Characterization of an Archaeological Mortar from the Ottoman Period in Algeria

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Abstract—**Knowledge of the characteristics of materials is an important source of information to understand the historical and archaeological evolution of mortars, with a view to finding a mortar with characteristics similar to ancient mortars, which can subsequently be used in the restoration of historic monuments. Mortars intended for the restoration of historic properties must be compatible with the characteristics of the materials to which they are to be applied.**

The purpose of this study is to characterize an ancient sand and lime mortar to which other components have been added, used in the construction of the Ottoman complex "Palais des Raïs"; and to identify its chemical composition and physical characteristics.

Through the results obtained, we will try to make a comparison between the mortars of the Citadel of Algiers located in the Upper Kasbah, and Bastion 23 located in the Lower Casbah.

Based on the various observations, we will be able to affirm or refute the hypothesis concerning the cause of the deterioration of the façades of Bastion 23.

Index Terms—**Characterization, mortar, lime, ottoman, heritage, physical properties.**

I. INTRODUCTION

Studies on lime plaster mortar are rare and cover only part of the historical heritage. Among the studies on mortars of the Ottoman period in Algeria, Mahindad's work [1] this consists of the mineralogical and physico-chemical characterization of mortars of the Ottoman period. Other sources [2]-[6] it undertakes the characterization of mortars, their composition and evolution. These studies were carried out on several samples obtained from the powder magazine and casemates located at the citadel of Algiers, a house in the Kasbah of Algiers and the Villa Mahieddine.

Other historic studies show evidence that the mortar of the Ottoman period consisted of lime and red clay or red sand, which were more or less clay-like [6]-[9].

II. METHODOLOGIES

The mortar can be used for several purposes, namely, masonry pointing mortar, finishing, coating and waterproofing mortar for the terrace.

In the restoration of a historic plaster mortar, it is necessary to respect the principle of authenticity [2] and to know how to preserve, maintain or repair what exists. In the case of renovation, the ideal solution is to try to reproduce a mortar identical to the old one, but this remains impossible to adopt. However, another one compatible with this one must be used.

To have knowledge of the historical mortar adopted in Bastion 23, the choice of samples is the basis for its characterization.

This choice requires a lot of sampling, which contradicts the principles of conservation of cultural property. The unique presence of a few archaeological windows does not allow us to take several samples. In this study, we selected two jointing samples found on site.

The coating mortars are deteriorated following the various aggressions and agents mentioned above and stripped during the restoration. Unfortunately, this prevents us from identifying the main pathologies that affect these historical coatings of Bastion 23.

This characterization allows us to identify the nature of the historical binder, its composition and its different physical characteristics. It also allows us to compare these historical mortars with those used in the same period but from different sites.

A. Sampling Procedure

We first chose the old support to be treated of the historical monument, on which we identified a mortar of possible dating and relatively satisfactory aspects. In order to achieve a better sampling, we have also based ourselves on the following criteria:

Color and texture;

Component elements and particles

The size of the aggregates.

Fig. 1. archaeological window of house 12.

The mortars collected were extracted from the existing archaeological windows. One is located on the ground floor of house 12 (Fig. 1). The other one is at the level of the "Roman bath" located in Palace 17 (Fig. 2).

Manuscript received November 25, 2019; revised April 16, 2019.

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doi: 10.18178/ijmmm.2019.7.3.450

The sampling was done with a chisel and a pallet. The top layer has been removed to ensure the best conditions for successful mortar characterization. Samples were taken from heights higher than 1m above the ground to avoid mortars affected by capillary rise.

Fig. 2. Specimens taken from archaeological windows.

B. Description of the Test Method and Results

The determination of the physico-chemical and thermogravimetric characteristics of mortars is a means of finding all the information on the construction of the monument, the composition and the state of deterioration of the construction mortars.

The scheduled analyses and tests are: SEM analysis, thermogravimetric analysis, chemical analysis and treatment, density, specific mass, porosity, moisture content and water absorption.

C. Scanning Electron Microscope (SEM) Analysis

1) Principle and methodology

The scanning electron microscope provides surface images of virtually all solid materials, at scales ranging from the magnifying glass (x10) to the transmission electron microscope (x500,000 or more).

