

LOCAL POZZOLANIC MATERIALS IN AZOREAN BUILT HERITAGE

A. Velosa^{1*}, R. Veiga² and C. Vale³

1: Departament of Civil Engineering, Geobiotec
University of Aveiro
Campus Universitário de Santiago, Aveiro
e-mail: avelosa@ua.pt, web: www.ua.pt

2: National Laboratory of Civil Engineering
Avenida do Brasil 101, Lisboa
e-mail: rveiga@lnec.pt web: www.lnec.pt

3: University of Azores
Ponta Delgada Campus, Ponta Delgada, Açores
e-mail: celinavale@hotmail.com web: www.uac.pt

Keywords: Azores, heritage, pozzolans, renders

Abstract *Pozzolan materials from the Azores islands have long been extracted from nature and used as a construction material. Studies on these materials performed during the 20th Century by Castanheira das Neves [1] and Sousa Coutinho [2] are linked to their use in important construction works in mainland Portugal, such as water retaining walls in Aveiro lagoon and the Harbour of Leixões. This attests to the high quality of the materials and need for characterization in order to ensure adequate use of building materials.*

Despite their discontinued use in mainland Portugal, mainly due to the massive use of cement, natural pozzolan materials have been used in Azores islands for all types of buildings, from monuments to ecclesiastic buildings and are present throughout the extended panorama of vernacular architecture in which they were used until recent times.

Local availability of these materials and their properties are an invaluable resource that may still be put to use in the conservation of buildings, ensuring compatible refurbishment solutions. Rendering mortars incorporating local pozzolan materials may be designed for this purpose. In order to establish a basis for this possibility, this paper focuses on the analysis of pozzolan materials from the island of S. Miguel and of mortars from various time spans with incorporation of these materials. These mortars were sampled from monuments and vernacular architecture and incorporate military, industrial and ecclesiastic buildings as well as common residential buildings. Time-wise, they date from the 18th to the 20th Century. They were analysed using current analytical techniques for characterization of mortars (TGA, XRD, XRF, among others).

The knowledge of the characteristics of local pozzolans and of custom mortar mixes

portrayed in this paper can be used as an invaluable starting point for the design of conservation mortars using local materials.

1. INTRODUCTION

The use of materials extracted at a local level is a common factor in mortars used in rendering systems in most buildings up to mid-20th century. In the Azores islands, this usually accounts for the use of local materials of volcanic origin, with a strong prevalence of natural pozzolanic materials.

Still extracted today, natural pozzolanic materials hold a great variability linked to extraction depth and zone, but have been a relevant element in mortar composition both in Azorean architecture as in specific seaside constructions in mainland Portugal.

The study of these materials and of the composition of rendering systems in the island of S. Miguel, Azores is presented as a basis for mortar design taking into account conservation strategies of Azorean built heritage.

2. SAMPLING AND CHARACTERIZATION METHODOLOGY

Samples of local natural pozzolans were provided by local cement industry, from the current extraction site.



Figure 1 – Residential building in Ponta Delgada, Azores

Sampling of mortars from buildings was performed in the island of S. Miguel, Azores encompassing mortars from various periods: 16th and 17th century samples collected from S.

Brás Fortress, samples from industrial constructions such as a chicory factory and an iron smelting building from the 19th century and samples taken from 19th and 20th century residential buildings in Ponta Delgada (Figure 1) and Furnas.

As sampling was often performed on monuments or buildings with a good conservation state, severe restrictions to materials' removal existed, resulting in collected samples with a small size and irregular shape. Although sampling at various heights due to the effects of capillary water and rain as well as sampling on different walls due to their orientation was preferable, this methodology was not always undertaken so as not to damage façades excessively. Original mortars were extracted, ensuring the least damage possible and conservation actions were performed in order to prevent any possible damage due to the sampling. So as to obtain a complete characterization of the obtained samples, mechanical, chemical, mineralogical and physical analysis and tests were performed using the listed testing/analysis procedures.

2.1. Scanning Electron Microscope (SEM) visualization coupled with EDS

This technique was applied to representative samples and allows detailed visualization, with a resolution amounting to 200Å and the consequent identification of compounds formed by the interaction between pozzolans and lime as well as the crystalline structures of the mortar's constituents [3]. For this analysis a small freshly cut sub-sample must be obtained and covered with a conducting material. The junction of information from EDS (Electron Diffraction Spectrometry) complements the visual analysis, through chemical element characterization of an area or point under observation. For this analysis a Hitachi S-4100 (beam 0-30kV; usually works at 25kV) equipment was used with an EDS count time of 120 seconds.

2.2. Mechanical resistance by compressive strength

Due to the great variability in extracted samples, compressive strength was determined by a method developed at LNEC [4,5] which consists in confining the mortar with a regular shaped mortar stronger than the mortar in analysis and 40x40mm, to provide a known surface for force application.

