Journal of Materials and Environmental Sciences
ISSN : 2028-2508
CODEN : JMESCN

J. Mater. Environ. Sci., 2018, Volume 9, Issue 3 Page 754-762

https://doi.org/10.26872/jmes.2018.9.3.83

http://www.jmaterenvironsci.com

From the stone to the lime for Tadelakt: Marrakesh traditional plaster

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Received 15 May 2017,
Revised 15 Nov 2017,
Accepted 18 Nov 2017

Keywords

- ✓ Tadelakt
- ✓ Lime,
- ✓ Marrakesh lime,
- ✓ Traditional plasters,
- ✓ Waterproof plasters.

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Abstract

Tadelakt is the Marrakesh traditional plaster that thanks to its waterproof qualities was used for flooring, ceilings, bathrooms, ponds and water tanks. It is realized using a particular hydraulic lime produced in the outskirts of Marrakesh. The paper investigates the compositional characteristics of this plaster starting from the nature of the original carbonatic stone used for the production of the lime. As a matter of fact, the traditional burning process causes the presence of underburnt fraction that coupled with the silicaticclastic fraction of the original raw material behave as aggregate. Therefore, the lime for Tadelakt is not a simple binder but a ready-to-use material that does not need the addition of any aggregate but only water, and pigments if necessary. Other two distinctive features of this binder are the presence of magnesium hydroxide (brucite) and palygorskite that thanks to their fibrous structure contribute to avoid the formation of shrinkage fractures in the finished product. The whole of these characteristics allow to univocally identify the original lime of Marrakesh and to realize this particular plaster.

1. Introduction

Used since the Neolithic age, lime is a natural material produced by burning limestone. It is a binder which, mixed with water and possibly other natural products like sand and volcanic materials, makes it possible to obtain a mortar, a product that can be utilized with many different functions (to connect bricks and stones of a masonry, to plaster, as sealing etc.).

A particular use of this material is Tadelakt, a finishing technique for supports (wall, floor, ceiling, ...). This traditional plaster consists of lime, water, black soap and often mineral pigments. The technique was created and developed in the Marrakesh region thanks to the very special lime produced in that territory. The knowledge of this artisanal product (raw materials, history, manufacturing process, etc.), its uses and interactions with the environment, will makes it possible to improve its production process and quality of the finished product promoting this ancestral heritage in the field of construction.

2. The Tadelakt: history

Tadelakt is a traditional Marrakesh lime plaster that thanks to its waterproof qualities and resistance to water, was used for flooring (dess), ceilings, arches and domes and it is the traditional plaster for water tanks, bathrooms, basins, etc. [1-2] (Fig.1). Tadelakt was known in the region of Marrakesh and it can be compared to marmorino, which is found in many Mediterranean countries. Nevertheless, it is much more than a traditional plaster, it is the identity, the symbol and the culture of a region [3]. This material and technique take the name from the Arabic name dellek meaning "rubbing". It requires lime, black soap, a polishing stone and other tools, and can be realized only by experienced craftsmen, the "maalems", who have inherited the expertise which remains the key factor to achieve a good finished artefact.

The history of this material goes back to the 12th century, in the Almoravid (1021-1147) and Almohad dynasties (1147-1262).

In Morocco, the Tadelakt technique was almost disappeared in the early 70s[4] in favour of new, premixed, materials that allow to accelerate the time of the application. His reappearance in the 80s was mainly due to the new interest for the traditional architectures, mainly in Marrakesh. Its sealing qualities together with durability, aesthetics and its varied hues accompanied by an ecological dimension increasingly appreciated, changed Tadelakt in a popular material appreciated in the world.

Figure 1: Bathroom in Riad of Marrakech

3. The materials of lime for Tadelakt

The materials used in the realization of Tadelakt are lime, water, olive oil black soap, stone for polishing process and mineral pigments.

