mm or 20 mesh). A finer one was not tried because in practice this never happens and the ancient workers would never employ a more pulverized material. It is important to clarify that there is a reason behind the better performance, in terms of resistance, of the additivated mortars initially (Fig. 3) and the results found in the second phase (Fig. 4). In the beginning the scholarship students used the mixture in mass, with a consequent relative increase of lime and of additive (1:2:1, lime+sand+additive, which results in 1:0.28:0.48 in volume approximately) and in the second phase the mixture was adjusted to some historical parameters (1:2:1 in volume)[9] indicated by some writers for coating mortars. However, there is a coherence of results[10] from which it is possible to notice that the increase of the proportion of ceramic powder increases also the mechanical resistance of the mortar. In this matter it must be observed that for the "Venetian" pavements, Cataneo recommended equal parts of lime and ceramic powder, adding to the mixture a small part of iron scoria, also known for its pozzolanic properties.

The tests of the lime mortars' adhesion to the wall are always a problem, when time is limited, because it is not easy to induce the carbonation of the plaster in the wall. In fact, the only tests that presented any result in pull-off tests were the ones performed over mortars with ceramic red powder (between 1 and 0.6 kgf/cm^2), applied directly to a wall of bricks.

The formation of the new silicates, and possibly aluminates, may be observed by comparing the regular lime and sand mortar with the additivated mortars in the graphic of the calcimetry tests (Fig. 5), the diagram of analytical results obtained with the test of loss on ignition (Fig. 6) and the table with the x-rays fluorescence analysis (Table 1).

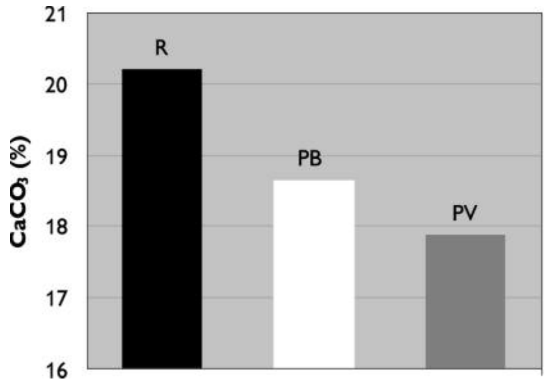

Fig. 5 Comparative graphic of the quantity of $CaCO_3$ contained in the samples. Samples' codes as in Fig. 3.

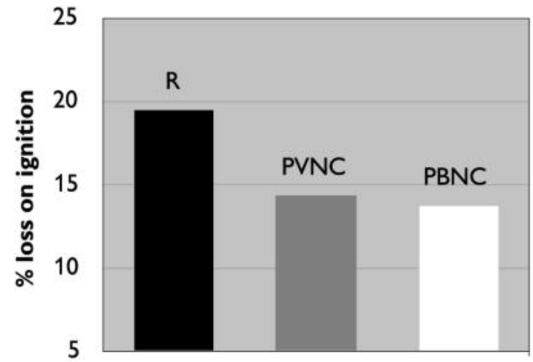

Fig. 6 Results of the tests of loss on ignition. Samples' codes as in Fig. 3.

[8] In this period the total carbonation had not occurred yet, as it was observed through tests with the phenolphthalein reactive solution.

[9] This mixture is suggested by Vitruvius, Alberti and others, but there are some other mixtures, in special mortars, in which the sand is not used, as suggested by Cataneo for the "Venetian" pavement and by Villard de Honnecourt.

[10] Regarding the proportions of lime and sand mortar in the Vitruvian texts there are no doubts: 1:2 when the sand comes from the river or sea and 1:3 when the sand comes from a quarry. On the other hand, the amount of ceramic powder additive is not clear. This subject is dealt with when it is related to the proportion 1:2 and talks about one third (tertia parte adiecerit). Will that be one third of the gross or one third of the sand? One way or another it will always be less than half the sand – 1:2:1 or 1:2:0.7.

Table 1 Comparative table of the mortar samples regarding the formation of oxides.

