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Thermochimica Acta 269/270 (1995) 809–816

thermochimica acta

# Characterization of the lumps in the mortars of historic masonry[☆]

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Received 16 September 1994; accepted 9 June 1995

**Abstract**

The present work focuses on the investigation of mortar joints of historic masonries consisting traditionally of aerial binder and inerts which contain lumps. The presence of lumps, usually white in colour and of various dimensions, was often recorded inside these mixtures and does not appear to be random, as they are rather frequent, These lumps could confer some physico-chemical properties to the mixture, that favour the overall compatibility of the system.

For this purpose, various samples taken from historic Venetian masonry were examined by TG-DTG and FTIR analysis. Moreover SEM and fibre optical microscope observations were performed. The results indicate mainly the presence of completely carbonated lime and lead us to assume that the lumps arise from technologies based on the non-seasoning of the lime.

*Keywords:* Fibre optical microscopy; FTIR; Historic masonry; Lumps in mortar; Mortar joints; SEM; TG-DTG

## 1. Introduction

A first evaluation of the historic joint mortars of aerial lime has been performed through macroscopic observations, which constitute the initial approach of the study of such materials. The macroscopic examination refers to the external aspects and provides the first information on the system that must be verified with chemical,

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☆ Presented at the 6th European Symposium on Thermal Analysis and Calorimetry, Grado Italy, 11–16 September 1994.


*SSDI* 0040-6031(95)02573-1

physical and mechanical analyses. The structural aspects of the mortar are also very important, as they concern the distribution of the binder, the form of the inert and the texture of the mortar as a whole. Appropriate workability would require the correct mixing of binder with the inert, producing a mixture with homogeneous physical and mechanical characteristics.

Macroscopic observation has revealed the presence of nodules, usually white in colour, in the mixture (Fig. 1). Their presence in the mortars could indicate that the treatment undergone did not meet the norms suggested [1]. The presence of the lumps/nodules in the joint mortars is a widespread peculiarity of various Venetian manufactures [2]. In the majority of the examined cases the high frequency in which the lumps were recorded is evident and cannot be considered completely fortuitous. However, the lump is a common element independent of the site, the construction period, the manufacture typology, and the raw materials employed in the preparation of the mixtures [3]. Investigation of this particular characteristic of traditional mortars could clarify both preparation technologies of the mixtures and application procedures. In the light of the great demand, recently expressed, for the conservation of the original materials, the knowledge of traditional mortar technologies is important.

This work studies the composition and morphological characteristics of the lumps and aims to interpret their origin and their impact on the mortars.

## 2. Experimental

### 2.1. Sampling

Samples of joint mortars containing nodules, were taken from churches and historic buildings of certain periods from different sites around the Venetian area. Sampling was performed considering the different construction periods. The samples stem from

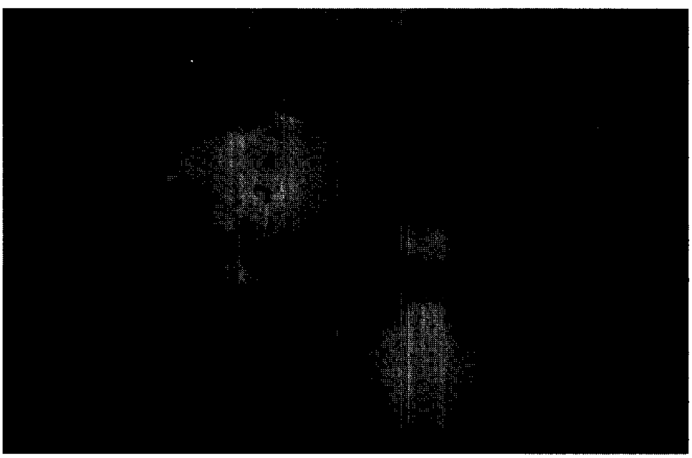

Fig. 1. The presence of the lumps in the mixture (50 ×).

the period of the 14th to the 18th centuries and have as a common ground the use of aerial lime as binder. Moreover, the different colour of the nodules, their dimensions and distribution in the mixture is carefully considered. However, the lumps, present various colours that are not always uniform, with white or whitish yellow prevailing, while their dimensions vary from a few millimetres 1–2 cm.