Lime has been used as the principal binder for mortars for the past 10,000 years. Lime is a product obtained burning a more or less pure carbonatic rock. Depending on the chemical composition of the original rock, different limes can be obtained. Two types of traditional lime are mainly used: air lime and natural hydraulic lime. In the production process, quicklime is first obtained by burning the rock at about 900ºC to drive off carbon dioxide. If the raw material is pure limestone or dolostone (containing no more than 5% of clay minerals), pure quicklime is produced (made respectively of CaO or $CaO+MgO$). If the rock contains more than 5% of clay minerals, then hydraulic quicklime is produced made of CaO + $C2S$ (calcium silicates) [3].In the second phase, water is added to quicklime to obtain the slaked lime.

Concerning Tadelakt, lime is the main constituting material and is obtained through calcination of a particular limestone which characteristics will be deeply discussed later in the experimental part of the paper.

3.1. *The burning process*

The burning of the stone for lime is carried out in traditional kilns sited near the supply areas of the stones. In the surroundings of Marrakesh, there are still about twenty active kilns. The burning lasts for 24 hours without interruption, with dry palm wood and leaves of green palm. Temperature inside the kiln is not homogeneous, not monitored, but generally estimated between850-1300°C.

3.2. *The black soap*

The black soap is a soft vegetable paste produced according to traditional manufacturing recipes, results of crushed black olives and oil mixture soaked in salt water and potassium hydroxide (KOH).

It is usually applied the day after the realization of the plaster. Its role is crucial to the sealing of Tadelakt, being able to close the micro cracks, promoting the final tightening of the plaster and giving it a remarkable sheen.

When the plaster is coming to set (generally within 24 h after application), its surface is coated with black soap then polished again with a smooth stone making it shiny and waterproof.

4. Preparation of the plaster

The lime for Tadelakt (slaked lime in powder) is placed in container pouring water, a little at a time, so that it is absorbed as it is poured, without stirring. You can practice some holes in the paste to facilitate the penetration of water. The paste must be left to stand for 12-72 hours, so that the water is completely absorbed, making sure to close or cover the container to prevent evaporation. After 12-72 hours, the paste can be mixed. It can be done by hand or using mechanical means (electric mixer, trowel, etc.). If necessary, small quantities of water can be added to obtain a plastic consistency paste. During the mixing, mineral pigments can be added, up to 10% by weight of pigment compared to slaked lime, according to the needs and personal taste.

5. Experimental part

As previously reported lime is the main constituting material of Tadelakt and is obtained through calcination of a particular limestone which can be found around Marrakesh. We have studied the following materials:

- stone for lime: it comes from an area located 15 km north east of Marrakesh (DouarOuladMessaoud) from carbonatic crusts of Quaternary age;
- calcined stone: it is presented in the form of clods. The analysed samples are obtained from the clods showing nodular appearance, ranging in colour from light brown to red ochre;
- slaked lime: it is supplied in a polyethylene bag and placed in turn inside a plastic bucket hermetically closed, it appeared as powder, light brown to reddish ochre;
- on site Tadelakt : a sample of Tadelakt plaster has been taken from the little wall (50 cm high) that surrounds the basin of the Menara gardens in Marrakech (Fig. 2).

Figure 2: The little wall surrounding the Menara basin

5.2 *Methods*

The stone for lime, the calcined stone, the slaked lime and the finished product (Tadelakt) were studied according to the following analytical methodologies:

- the mineralogical compositions of stone for lime, calcined stone, slaked lime were determined on powder utilizing an X-ray diffractometerPANalyticalX'PertPRO equipped with X'Celerator detector and High Score data acquisition and interpretation software. The following operative conditions were applied: Cu $K\alpha1$= 1.545Å, current intensity of 30mA, voltage 40kV, explored 2Θ range between 3°-70°, step size 0.02°, time to step 50 s and scan speed of 0.04°/s. The quantitative determination of the mineralogical phases was performed through Rietveld analysis method;
- the amount of $CaCO_3$ of the stone for the lime was determined through the Gasometric technique, by using a Dietrich Fruhlingcalcimeter[5];
- the petrographic investigations on the stone for lime and the on site Tadelakt plaster were carried out through observation in transmitted light of thin sections (30 microns thickness) with an Optical Microscopy (ZEISS

