BORSA SIMP 2009 PER UN SOGGIORNO DI STUDIO ALL'ESTERO: RELAZIONE SULL'ATTIVITA' SVOLTA

PROJECT: IMAGE ANALYSIS IN MODELLING MIXTURES OF RAW MATERIALS USED IN THE PRODUCTION OF ANCIENT MORTAR

REBECCA PIOVESAN
Dipartimento di Geoscienze, Università degli Studi di Padova, Via Gradenigo 6, I-35131 Padova

INTRODUCTION

Archaeometrical researches on ancient mortars commonly focus on provenance and production technology, through a combined petrographic, microstructural, geochemical and spectroscopic approach. The definition of minero-petrographic composition and textural features of aggregates provides important information on the provenance of raw materials and on the nature of inclusions (sand, grinded rocks, grog), as well as on performances of mortars, allowing adequate planning in restoration. In some cases, microscopic analysis can be used to distinguishing original mortars from those produced during a renovation, or to date buildings belonged to various historical periods in the same archaeological site (Bruno *et al.*, 2004; Miriello *et al.*, 2010). The determination of textural parameters in ancient mortars is normally achieved either through sieving of previously disaggregated material or by microscopic observations in thin sections. Recently, alternative investigation approaches based on image analysis (IA) techniques to process optical and electronic microscopic digital images (Carò & Di Giulio, 2004; Carò *et al.*, 2008), have been proposed. This approach allows higher automation and lower subjectivity in the data acquisition and quantification. Nevertheless, many issues still exist and need to be solved, such as sample representativeness of images, accuracy and reliability of the method, presence of compositionally different and complex textural elements, orientation of non-equidimensional fragments and heterogeneity of inclusion distribution (Carò *et al.*, 2006). For this reason, it is essential to develop an investigation method, which allows quantifying volumetrically the different textural elements (*i.e.* types of aggregate, binder and pores) using standardized automatic and semi-automatic procedures for calculating the percentage contribute of each of them.

This research project, financially supported by the SIMP scholarship, was partially carried out during the permanence at the University College of London and aimed to investigate the possibility of applying automatic procedures of IA for characterising textures in mortars, both using already applied procedures based on image thresholding – *i.e.* using electron back scattered (BSE) images – and developing new approaches based on the combination of different image processing, depending on the nature of the specific object. In particular, the distinction of grains, differing in minero-petrographic composition, was performed by multi-layered comparison of elemental maps and BSE images. In order to verify the applicability of this approach to mortars, a set of 15 experimental mortars, differing both in terms of minero-petrographic composition of aggregate and aggregate grain-size, was prepared at the Dipartimento di Geoscienze of the Padova University. More in detail, the aggregates used were produced grinding and sieving pure minerals (quartz, feldspar), rocks (marble, limestone, trachyte), artificial material (pottery), and sands (that were only sieved) in several different grain-size classes and discarding the fraction larger than 4 mm and smaller than 0.125 mm. They were then used alone, or mixed with other different types, according to different grain classes, as described in Table 1, in order to obtain a wide variety in terms of composition and texture of the aggregate, whereas the binder type and the aggregate/binder ratio were maintained constant. After 60 days of setting, each sample was thin-sectioned and analyzed by scanning electronic microscopy (SEM). Observations were made at 20 KeV and WD 20 mm (working distance). The magnification and the image resolution were chosen with care to guarantee a frame large enough to contain the largest grains, but at the same time a pixel size small enough to detect the smallest detail. For each thin-section, a set (from 11 to 18) of backscattered pictures (1280×1024 pixels) was captured from the SEM video output channel, for a total of about 200 images.

Table 1 - Composition of the experimental mortars

N°	Code	Composition	Aggregate/Binder ratio	Grain Sizes (mm)
1	QtU	Quartz	0.6	2÷0.8
2	QtB	Quartz	0.7	2÷0.8 + 0.63÷0.4
3	FdU	Feldspar	0.6	0.8 ÷0.63
4	FdB	Feldspar	0.6	2÷0.8 + 0.63÷0.4
5	MrU	Marble	0.6	2÷0.8
6	MrB	Marble	0.6	2÷0.8 + 0.31÷0.2
7	CMU	Limestone	0.5	2÷0.8
8	CMB	Limestone	0.5	2÷0.8 + 0.63÷0.4
9	CcU	Grog	0.5	0.8 ÷0.63
10	CcB	Grog	0.5	2÷0.8 + 0.63÷0.4
11	TA3	Volcanic sand	0.9	0.4÷0.31
12	CF6	Volcanic sand	0.5	0.63÷0.4
13	Tr	Trachyte	0.6	2÷0.8
14	Mx1	Marble+Grog+Quartz	0.5	2÷0.8
15	Mx2	Marble+Grog+Quartz	0.6	0.8 ÷0.63

