

Thermal Analysis and Chemometrics for the Differentiation of Mortars from Ancient Roman and Renaissance Frescoes

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Abstract

Using Principal Component Analysis (PCA) applied to the main thermogravimetric data collected on ancient Roman and Renaissance frescoes, it was possible to compare and differentiate, in a simple but rigorous way, several samples of frescoed mortars studied. The research also proved how the ancient Roman mortar samples are richer in binder (calcium carbonate) and contain less filler (aggregate) than the Renaissance mortar samples.

Introduction

Over the past few years we have studied and characterized several mortar samples from ancient Roman (2nd Century AD) and Renaissance (16th Century) frescoes [1-2]. Recently, we showed how thermal analysis, in particular thermogravimetry (TG-DTG), coupled with chemometric methods, can be a valid tool for the characterization and classification of several types of archaeological finds and cultural heritage, e.g. pigments [3], marbles [4], pottery, fossil bones, and so on. Therefore, we decided to apply thermogravimetric analysis also to several mortars sampled from different ancient Roman and Renaissance frescoes to determine whether, also in the case of ancient mortars, the simple combination of thermal analysis and chemometric methods could be used to obtain a fast but correct classification of ancient mortar samples of different ages.

Materials & Methods

The ancient Roman frescoes studied are currently preserved in the Museo Nazionale Romano (Terme di Diocleziano, Rome, Italy) and denoted as "Termini Station" samples in the present paper, as the site from which they originate is near the present-day Termini Central Railway Station (Rome, Italy). The Renaissance fresco samples come from the "Old Vatican Rooms" (Vatican Museum, Rome, Italy) and belong to three different frescoes known as: "Il Parnaso", "Il Passaggio del Mar Rosso", and lastly the specimen from the "Heliodorus Room".

10-20 mg of each sample (i.e., few granules of material coming from the *intonachino* or *intonaco/arriccio*, in the case of Roman and Renaissance frescoes, respectively) obtained during the restoration of the frescoes were gently ground up and placed in an alumina crucible. They were then subjected to controlled thermal scanning from room temperature up to 1000 °C at a heating rate of 10 °C min⁻¹ in an air flow of 100 cm³ min⁻¹, using a TG 50 thermobalance connected to a TC10A processor (from Mettler, Swiss) and a TA1200 DTA instrument coupled to a TA2000 processor (from Dupont, USA).

Results

All the main TG data (% mass losses and temperatures of the three principal decomposition step and residue at 1000 °C) were processed after autoscaling by Principal Component Analysis [5] for the purpose of demonstrating how thermal data alone, simply with the help of chemometric projection techniques, are sufficient to separate mortars belonging to frescoes of different ages into different clusters. Indeed, the representation of the samples onto the space spanned by the two

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significant principal components clearly evidences a separation between Roman and Renaissance frescoes, and also the subgrouping of the latter (Figure 1).



Figure 1 – PCA of the main TG data after autoscaling: scores plot. Legend: p "Il Parnaso"; pmr "Il passaggio del Mar Rosso"; hr "Heliodorus Room"; Ta-Te "Temini" samples.

Indeed, results show that the two groups of samples (i.e. ancient Roman and Renaissance frescoes) are clearly separated along the first principal component, the differentiation being related in particular to the mass loss due to the carbonate decomposition step and the percentage of final TG residue at 1000 °C.

Conclusions

It was demonstrated how, also in the present research, the coupling of thermal analysis and chemometrics represents a useful, simple and advantageous approach for dealing with problems of characterization and classification of different kinds of archaeological finds and cultural heritage.

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