In addition to the chemical composition, the properties of the mortar also depend on its structure. The latter is observed using images that allow the study of texture at the micrometer scale (μm), in which one can identify: the presence of non-luminescent crystals, the presence of crushed brick, mica, bones, etc.; and the presence of adjuvants in the pores.

2) Results and discussion

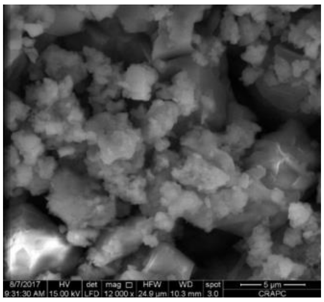

Fig. 3. a. SEM picture of the mortar at different magnifications, Ech : 5μm

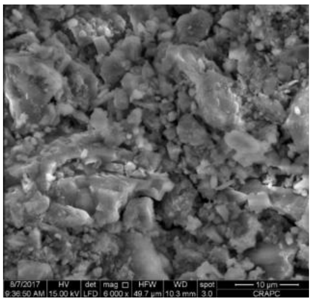

Fig. 3.b. SEM picture of the mortar at different magnifications, Ech : 10μm

This analysis was performed using a Scanning Electron Microscope (SEM) on a slice of the sample, in several areas, and with different magnification ranging from 400 times to 12000x. In addition to the knowledge of grain size distribution, it allows to characterize the morphology and microstructure of the mortar sample at different scales.

The sample has a rigorous external appearance affected by traces of dissolution. Alteration microfacies appear as porosity. The mortar, composed mainly of lime, shows a very porous morphology to the SEM (Fig. 3. a & b). These pores allow the mortar to breathe. The grains are of any shape and size and the aggregates are spaced by empty clutter at depth. The whitish fine limestone particles, due to the Mediterranean climate, have an amorphous appearance of variable dimensions.

D. Thermal Analysis ATG / ATD / DSC

1) Principle and methodology

The ATG /ATD/DSC thermal analysis was obtained using an SDT Q600 TA instrument. The experiments were performed in an alumina crucible and in an atmosphere under inert gas N2 (at 100ml/min) to protect the sample from oxidation. The heating rate is 10°C/min and the thermal cycle is 50-1000°C.

This analysis allows to observe a mass variation and its associated thermal effect over time. Thermo-gravimetry (ATG) is used to measure mass variations. This mass variation can be dehydration, decarbonation, etc., or mass gain (gas fixation).

Differential thermal analysis (DTA) is a technique in which the temperature difference between a sample, and a reference material, is measured as a function of time or temperature while the sample temperature is programmed, in a controlled atmosphere. While differential enthalpy analysis (DSC) determines the variation in flow thermal energy emitted or received by a sample when it is subjected to temperature programming.

2) Results and discussion

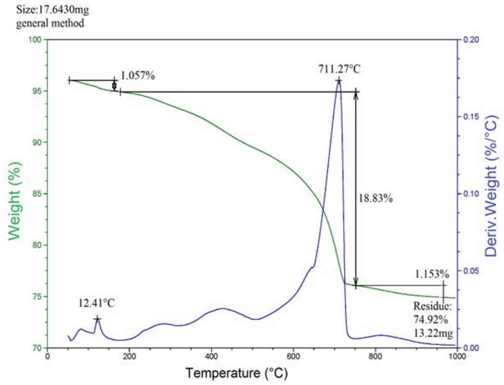

Fig. 4. ATD/ATG curve of the plaster mortar.

The table and curve (Fig. 4) show the results of the mortar thermo-gravimetry. The heat treatment allowed us to define five temperature ranges corresponding to significant mass losses. According to [3], [4], the loss of mass produced between 50 and 120°C is considered to be due to the loss of absorbed water. The one, between 120°C and 200°C at the loss of water from hydrated salts. Between 200 and 600°C, weight loss is due to dehydroxiliation or loss of water due to hydraulic compounds. Finally, for mass losses between 600 and 1000°C, they are due to a departure and loss of CO_2 which is caused by the decomposition of calcilum carbonate (calcium lime) ($CaCO_3$).