PERFORMED RESEARCH AND PRELIMINARY RESULTS

The research at the UCL - Earth Sciences Department (London) was overseen by Dr. Ruth Siddall, which has a wide experience in the study and characterization of historical mortars (Siddall, 2000, 2006). During the scholarship the undersigned have carried out the main phases of the IA process. Two open-source and widely used programs were selected to perform the analyses (MultiSpecUniv, Image J). After a preliminary test of the applicability and capability of the two programs, MultiSpecUniv (MS) was chosen to measure the percentages of the main components of mortar (aggregate, binder and porosity); while Image J (IJ) was preferred to define the dimensions of the grains and theirs grain-size distribution. Before starting the real IA processing, all the images were moderately elaborated with IJ to increase the quality of the image and improve the resolution of the grains boundaries.

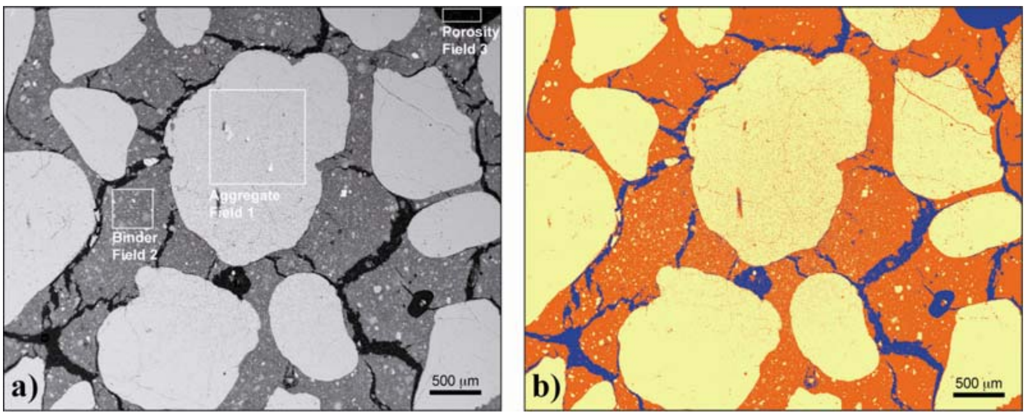

Fig. 1 - Steps of the MS processing on a quartz-mortar: a) example of a selection of regions of interest (ROI) for each class on a BSE original image; b) resulting false-colours image in which in yellow is the aggregate, in blue the binder, and in orange the porosity.

With MS, the characteristic grey tones of each textural component (aggregate, binder and porosity) were selected and adopted by the program as reference for the definition of the areas belonging to the three components. These indicative selected areas are known as "Regions of Interest" (ROI). After that, the program assigned every single pixel of the original image to the corresponding classes, and rendered a new image in false-colours (Fig. 1) from which the percentages of the defined classes were derived. From these percentages, has also been calculated the aggregate/binder ratio, considering as binder the sum of binder plus porosity.

Image elaboration for measuring dimensional parameters of grains was more complicated (Fig. 2). Adopting IJ, an automatic histogram-based thresholding was applied to discerning the grains from the binder and the porosity (Fig. 2b); after that the image was turned into a binary product, through a segmentation process (Fig. 2c). On this image, derivative-base edge detection was performed to define the grain boundaries (Fig. 2d) and to compute, for each grain, three relevant dimensional indices (Area, Feret max diameter, and Feret min diameter) in addition to the standard parameters (perimeter, diameters, ellipse dimensions, etc.).