### *2.2. Analysis*

The samples of the nodules included in the mortars were examined by using the following analytical procedures: infrared spectroscopy (FTIR) for the qualitative identification of the substances present (Biorad FTS 40); thermogravimetric and differential thermogravimetric analysis (TG – DTG) for the quantitative determination of the compounds, which present a weight loss in the temperature range 30–1000°C and with a temperature gradient of 10°C $min^{-1}$ in static air atmosphere (Mettler TG 50); fibre optical microscopy (MFO) for observations on polished sections, for morphological examination of the samples (Keyence monitor microscope); scanning electron microscopy (SEM) for the examination of the texture, with greater magnification equipped by an electron probe for chemical microanalysis (EDX) (Cambridge Stereoscan 250).

## 3. Results and discussion

The infrared analysis performed on a significant number of nodules included in various mortars has revealed the nearly exclusive presence of calcium carbonate (2513, 1796, 1446, 873, 713 $cm^{-1}$) (Fig. 2). However in some cases the presence of silicates was

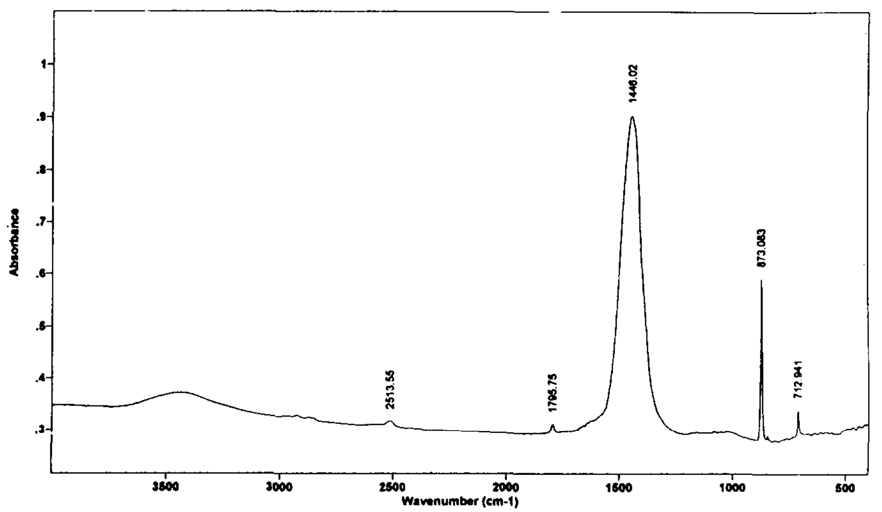


Fig. 2. Infrared spectrum of a lump containing calcium carbonate.

recorded (1158, 1055 $cm^{-1}$) (Fig. 3). Nevertheless the infrafed qualitative analysis indicated that the lumps are completely carbonated, and the presence of calcium hydroxide was not noticed in any sample.

The thermogravimetric analysis was performed to quantify the $CO_2$ present in the samples and therefore the corresponding quantity of calcium carbonate. The $CO_2$ was also determined by calcimetry (gas volumetric method) that revealed the typical kinetics of decomposition of the calcium carbonate [4]. Also it should be noticed that the percentages of $CO_2$ measured by calcimetry are similar to those measured by thermogravimetry. Moreover, the spot tests performed for the presence of magnesium gave negative results.