Axioscope A1 microscope equipped with a camera (resolution 5 Megapixel) and dedicated image analysis software (AxioVision) for evaluating the microstructural parameters);

- the granulometric analysis of the slaked lime was verified by a MATEST A058-05N Air Jet Sieving Machine. The instrument is suitable for sieving powder and dry grain products by obtaining sieving results between 5 to 4000 microns, by using appropriate test sieves 200 mm dia.;
- the chemical composition of the stone for lime and slaked lime has been performed through XRF measurement by a PHILIPS PW 1480 Spectrometer, powder samples on fused disks;
- the study of the neoformation hydraulic phases has been performed through TG/DSC analyses were carried out in the 20–1400 °C temperature range with a heating rate of 20°C/min in a air atmosphere, with a STA 429 (CD) Netzsch instrument;
- a deeper study has been carried out in the case of the Tadelakt to identify the possible presence of the reaction products between lime and black olive oil soap utilizing infrared spectrometry analysis with Bruker Optics ALPHA Spectrometer in the mid-infrared region (4000-400cm^{-1}). Spectra were acquired in total reflection mode collecting 16 scans, with 4 cm^{-1}resolution on KBr pellet.

6. Results and discussion

6.1 *The stone for lime*

Microscopic observation shows that the stone for lime is constituted by a matrix of impure micritic calcite and very abundant clastic fractions where, however, the grains are not in contact each other (Fig.3). This fraction is composed mainly of quartz and feldspars in single crystals. The texture is not homogeneous due to the presence of microsparitic plagues in which the clastic fraction is scattered. There are frequent areas of sparitic recrystallization.

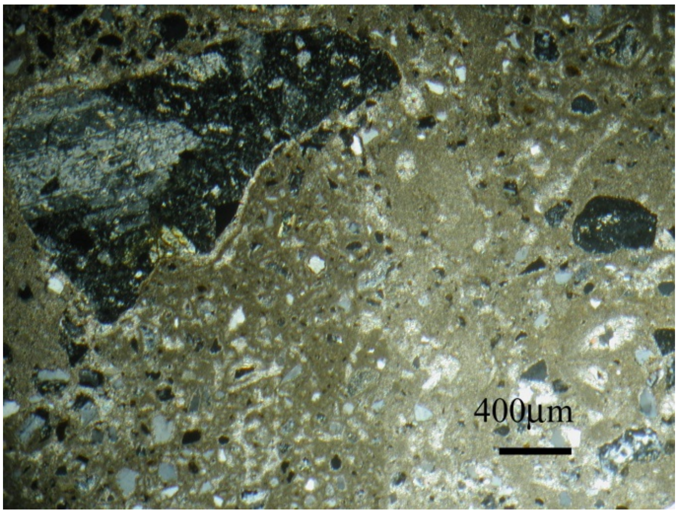


Figure 3: Thin section image of the stone for lime in XPL

The result of the gasometric analysis indicates an amount of 63 ± 2 % of $CaCO_3$.This result is in agreement with the quantitative value obtained through XRD with Rietveld method, as reported in Table 1.

Table 1: Mineralogical composition of the *stone for lime*

Composition	*%**
Calcite	67.5± 0.9
Dolomite	6.8± 0.2
Quartz	9.4± 0.4
Mica	4.4± 0.3
Palygorskite	3.4± 0.2
Albite	4.5± 0.2
Microcline	2.1± 0.2
Amorphous phase and other clay minerals	2.0± 1

*on dry sample (105°C)

The mineralogical composition indicates a total amount of carbonates of about 74%. Therefore the stone is at the limit between a marly limestone and calcareous marl rich in dolomite [6]. The presence of palygorskite is particularly interesting both for a geographical characterization of the raw material and for its chemical and physical properties that makes it a special mineral. Palygorskite, indeed, is an important phyllosilicate which, thanks to its micro-fibrous morphology, is extensively used in the formulation of various industrial products. Palygorskite is characterized by high crystalline structure, high specific surface area and excellent sorptive capacity.