The initial weight of the sample is 96.07 mg and after analysis, there was a weight loss of approximately 13.22mg. (Table I).

TABLE I: LOSS OF MASS AS A FUNCTION OF TEMPERATURE

Temperature	< 120°C	120-200°C	200-600°C	600-1000°C	> 1000 °C	Mass difference (mg)
weight	0.065	1.056	18.83	1.153	0.019	13.22

E. Physical Analysis

1) Density test

The density of a material is the mass of that material per unit volume, taking into account pores and capillaries. It is determined by means of a graduated cylinder. It is expressed by the relationship:

$$Ms = \frac{M1.\rho}{M3-M2} \qquad (\mathrm{g/cm^3})$$

M_1: dry sample mass: g.

M_2: mass of vial filled with petroleum ether; g.

M_3: mass of vial full of petroleum ether containing the fully immersed sample, g.

ρ: specific mass of petroleum ether, ρ= 0.65g/cm^3

2) Specific mass test

The specific mass of a material is the mass of this material per unit volume without taking into account pores and capillaries. It is determined by the following formula:

The porosity of a material is the ratio of the volume of pores and capillaries contained in the material to its total volume. It is calculated from the values of specific mass and density. The relationship that determines this porosity is as follows:

$$Ms = \frac{(M_1-M_3)(d_t-d_\alpha}{M+(M_1-M_3)-M_2} \qquad (\mathrm{g/cm^3})$$

d_t: toluene density: d_t = 0.65

$d\alpha$: air density; d = 1.29.

M_1: Empty pycnometer mass; g.

M_2: Pycnometer mass with sample powder

M_3: Mass of pycnometer with toluene, g.

3) Porosity test

The porosity of a material is the ratio of the volume of pores and capillaries contained in the material to its total volume. It is calculated from the values of specific mass and density. The relationship that determines this porosity is as follows:

$$Pc = \left(1 - \frac{Mv}{Ms}\right).100 \qquad \%$$

4) Moisture test

The mortar, through its pores and interstices, allows the penetration of aggressive agents. The humidity test is an essential indicator of its durability.

This test consists first of all in creating a wet environment in which the moisture retention capacity is tested at room temperature for a time in minutes. The moisture content retained by the samples is calculated by the following formula

$$Ms = \frac{(Mf-Mi)}{Mi}.100 \qquad \%$$

M_S: moisture content in %.

M_f: mass of the final sample in g

M_i: mass of the initial sample in g.

5) Results and discussion

In order to enrich our research, we wanted to compare the results of the analysis of the mortar of the Raïs Palace with those used in the Upper Kasbah.

The water absorption of the mortars shows the importance of the pores present in it. Porosity was determined from the density and specific gravity values. The old lime-based mortars are porous, as evidenced by their porosity, which is 30.95% for the sample taken from Bastion 23, and from 23% to 35% 10 for the mortars of the citadel of Algiers.

More porous of the mortar, easier is the diffusion of CO_2. and the higher the infiltration of water from capillary upwelling. The absorption rate of 20.85% for Bastion 23 and from 14% to 25% 12 for the citadel of Algiers, confirms this.

As for the humidity rate, it is 1% for the mortar of Bastion 23 and varies from 1.08 to 9.7% for the citadel of Algiers. 13

The two results allowed us to observe that the mortar samples from the citadel of Algiers (Table III) and the mortar sample from Bastion 23 (Table II) have practically the same values and physical properties even if they are in two different sites.