2.3. Water absorption by capillarity

Water absorption test followed a specific testing procedure was developed at LNEC [6], involving the use of a metallic grid basket with a geo-textile, inside which mortars are placed and weighed. Capillary absorption testing was performed taking into account water intake and subsequent drying.

2.4. Acid dissolution

Acid dissolution was performed, based on ASTM -C 25-98 "Standard Test Methods for Chemical Analysis of Limestone, Quicklime and Hydrated Lime" [7] in order to dissolve carbonates (calcite, magnesite and dolomite) and create a residue composed by the aggregate fraction.

2.5. X-Ray Diffraction (XRD)

Through the use of this analysis technique qualitative information on the crystalline compounds existent in the samples is obtained. For this qualitative analysis, mortars were crushed and dried at a low temperature. The equipment used was a Philips X'Pert PW 3040/60 using CuK α radiation, with operational conditions of 30 mA and 50 kV, automatic divergent notch graphite monochromator and speed registry 1°/2 θ /min, with data acquisition by Philips X'Pert Data Collector v1.2.

2.6. X-Ray Fluorescence (XRF)

This technique allows the detection of elements present in the samples, by X-ray excitation of the sample [8]. Sample preparation was performed with the procedure used for XRD followed by the execution of powder pellets on which a Philips Fluorescence Spectrometer PW 1400 X-Ray was used. When petrographic analysis cannot be undertaken the quantities of calcium oxide and silica give us a first indication, to be corroborated by other methods, on the type of aggregate found in the mortar sample.

2.7. Thermogravimetric Analysis (DTA/TG)

Thermogravimetric analysis produces a continuous thermogram of the mass variation of a material with temperature rise, when it is heated at constant speed. In this register variations due to chemical alterations in materials such as dehydroxilation, oxidation, decarbonation or hydration can be observed. Each material has its typical curve permitting its detection through this technique [9,10]. This technique may be for the detection of carbonate aggregate when thin section analysis may not be applied, as the aggregate fraction decarbonates at a temperature range higher than that of current binder decarbonation [10].

For this analysis a Netzsch STA 409 C was used and the temperature range was between 20°C and 1100°C, with a heating rate of 10°C/minute.

2.8. Pozzolanic reactivity

Pozzolanic reactivity tests were performed after drying and sieving (75 μ m sieve) following “Caderno de Encargos para Fornecimento e Recepção de Pozolanas, Regulamento para Betões e Ligantes Hidráulicos” [11].

3. RESULTS AND DISCUSSION

3.1. Natural pozzolans

Natural pozzolan from the extraction site located in Água de Pau, Pisão was used, where there is the presence of several different layers and therefore materials with different characteristics and different degrees of pozzolanic reactivity.

Was removed material from two different strata containing pozzolans, located at depths of 5m and 12m from the surface, which was subjected to analysis. Additionally it was used directly from the excavation site, encompassing various extraction depths as prepared for cement production plant. The latter material was then subjected to a homogenizing crusher drying at

700 ° C. XRF results revealed materials with a high level of SiO₂ (53-57%) and Al₂O₃ (18-20%); Fe₂O₃ values range is 5-7% and alkaline oxides are present in the form of potassium and sodium oxides.

The XRD analysis revealed the presence of feldspars, (mainly anorthoclase) and quartz, olivine and mica/ilite. The visualization of a slope in the diagram between 20° and 33° indicates the presence of amorphous material.

In terms of pozzolanic reactivity, none of the analysed materials can be classified in a reactivity class following RBLH specifications [11], due to low flexural and compressive strength values achieved. However, the material with highest reactivity is that which was mixed, homogenized and calcined.

3.2. Rendering mortars

Mortars were extracted from two locations in the Azores island of S. Miguel: Ponta Delgada and Furnas. Upon an initial visual overview, it is clear that these renders show a brown/beige colour, sometimes quite strong, due to the use of local materials (Figure2). In this last case, the soluble fraction was high, around 50% and the mortars may have some earth content. In terms of mortars from Ponta Delgada, results showed a great variability and soluble fractions varied from 25% to 59%, although the range of 20% to 35% is characteristic of most of the analysed samples. Azores island mortars revealed an insoluble fraction composed of natural pozzolan, basaltic aggregate or volcanic tuff and in a few cases, black volcanic sand composed by ground volcanic rocks.



Figure 2 – Extraction of mortars in S. Miguel, Azores

In terms of the binder used in the studied mortars, a calcitic or dolomitic lime is the most

probable material, determined no doubt by the availability of the original rock coming to the island from neighbouring islands or the mainland itself.. Regarding this aspect, lime furnaces exist throughout the Azores, one of which in the town of Ponta Delgada,

Mortars from the Azores islands showed a great variability in terms of aggregate particle size distribution, not dependant on building type or time of construction. In this case, local available aggregates such as basalt, volcanic tuff and black volcanic sand, were used. Images from SEM clearly indicated the use of volcanic aggregates of local origin in these mortars.