The chemical analysis (XRF) is in agreement with the mineralogical data as confirmed by the detection of elements as Calcium (CaO, about 61%) and silica (SiO_2 about 24%), while to a lesser extent Al_2O_3(5%), MgO (4%), Fe_2O_3(2%), Na_2O (~ 1.5%) and K_2O (< 1%) are present. Only traces of elements such as titanium, sulphur, strontium, manganese, phosphorus, chlorine, barium, arsenic have been recorded.

6.2 *The calcined stone*

The XRD analysis of the calcined stone shows the presence of minerals that indicate an average temperature of firing between 900 - 1200°C (Tab.2). Burning temperature is evidenced by the almost total absence of residual carbonates (calcite 0.5%) and by the abundant presence of C2S (bicalcium silicate) and by the incipient formation of C3S (tricalcium silicate), C3A (tricalcium aluminate) and C4AF (Brownmillerite). These last three phases are typical of temperatures between 1300-1500°C. The presence of these high temperature phases can be attributed to hot spots occurring within the kiln and caused by direct flame contact with part of the limestone loaded.

Generally speaking, the presence of calcium silicates and calcium aluminates phases is due to the reaction of calcium oxide with the silica and alumina originating from the destruction of clay minerals (i.e. palygorskite) and feldspars (albite and microcline) and from the partial fusion of quartz.

Table 2: Mineralogical composition of *calcined stone for lime*

Composition	***%****
Calcite	0.5± 0.1
Dolomite	0.3± 0.1
Alite	1.1± 0.2
Tricalcium aluminate-alite	2.5 ± 0.2
Bicalcium silicate-belite	22.1± 0.4
Lime	37.2 ± 0.2
Quartz	8.0 ± 0.1
Brownmillerite	1.0 ± 0.2
Periclase	1.1 ± 0.1
Portlandite	25.9± 0.3
Amorphous and minor phases	0.7 ± 0.8

*on dry sample (105°C)

The abundant presence of portlandite, $Ca(OH)_2$, indicates an occurred hydration of the calcium oxide (CaO) after the burning step in the weeks prior to testing due to unsuitable storage conditions.

6.3 *The slaked lime*

From the mineralogical point of view, the prevalence of C2S phase (13.3%) compared to phases of higher temperature like C3A (1%), C3S (absent or <1%) C4AF (about 1%) confirms an average burning temperature between 900°C and 1200°C (Tab.3).The presence of these compounds gives the slaked lime the characteristic of a natural hydraulic lime.

The katoite presence is attributable to a partial hydration of the C3A occurred during the slaking process in which the supply of water is required to convert all the calcium oxide into calcium hydroxide (portlandite): it is possible that part of the water combines with C3A to form hydrates phases (katoite), obviously not more active compound for the hydraulic setting of lime.The TG/DSC analysis (Tab.4 and Fig. 4) point out an amount of portlandite (available lime) between 33-36%, together with the presence of 8-9% of carbonates due to residual unburned material and possible recarbonation phenomena due to storage conditions in direct contact with the air (atmospheric CO_2). These data are in agreement with the XRD data.

Table 3: Mineralogical composition of slaked lime

Composition	*% **
Calcite	5.7 ± 0.1
Tricalcium aluminate	1.0 ± 0.3
Belite	13.3± 0.4
Katoite	5.7± 0.3
Portlandite	35.5 ± 0.6
Dolomite	2.1 ± 0.2
Quartz	9.5 ± 0.2
Periclase	0.8 ± 0.1
Palygorskite	1.0± 0.1
Brucite	1.0± 0.1
Amorphous and minor phases	24.0± 1.0

*on dry sample (105°C)

Table 4.TG/DSC of slaked lime

Parameter	*%*
free water*	5.6
bound water**	8.0
portlandite	32.9
calcite***	7.2

* Determined by drying to constant weight in an oven (105 °C)
** The weight loss in the temperature range corresponding to the TG/DSC peak at about 450 °C.
***Assuming only the decomposition of calcite ($CaCO_3$) in the temperature range corresponding to the TG/DSC peak at about 750 °C.