Analysis	Reference (B) 1 year and 3 months	Red Powder 1 year and 3 months	White Powder 1 year and 3 months
CaO	76.41 %	54.33 %	58.66 %
SiO_2	17.53 %	30.98 %	30.23 %
Al_3O_3	0.29 %	5.89 %	4.95 %
MgO	4.73 %	3.36 %	3.50 %
Fe_2O_3	0.28 %	3.41 %	1.40 %
K_2O	0.04 %	1.05 %	0.51 %
TiO_2	0.18 %	0.60 %	0.50 %
SO_3	0.34 %	0.08 %	0.13 %
ZrO_2	0.03 %	0.04 %	0.06 %
SrO	0.03 %	0.03 %	0.03 %
P_2O_5	—	0.03 %	0.02 %
Rb_2O	—	0.01 %	0.01 %
In_2O_3	0.12 %	—	—
Cl	0.03 %	—	—
Na_2O	—	0.13 %	—
MnO	—	0.04 %	—
Cr_2O_3	—	0.03 %	—

In this case, it may be observed clearly that, while the first sample of lime and sand integrates only $CaCO_3$, with the addition of the ceramic powder a part of the calcium is digressed to form calcium silicates, much more stable. This may be noticed also when we evaluate the loss on ignition (Fig. 6), because the procedure of the ABNT-NBR 6473/1998, that establishes a calcination at 950 ± 50 °C, does not affect silicates and aluminates, whose decomposition occurs at around 1500 °C.

What remains evident, besides the improvement of the mechanical resistance to axial compression and of the adhesion to the substrate, is the increase of the porosity that can be observed not only through the total water absorption tests (Fig. 7), but also through the water absorption by capillary uptake (Fig. 8) and surface water absorption (Fig. 9). It would be very important to understand the porosity distribution of the material and to verify the pore typology and size in the additivated mortar[11]. Consequently, it makes sense to compare them with the "sanitation" mortars, of which the main characteristic is the porosity.

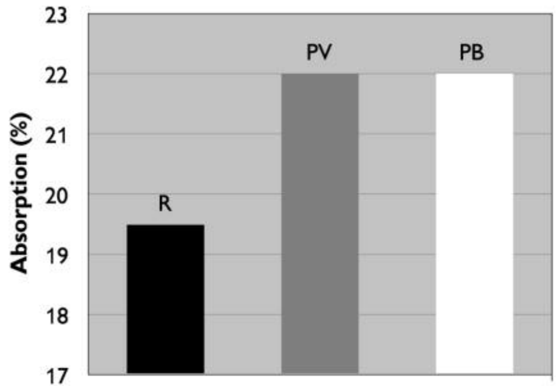

Fig. 7 Test of porosity accessible to water. Samples' codes as in Fig. 3.

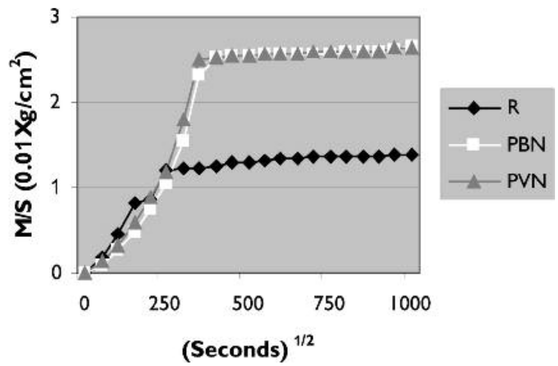

Fig. 8 Compared capillary water uptake of the different kinds of mortars. Samples' codes as in Fig. 3.

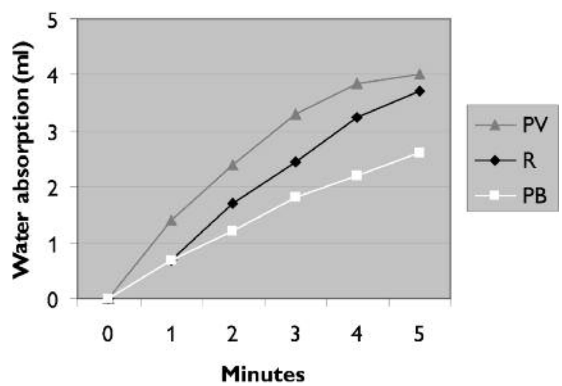

Fig. 9 Surface water absorption of the wall employing the Karsten pipes. Samples' codes as in Fig. 3.

[11] Preliminary porosimetry tests were performed which indicate that the addition of ceramic powder changes strongly the pore size distribution.

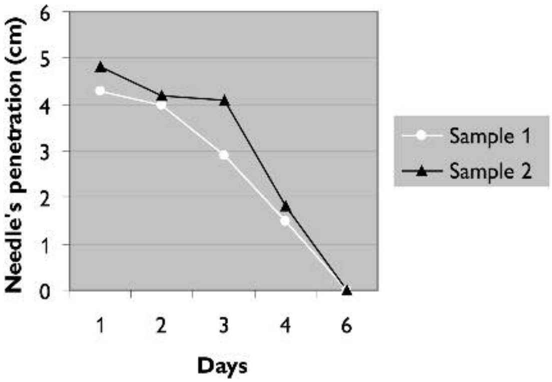

Fig. 10 Vicat's needle teste (adaptation).