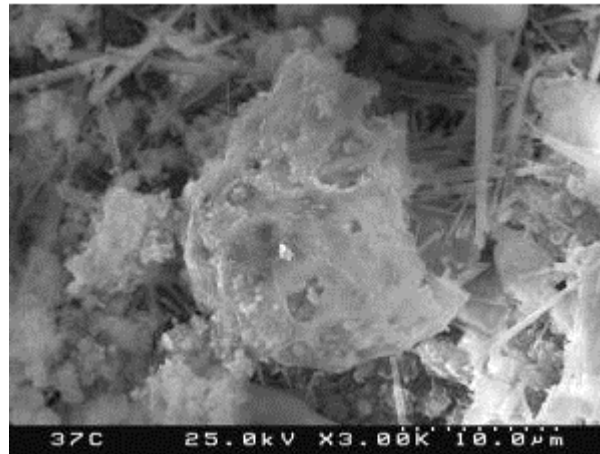


Figure 3 – Volcanic aggregate in mortar from S.Miguel, Azores

In the case of mortars from the Azores island of S.Miguel, a peak appears in the DTG curve at 460°C (Figure 3.3), that is due to the loss of hydration water either from CSH, from aluminosilicate phases or iron oxide contained in the mortar, due to pozzolanic additions.

Natural pozzolans are a common presence in mortars from the Azores, due to their vast availability. They may be detectable by their high content in feldspars and minerals such as pyroxenes, by XRD, and a high siliceous and aluminates content by XRF. Although they were probably used as an aggregate and are a part of the insoluble fraction, their action is also that of a binder and products from pozzolanic reaction may be detected by XRD or, as above said, by TGA.

Halite was an expected presence due to the effect of the sea and sea mist in the island. In terms of compressive resistance tests on the extracted samples, these resulted in values from 1MPa to 2MPa, which may be characterized as relatively low, but clearly intertwined with mortar composition and possible degradation.

Water absorption displays high variability, with the highest value was of 18,71kg/m².h^{1/2} from a Ponta Delgada mortar which had a very low mechanical resistance, presenting evident degradation, Drying of mortars was registered and usually, the drying process was fast, although lower than absorption.

4. CONCLUSIONS

Pozzolans from the island of S. Miguel have been widely used in mortars in Azorean built heritage, as was expected due to wide local availability. Although their pozzolanic reactivity may not be classified as high, according to the followed testing procedures, the presence of hydraulic compounds as products of pozzolanic reactions was detected in the sampled mortars.

The availability of local materials allows the execution of similar mortars that may be applied in the conservation of the Azorean built heritage.

REFERENCES

- [1] Castanheira das Neves (1906), Subsídios para o estudo das pozolanas e sua aplicação nas construções, LNEC, Lisboa, Portugal.
- [2] Coutinho, A. S. - Pozolanas, betões com pozolanas e cimentos pozolânicos, LNEC, Lisboa, 1958.
- [3] Pinto, J. Bessa; Silva, A. Santos (2003) Emprego de pozolanas em argamassas de cal. O exemplo dos Fortes de S. Julião da Barra e da Ericeira, 3º ENCORE, Encontro sobre Conservação e Reabilitação de Edifícios, LNEC, Lisboa, Portugal, Vol.1, pp. 337-344.
- [4] Magalhães, A. C.; Veiga, M. R. (2006) Avaliação da resistência de amostras de argamassas históricas recolhidas em obra. Metodologia e correlação de resultados. Lisboa: LNEC. Relatório 259/06 – NRI.
- [5] Valek, J., Veiga, R. (2005) Characterisation of mechanical properties of historic mortars - Testing of irregular samples. Advances in Architecture Series Volume 20, pp. 365-374.
- [6] Veiga, M. R. (2005) Characteristics of repair mortars for historic buildings concerning water behaviour. Quantification and requirements. In Workshop Repair Mortars for Historic Masonry, TC RMH. Delft, RILEM, 25-28.
- [7] ASTM -C 25-98 "Standard Test Methods for Chemical Analysis of Limestone, Quicklime and Hydrated Lime"
- [8] Silva, A. S. (2003) Nova abordagem na caracterização de argamassas antigas, 3º ENCORE, Encontro sobre Conservação e Reabilitação de Edifícios, LNEC, Lisboa, Portugal, Vol. 2, pp. 917-926.
- [9] Hakateyama, T.; Zhenhai, L. (1998) Handbook of Thermal Analysis, John Wiley and Sons.
- [10] Mackenzie, R.C. (1957) The differential thermal investigation of clays, Mineralogical

Society, London.

- [11] Platret, G.; Deloye, F.X. (1994) Thermogravimetrie et carbonatation des ciments et des bétons. Journée des Sciences de l'ingénieur du réseau des LPC,. Vol 1, pp. 237-243.
- [12] República Portuguesa (1995) Decreto-Lei n.º 330/95 de 14 de Dezembro, Caderno de Encargos para o Fornecimento e Recepção de Pozolanas, Regulamento de Betões de Ligantes Hidráulicos (RBLH).