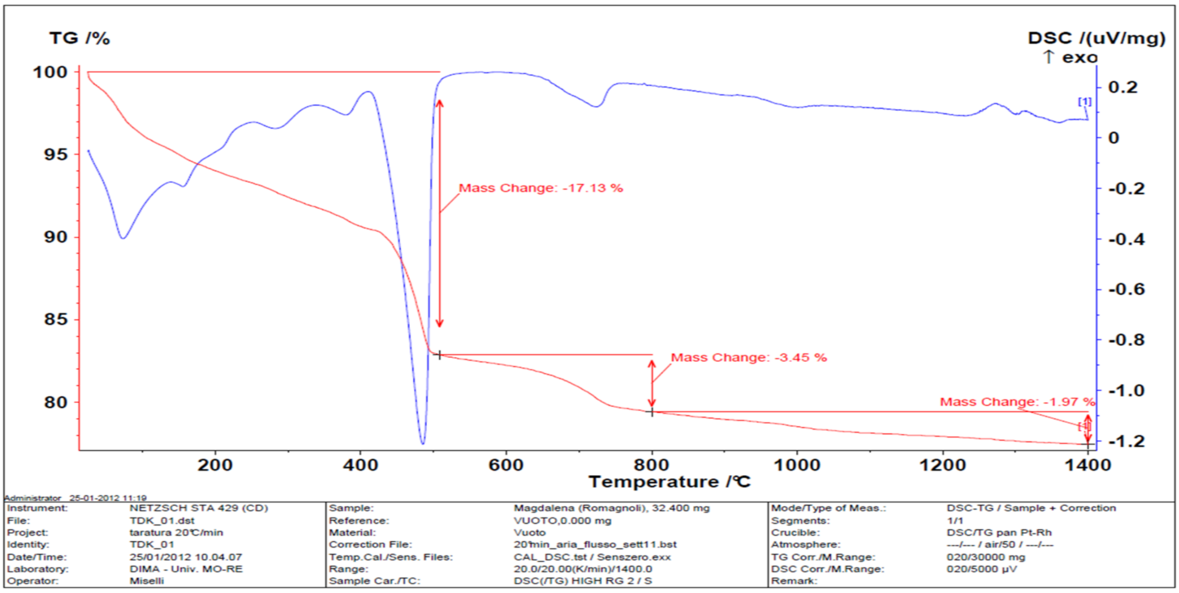


Figure 4: TG/DSC curve of slaked lime

Considering that the composition of the slaked lime is not standardized, but varies with each calcination process, we can say that, from a chemical point of view, the finished product is fully compatible with the stone for lime. (Table 5). Many indexes, based on oxides composition of cement materials, have been defined to quantify their hydraulic properties. For hydraulic limes, the cementation index (CI) has been represented by the equation [7]:

$$CI= (2.8\%SiO_2+1.1\% Al_2O_3+0.7Fe_2O_3) / (\%CaO+1.4\%MgO)$$

Limes showing CI within 0.3 and 0.5 were defined weakly hydraulic, for CI values between 0.5 and 0.7 moderately hydraulic, for CI values between 0.7 and 1.1 highly hydraulic lime. Cementation Index of the slaked lime for Tadelakt has a value of 1.0-1.1 (highly hydraulic lime).

Chemical analyses conducted on two types of natural hydraulic lime available on the EU market, in particular, NHL 3.5 according with EN 459-1:2010 [8], showed Cementation Index values of 0.95 and 0.56 [9].