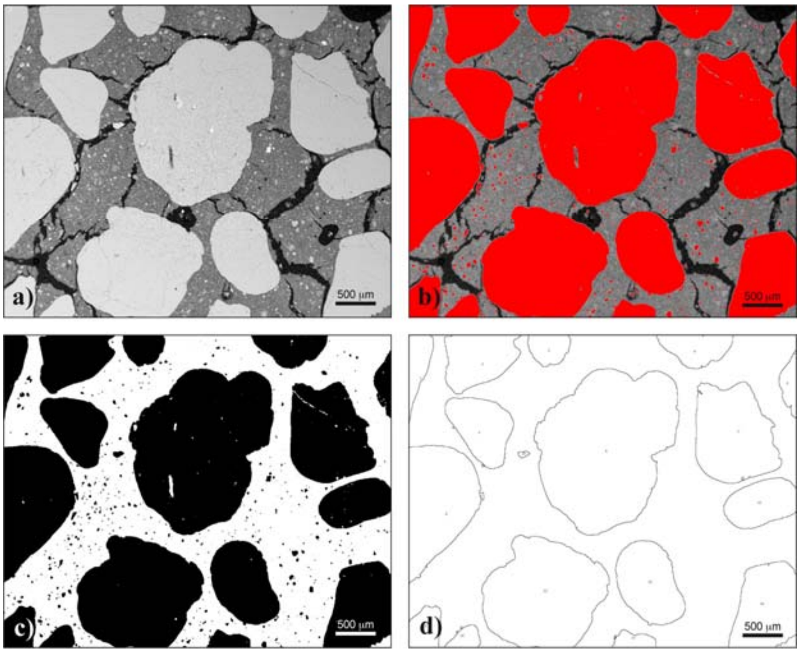

Fig. 2 - Steps of the IJ processing on a quartz-mortar: a) original BSE image; b) thresholding of the same image; c) resulting binary image; d) drawing of the detected grains obtain by removing object less than 300 pixels.

During this work, the data were recorded on electronic tables and the images stored for each step. As regard the percentages of the components measured by MS, the preliminary results have showed a good accuracy of the measurements as demonstrated by the comparison of the real aggregate/binder ratio and the one obtain by the IA. As shown in Fig. 3, some variances with the real values are present; the reasons of these discrepancies are not yet clarified and further sampling, analyses and processing need to be done on them.

In addition, as regard the grain dimensional parameters, the min Feret diameter was taken as reference to verify if the grain size distribution measured by IA were comparable with the real adopted fractions. This parameter was chosen because it can be considered the index that better approximate the sieve size used in the mechanical sieving. The statistical processing on min Feret diameter, performed mainly by the means of frequency distribution, revealed a shift of the observed IA grain-size data to the finer dimensions with respect to the real grain size of the aggregate (Fig. 4). This underestimation of the aggregate grain size can be imputed to the cut-effect that influences the sampling of a three-dimensional body by a two-dimensional surface (Borradaile, 2003).

Further analyses and elaborations are needed and planned to better understand the real validity of this method and also to define what problems can affect these measurements and how to anticipate and/or solve them. Moreover, this method was also used to study a series of mortars from Romano–Punic cisterns sampled at Nora (Cagliari-Italy), mainly to verify the applicability of the method to historical samples, and to define the production recipes used to made these materials in terms of volumetric ratio between aggregate and binder.

These procedures permitted, on one hand to identify the composition of the aggregate and the relative abundance with respect to the binder, and on the other to define the morphometry and grain-size of aggregates. The acquisition and processing of the historical mortars are still in progress, as the performing of elemental maps by SEM-EDS on representative samples, to discriminate also aggregate with different minero-petrographic composition (samples Mx1, Mx2).