Table 1 reports the weight loss percentage estimated from the TG-DTG curves, only for some of the samples analysed, in the temperature range 30-1000°C. In particular,

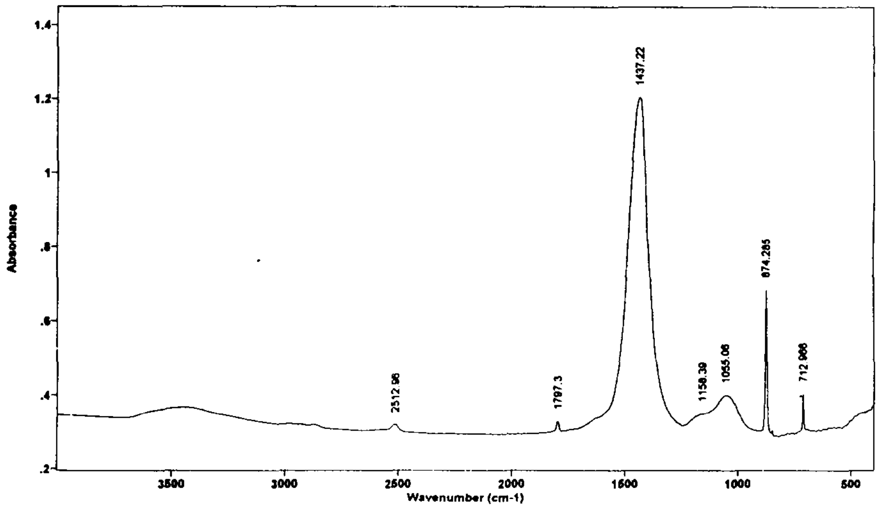


Fig. 3. Infrared spectrum of a lump containing calcium carbonate and silicates.

Table 1
TG-DTG analysis of some of the analysed samples

| Sample | Weight loss per temperature range (°C)/% | | | |
|---|---|---|---|---|
| | <120 | 120-600 | >600 | $CaCO_3$/% |
| GE1 | 2.01 | 4.13 | 39.54 | 89.95 |
| GH1 | 2.18 | 5.75 | 37.37 | 85.02 |
| GI1 | 4.50 | 7.11 | 34.28 | 77.99 |
| GP1 | 1.87 | 4.81 | 34.88 | 79.35 |
| GR1 | 3.46 | 3.77 | 33.06 | 75.21 |
| GT1 | 6.60 | 8.77 | 28.60 | 65.06 |

the weight loss below 120°C was ascribed to hygroscopic water, i.e. physically adsorbed water, whereas the weight loss in the temperature range 120–600°C could be mostly attributed to the water bound to hydraulic compounds or clays. The organic substances (200–500°C) and hydrate salts, e.g. gypsum (120–160°C) [5, 6], which also undergo weight losses in the above mentioned temperature range, were not taken into consideration as they were not noticed in the infrared analysis. The weight loss above 600°C was attributed to the $CO_2$ due to the decomposition of calcium carbonate.

The weight loss below 120°C varies between 1.5 and 6.5%, the loss in the temperature range 120–600°C varies between 3 and 8.5%, while the loss over 600°C fluctuates between 28 and 39.5%. In general, there is a connection between the above mentioned temperautre ranges. For example, a relatively high weight loss at temperatures below 120°C corresponds to a greater loss in the temperature range 120–600°C and to a low weight loss above 600°C (relatively low $CO_2$ content). This fact confirms the presence of hydraulic compounds that could be formed from lump/inert interaction or impurity content in the inert; on the other hand the presence of silicates was recorded by infrared spectroscopy.

The fibre optical microscope observations (MFO) enable better examination of the lumps on polished sections, from a morphological point of view. The lumps present various shapes and dimensions. The prevailing colour is white or whitish yellow, while other colours recorded are brown, yellow–brown, pale pink and grey. In some cases colour differences were noticed in the inner part of the lumps (Fig. 4), probably due to different levels of compactness of the calcium carbonate that commonly constitues the lumps. The colour differences could also be attributed to neoformation compounds. The lump structure is often porous but usually homogeneous. In some cases fractures in the structure of the lumps were recorded. The latter probably developed during the working procedure of the lime with the inert, or from the contraction which the lime undergoes during the loss of water and the successive carbonation evident in the

Fig. 4. Colour differences in the inner part of the lumps (50 × ).

absence of the inert. It is possible to distinguish some situations where the surface separation between lump and mortar matrix is clear, and others where part of the inert has partially penetrated into the lump. In the former, we can suppose that the lump was already present in the mixing step and was completely carbonated or that this was in a uncarbonated form but successively carbonated, at a slow rate, probably contracting slightly on the surface. In the latter it is possible that the lump (accumulation of the binder) has initiated the carbonation process after the working of the mixture as the rest of the binder or from the application technologies of the mortars.