Table 5: Comparison between the chemical composition *of stone for lime* and slaked lime

Chemical composition	***Stone for lime %****	***Slaked lime %****
CaO	61.33±0.5	65.77±0.5
SiO_2	23.98±0.5	23.32±0.5
Al_2O_3	5.27±0.5	4.01±0.5
MgO	4.04±0.5	2.54±0.5
Fe_2O_3	2.00±0.5	1.97±0.5
SO_3	0.28±0.5	0.14±0.5
K_2O	0.77±0.5	1.08±0.5
Na_2O	1.41±0.5	0.59±0.5
TiO_2	0.36±0.5	0.35±0.5
SrO	0.12±0.5	0.06±0.5
MnO	0.03±0.5	0.04±0.5
P_2O_5	0.03±0.5	0.09±0.5
Cl	0.15±0.5	-
BaO	0.12±0.5	-
As_2O_3	0.06±0.5	0.07±0.5
HM**	2.10	2.41
SM***	3.29	3.90
AM****	2.64	2.04
CI*****	1.10	1.02

* value without LOI (lost on ignition)

** Hydraulic Modulus (HM) = $CaO/(SiO_2+Al_2O_3+Fe_2O_3)$

*** Silica Modulus (SM) = $SiO_2/(Al_2O_3+Fe_2O_3)$

**** Aluminium Modulus (AM) = Al_2O_3/Fe_2O_3)

***** Cementation Index (CI) = $(2.8\%SiO_2+1.1\%Al_2O_3+0.7Fe_2O_3)/(\%CaO+1.4\%MgO)$

With regard to the particle size of the slaked lime, it is observed that 22% by weight of the material has sizes < 0.180 mm, while the remaining 84% between 2 mm and 0.180 mm (Tab.6). The average value is approximately 0.250 mm (Fig.6). Considering that only the finer fraction (< 0.200 mm) is the one that actually carries out a binding function, we can consider the slaked product as a mixture by weight of 1 part of binder and 3 parts of aggregate. Therefore it is not a simple binder but a ready-to-use material that does not need the addition of any aggregate.

Table 6: Granulometric analysis of the slaked lime

Sieve size (mm)	***Partial cumulative % (w/w)***	***Cumulative% (w/w)***	***Passing % (w/w)***
4	0	0	100
2	1	1	99
1.4	6	7	93
1.18	7	14	86
1	4	18	82
0.85	5	23	77
0.6	9	32	68
0.3	14	46	54
0.18	9	55	45
0.09	10	65	35
0.063	6	71	29
0.04	6	77	23
0.01	0	100	0

In summary, the particular characteristics of the slaked lime for Tadelakt can be explained by the composition of the stone used for calcination and by the traditional burning technology. As a matter of fact, the stone contains an abundant silicaticclastic fraction made of quartz, feldspars, mica, palygorskite. These minerals behave partly as aggregate (quartz, micas) and partly contribute to the formation of calcium silicates and aluminates which give the product an hydraulic behaviour. Another interesting aspect of the stone for lime is due to the presence of clastic dolomite. During burning, this mineral decomposes in CaO and MgO that after slaking change in $Ca(OH)_2$ and $Mg(OH)_2$. This last compound, on the contrary of $Ca(OH)_2$, suffers only a partial carbonation due to is low solubility. Nevertheless its presence improves the cohesion of the binder due to its felt structure caused by the fibrous shape of its crystals.