TABLE II: RESULTS OF THE PHYSICAL ANALYSIS OF THE "BASTION 23" SAMPLE

Designations	Density g/cm^3	Specific mass g/cm^3	Porosity %	Water absorption %	Humidity % Moisture
Sample	0.87	1.26	30.95	20.85	1

TABLE III: RESULTS OF THE PHYSICAL ANALYSIS OF THE "CITADEL OF ALGIERS" SAMPLE

Designations	Density g/cm^3	Specific mass g/cm^3	Porosity %	Water absorption %	Humidity % Moisture
Sample	1.42-1.67	1.96-2.32	21.59-32.08	12.75-20.18	1.08-

F. Chemical Analysis

1) Methodology

The tests for the determination of the chemical characteristics of the mortar were carried out at the (*Centre d'étude et de services technologiques de l'industrie des matériaux de construction (CETIM)*) in accordance with standard NF EN 196-2. Chemical analysis makes it possible to obtain the quantitative chemical compositions of the mortar, particularly the compounds. This basic study helps in the technological characterization of mortars using X-ray fluorescence.

2) Outcome

The table below (Table IV) summarizes the results of the compositions and chemical characteristics of the mortar.

By comparing the results of the analysis of the Palais des Raïs mortar with those used in the Upper Kasbah (Table V), we noticed that both types of mortar contain the same compounds, with rates that are similar. In both cases, we found that SiO_2 is the most important component of the mortar with a rate of 42.25% for the Palais des Raïs 23 and varies from 45.42% to 52.98%. The CaO content is also important. It is 17.16% for the sample of the Palais des Raïs, and varies between 13.25% and 17.27% for the joint mortars of the Kasbah. We also found significant levels of Al_2O_3, with a content of 10.81% for the first and ranging from 7.29 to 10.96% for the second mortar.

3) Discussion

Chemical analysis shows the existence of a large quantity of silica (SiO_2) in the composition of the sample taken; the origin of SiO_2 is mainly sand. There are also significant quantities of lime, which implies the existence of calcium carbonate in the mortar, but the quantities are much lower than those of silica. Note: are also significant quantities of aluminium oxide (Al_2O_3), while the other components Fe, Na and K exist only in moderate quantities.

Note: for all samples, the presence of these small quantities of Albite (traces of K and Ca) and Moscovite (traces of Fe), the source of which is likely to come from the addition of components, such as baked and ceramic brick crushed or ground 14.

The crushed brick gives the mortar a pozzolanic and hydraulic character, which activates hardening and rapid setting. Pozzolan, often used in the Roman period, is defined as a siliceous or silico-aluminous material, which does not have binding properties, but which, in the form of a very fine powder and in the presence of moisture, reacts chemically with calcium hydroxide at ordinary temperature to form compounds with binding properties. Finally, the negligible content of P_2O_5 (Phosphorus pentoxide) in the sample indicates the absence of organic matter in the composition of the mortar.

TABLE IV: RESULTS OF THE CHEMICAL ANALYSIS OF THE "BASTION 23" SAMPLE (CETIM-SOURCE)

	Content										
Code	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	P_2O5	TiO_2	PF
Sample	42.25	10.81	4.55	17.16	0.89	0.16	2.44	1.31	0.10	0.61	19.7

TABLE V: RESULTS OF THE CHEMICAL ANALYSIS OF THE "CITADEL OF ALGIERS" SAMPLE (MAHINDAD-SOURCE)

	Content									
Code	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	P_2O_5	TiO_2
Sample	45.42	7.29	2.86	16.23	0.61	0.09	1.49	0.69	0.05	0.32
	52.98	10.96	4.31	17.27	1.05	0.28	2.76	1.60	0.07	0.62

III. CONCLUSION

The results allowed us to observe the similarities of the mortar components, their respective proportions and their physical properties.

By crossing the results of the physico-chemical characterization of the old lime-based plaster with the diagnosis established on the various pathologies altering the Palais des Raïs, we can put forward the hypothesis that, the use of cement as plaster on all the facades of "bastion 23", remains the initial cause that disintegrates the buildings and the material that damages the condition of the facades. Due to its compactness, it helps moisture to remain stagnant inside the wall due to its impermeability.

The percentage of porosity and absorption are significant for most original mortar samples. These two characteristics are influenced by the quantity of mortar components, particularly lime.

Cement used in buildings as a covering material on the facades of old walls, keeps old masonry behind an unsuitable plaster that retains moisture and prevents water from draining away normally.

The persistence of this humidity phenomenon constitutes, in the long term, a major risk to the stability of the load-bearing structures of buildings.

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