The observation of the lumps by scanning electron microscopy (SEM) has shown a homogeneous structure, characterized by the diffuse presence of the pores on the surface (Fig. 5). The matrix of the mortar surrounding the lumps appears to be of compact texture with smaller porosity in respect to the lumps (Fig. 6). In many cases the growth of the binder crystals is greater than of the lumps. The examination with EDX microanalysis, confirmed the presence of calcium as the predominant element, and identified the presence of both silicon and aluminium. This fact further confirms the presence of hydraulic compounds in the lumps.

The overall results obtained lead to the following remarks on the origin and impact of the lumps on the mortars. First of all, the presence of the lumps is a common element independent of the site, construction period and manufacturing typology and consequently testifies to similar manufacturing technologies. Their composition is prevalently carbonatic. This indication in conjuction with the microscopic observations leads us to consider that these lumps were probably in the mixture in calcium hydroxide form (slaked lime) and that a low working of the mixture has not permitted the lime to be properly mixed with the inert. Another hypothesis could be that the lime was slaked just before being used. The plasticity of the lime mixture was therefore insufficient as it was not properly seasoned so as to allow for the growth of big portlandite crystals to

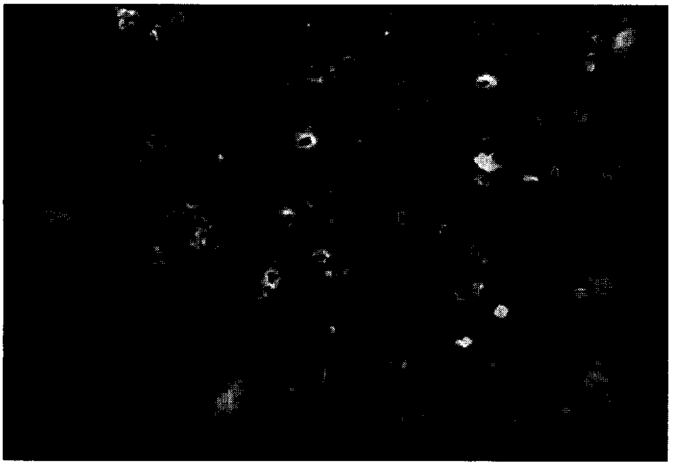

Fig. 5. Lump of calcium carbonate with the diffused presence of pores (3000 × ).

Fig. 6. Interface lump (below)/mortar matrix (above) (600 × ).

hexagonal laminae. This fact combined with a low water/lime ratio in order to obtain better mechanical properties, rendered the workability of the mixture difficult and consequently led to the insufficient mixing between lime and inert.

The frequent presence of the lumps in the mortars therefore implies that they should confer particular mechanical characteristics to the mixtures; they could favour the adaptability of these mixtures into the masonry structure and enhance their elastic behaviour. In many cases the use of mortars with less capability of mechanical compression strength, but with higher elasticity and mechanical tensile strength seems to be international [7]. In fact the lime mortar after hardening is less rigid than the cement materials. The relative elastic strain of the joint mortar seems, however, to be a fundamental condition for a satisfactory behaviour of traditional masonry, on the basis of an analogous mechanism to that of modern composite materials.

## 4. Conclusions

The analyses performed on various lumps have revealed that their main constituents are calcium carbonate and variable quantities of silicoaluminates.

The presence of the lumps in the mortars is probably due to the absence of suitable working of the mortar, so as not to enable complete mixing of lime and inert and to the use of technologies based on the non-seasoning of the lime, producing in this way mixtures with low plasticity. The presence of the lumps in the mortars of various construction periods implies that they may confer particular physico-mechanical features on the mixture, which should favour the elastic behaviour of the masonry structure. Mechanical tests on the mixtures could better clarify the impact of the lumps in the lime mortars on the behaviour of the masonry.

## Acknowledgement

This work has been financially supported by the Italian National Research Council (CNR).

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