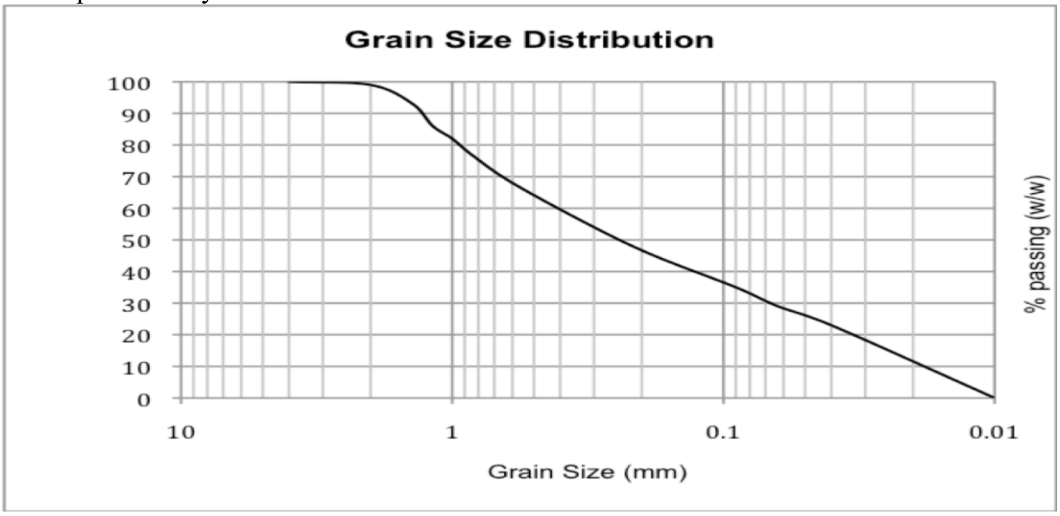
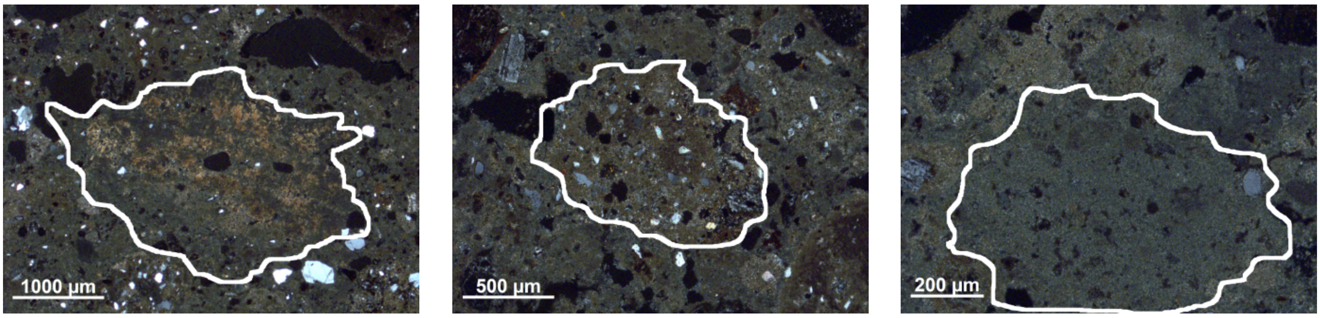


Figure 5: Diagram showing the grain size distribution of slaked lime

Concerning the effect of the traditional burning technology, we can say that it can cause the presence of a significant amount of underburnt material therefore acting as aggregate. In this underburnt fraction, it is important the presence of palygorskite which, thanks to its micro-fibrous morphology contribute to avoid the formation of shrinkage fractures in the Tadelakt plaster.

6.4 *On site Tadelakt*

The microscopic aspect of the Tadelakt plaster is quite similar to that of the carbonatic stone used to produce the lime. As a matter of fact, the presence of underburnt and overburnt fragments, together with lime lumps, can be observed. Moreover there is a large amount of quartz and feldspars grains with dimensions of 200-400 µm. The binder has the typical aspect of a natural hydraulic lime with micritic texture rich in little dark grains to be referred to calcium silicates. The macroporosity is constituted by rounded pores with dimensions 300-600 µm (Fig. 6).



a) **b)** **c)**

Figure 6: Thin section images in XPL of Tadelakt: overburnt fragment (a), underburnt fragment (b) and binder (c)

The FT-IR analysis performed to identify the compounds of reaction between the lime and the olive oil black soap (calcium salts, in particular calcium stearate) has given a negative result. It is probable that these reaction products have been leached from almost 100 years of weathering exposure. ATadelaktsample was produced in the laboratory mixing the soap and the lime putty (high-calcium air lime) and leaving the mixture to age for about a month. At the end of curing the FT-IR analysis, was performed. The comparison between the spectrum of the sample prepared in the laboratory with the spectrum of the calcium stearate reference highlights the presence of the absorption bands related to this compound (Fig.7). So, that it would confirm the hypothesis that, in the original sample of Tadelakt plaster, the calcium salt is not present because leached.