The speed of hardening of the mortar was observed by the traditional Vicat's needle, demonstrating that the material was hard by the sixth day (Fig. 10).

Practical experiments

As Brandi very well indicated, the theory would not make sense if it was not to be necessarily put in practice [15, p. 55]. So, the lime mortar additivated with ceramic powder was applied in a specific and critical case of a wall with a massive presence of soluble salts. The opportunity came during the restoration of the Main Church of the historical city of Cachoeira. In this town, the occurrence of "leprosy" of the mortar (Fig. 11) is very frequent, produced by soluble salts that migrate by rising capillarity or even in isolated spots caused by contaminated construction material. The verification of the presence of soluble salts in the buildings of the referred town, including its Main Church, has been requested several times, and it was confirmed, almost always, a strong presence of NaCl and an even worse one of Na_2SO_4.

The preliminary tests for the coating of the most affected area, using a mortar similar to the one found in that same place (lime+sand+"saibro"), showed that this mortar, in little time, exhibited signs of decay, which also happened when the lime was substituted by a cementitious material. Only the mortar with ceramic powder is holding on to the present day, with the final application of mineral paint (Fig. 12).

Fig. 11 Situation before the restoration (2001).

There is no pretence of thinking that the problem has been solved definitively. The salts are still in the wall and sooner or later they might affect the surface. However, it may be affirmed that the additivated mortar is resisting better to the problem.

Conclusions

The conclusions may be summarized as follows:

- The procedure of employing ceramic powder as additive of lime mortars comes from thousands of years ago and was applied even in places where other pozzolanic materials were available.
- It is one of the most mentioned uses by the writers since Vitruvius;
- It is a process that brings, in general, meaningful improvement to the lime mortars;
- The more ceramic additive is added, the better the mechanical resistance;

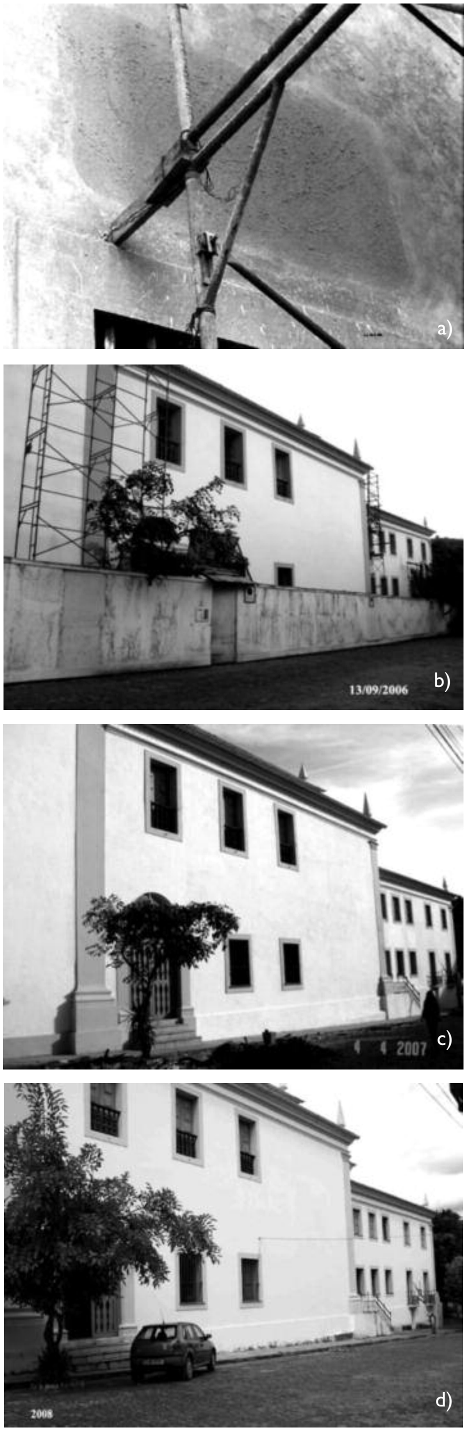

Fig. 12 a) Coating tests; b) After the application in 2006; c) Situation 2007; d) Condition in 2008.

- It was widely used in Brazil during its colonial period, especially by the military engineers in the construction of fortresses;
- It may be used as alternative material for modern restoration, not only in the lime coatings that need a faster hardening, but also in the walls contaminated with soluble salts.

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