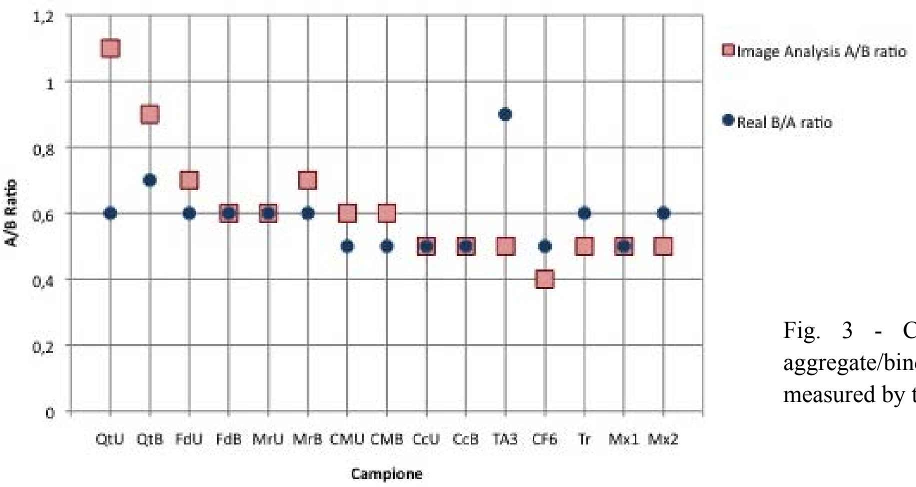

Fig. 3 - Comparison between real aggregate/binder (A/B) ratio and the one measured by the image analysis.

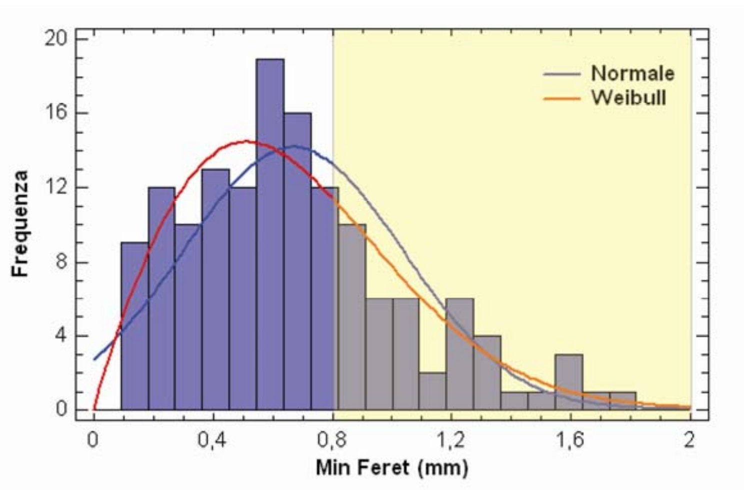

Fig. 4 - Frequency distribution of aggregate grain-size for mortar sample produced with fragment of limestone. The yellow square indicating the real grain size adopted.

All the results obtained in this research will be soon presented to the scientific community as communications at conferences, and submitted as scientific papers for publication on ISI international journals with targets in applied petrography, archaeometry and materials sciences.

REFERENCES

Borradaile, G. (2003): Statistics of Earth Sciences Data. Springer, Berlin, 351.

Bruno, P., Calabrese, D., Di Pierro, M., Genga, A., Laganara, C., Manigrassi, D.A.P., Traini, A., Ubbriaco, P. (2004): Chemical-physical and mineralogical investigation on ancient mortars from the archaeological site of Monte Sannace (Bari-Southern Italy). *Thermochim. Acta*, **418**, 131-141.

Carò, F. & Di Giulio, A. (2004): Reliability of textural analysis of ancient plasters and mortars through automated image analysis. *Mater. Charact.*, **53**, 243-257.

Carò, F., Di Giulio, A., Marmo, R. (2006): Textural analysis of ancient plasters and mortars: reliability of image analysis approaches. *Geol. Soc. London, Spec. Publ.*, **257**, 337-345.

Carò, F., Riccardi, M.P., Mazzilli Savini, M.T. (2008): Characterization of Plasters and Mortars As a Tool in Archaeological Studies: the Case of Lardirago Castle in Pavia, Northern Italy. *Archaeometry*, **50**, 85-100.

Miriello, D., Barca, D., Bloise, A., Ciarallo, A., Crisci, G.M., De Rose, T., Gattuso, C., Gazineo, F., La Russa, M.F. (2010): Characterisation of archaeological mortars from Pompeii (Campania, Italy) and identification of construction phases by compositional data analysis. *J. Archaeol. Sci.*, **37**, 2207-2223.

Siddall, R. (2000): The use of volcaniclastic material in Roman hydraulic concretes: a brief review. *In*: “The Archaeology of Geological Catastrophes”, B. McGuire, D. Griffiths, I. Stewart, eds. *Geological Society, London, Spec. Publ.*, **171**, 339-344.

Siddall, R. (2006): Hydraulic Mortars in Antiquity: Analyses from Fountains in Ancient Corinth. *In*: “Common Ground: Archaeology, Art, Science and Humanities”, A. Brauer, C. Mattusch, A. Donohue, eds. Oxbow Books, London, 208-211.