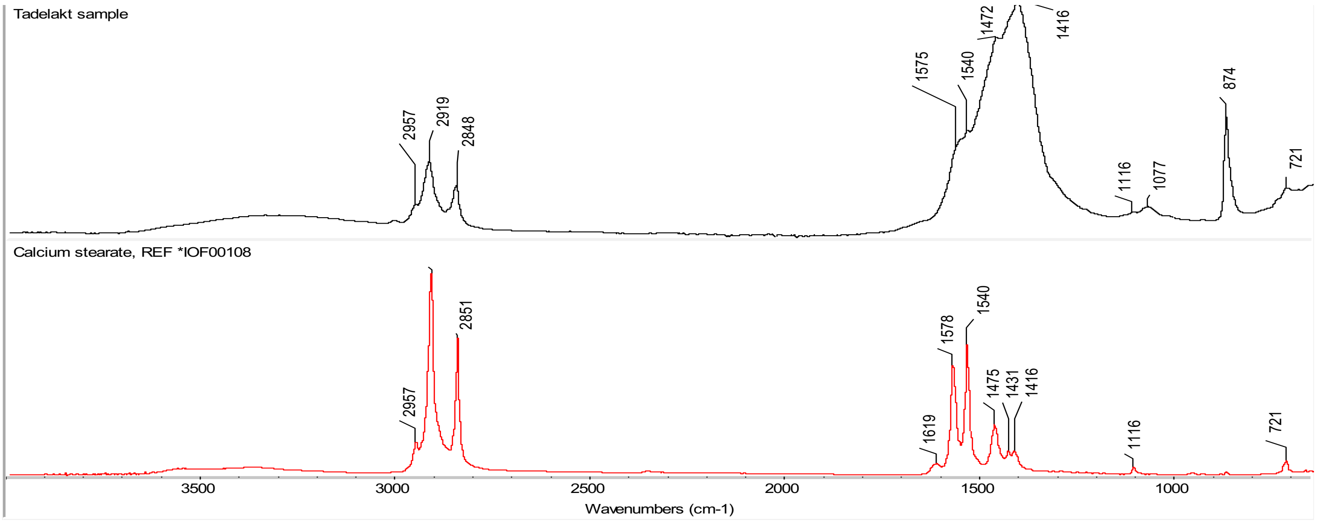


Figure 7: FT/IR spectra of Tadelakt sample (black soap/lime putty) and calcium stearate reference.

Conclusions

Tadelakt is an ancient plastering technique of Marrakesh tradition realized using a particular lime produced in the outskirts of the city. The raw material for the lime comes from carbonatic crusts of Quaternary age and it shows a composition between a marly limestone and calcareous marl rich in dolomite.
The burning of the stone in traditional kilns at quite low temperature (900-1200 °C) and the following slaking, lead to a binding product rich in $Ca(OH)_2$ and C2S which has the characteristic of a highly hydraulic lime. Nevertheless small amounts of high temperature phases (C3S, C3A, C4AF) can be present due to *hot spots* in the kiln and to direct contact of part of the stone with the flame.
The composition of the slaked lime for Tadelakt is not standardized, but varies for each calcination process.
The traditional burning causes also the presence of an underburnt fraction that coupled with the abundant silicaticclastic fraction behaves as aggregate: as a matter of fact, the slaked product is generally a mixture by weight of 1 part of binder and 3 parts of aggregate. Therefore it is not a simple binder but *a ready-to-use material* that does not need the addition of any aggregate.
Other two distinctive features of this binder are the presence of $Mg(OH)_2$ (originating from dolomite) and palygorskite (from the underburnt clay fraction of the stone). Both these crystalline phases have a fibrous structure and contribute to avoid the formation of shrinkage fractures in the *Tadelakt* plaster.
Many imitations of slaked lime for *Tadelakt* are offered by European companies but under this name we found materials based on natural hydraulic limes that have little in common with the unique compositional characteristics and performances provided by this *special* lime of Marrakesh.
As a matter of fact we have seen that from the mineralogical point of view is possible to univocally identify the original *lime* of Marrakesh, the only product that makes it possible to realize this particular plaster.

Author Contributions - To be noted: all the authors should be considered as principal authors

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