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ORIGINAL ARTICLE

Traditional gypsum renders in the Paris area: focus on a particular typology

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Abstract This study aims to characterize the "Parisian" traditional gypsum renders in order to understand its former fabrication process. A large sampling campaign covering the historical region of Paris and all building typologies from the sixteenth century to the early twentieth century was conducted. This paper presents the results obtained on the most typical gypsum render typology, which we identified as "Parisian." Cross-sections of the renders were first observed with the naked eye and then under the microscope to characterize the number, thickness, and grain sizes of their layers. X-ray diffraction analysis and mercury intrusion porosimetry were performed on each render layer and the proportion of each crystalline phase was estimated using the Rietveld method. Calcite and quartz contents are low and vary from building to building and even between two layers in a single render. Gypsum appears to be the main component as only calcined gypsum was used to prepare the renders. The different layers were made from a single coarse calcined gypsum powder, sieved at different grain sizes, and mixed with water without any addition of lime or aggregates.

Keywords Gypsum · External render · Facade · Historical monuments

1 Introduction

Gypsum is an abundant resource in the Paris area subsoil and largely used in the architecture of the region. Gypsum renders were employed in all building typologies (farms, stables, castles, churches, apartment buildings, and Paris's "hôtels particuliers") since the Middle Ages. Gypsum used outdoors is not uncommon in Europe [1–5]. With the proper techniques, gypsum renders can have remarkable physico-mechanical properties [6] and very good durability. Hitting all European countries, the technological evolution that began in the mid-nineteenth century until the mid-twentieth century led to the abandonment of the traditional building materials and techniques [7]. Since the 1980s, old gypsum renders in France are currently renovated with lime mortars or gypsum-air lime mortars [8].

Until the first half of the twentieth century, the powder, used to cast the traditional renders around Paris, is made by calcination of the gypsum rock originating from Tertiary terrains in the local subsoil

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[9]. Traditional kilns used in the past produced a powder where various calcium sulfate phases coexisted due to the heterogeneous temperature inside the kilns [10] cited in [11]. The temperature needed for the gypsum calcination is around 120 °C for the formation of hemihydrate ($CaSO_4.½H_2O$), depending on the water vapor pressure [12, 13]. Around 200 °C the hemihydrate forms the anhydrite III. This compound is easily rehydrated in a humid atmosphere and usually doesn't subsist in the powder used on site [11, 13]. Above 350 °C the calcination leads to the formation of the anhydrite II ($CaSO_4$) [12]. The products obtained by gypsum calcination react with water and recover their initial state of hydration, thus reconstituting gypsum.

Over the past ten years, there has been a resurgence of interest in buildings using gypsum renders all over Europe [2, 5, 8, 9, 14]. It has become clear that studies on the materiality of traditional gypsum renders, has become fundamental. In a previous published study [15], the authors have presented an overview of the diversity of the gypsum renders in the Parisian region based on a corpus of 56 old renders dating from the 16th to the early twentieth century sampled on 33 buildings. Old gypsum render in the Paris area could be pigmented or not, imitating brick or stone, constituted in one coarse coat or in several layers (until 5 coats per render). Various textures and types of surfaces were also described (roughcast, brushed, smooth…). From the buildings and the renders' description, a typology distribution was proposed (brick imitation, stone imitation, rural, multicoat-pigmented…). The performed analyses brought information on the mineral composition, and the water to powder ratios used to cast each coat of the renders. But with the diversity of the samples presented, several questions still remain about the fabrication process "by the book."

In this paper, a focus is made on a part of the corpus representing 16 renders. They are part of the most represented render typology in the corpus, identified on the most prestigious urban monuments and have usually respected the same and constant characteristics over time, such as described in architectural treatises [16, 17]. In the present paper, this particular render typology is called the "Parisian" typology. A focus on these renders allows a better understanding of the traditional standard practices and their impacts on the render characteristics.

2 Material and methods

2.1 Corpus

The corpus consists of render samples collected from buildings constructed in a period of time ranging from the sixteenth to the early twentieth century in a region of 70 km around Paris. The renders could be dated with the help of land registers, historical investigations or documentation on past renovation works [4]. In the first paper [15], the number of renders analyzed is 54, originating from 33 buildings. A lot of renders were composed of different layers. Each layer corresponds to a coat which was applied using a specific mixture of powder and water.

The current paper focus on 16 renders sampled from 11 buildings of the Paris region (Table 1). More information, including historical, environmental architectural and analytical details on the buildings studied can be found elsewhere [8, 15, 18].

The sampling is performed with an angle grinder or with a hammer and a chisel. Each sampling must reach the support to include all the render's coats. The size of the collected samples ranges from approximately 10 × 10 cm^2–40 × 40 cm^2. Collecting large samples was possible because the sampling campaign was often conducted during the renovation of the facade just before the renders were demolished. In fact, the sampling was the only way to preserve some parts of the original material. The sampled render is dry cut in order to make a cross-section showing the entire render stratigraphy. The overall render thickness, the number of constitutive coats, and their thickness and color are measured.

2.2 Visual analysis

The measurement of each coat thickness and the estimation of the grain sizes is performed with a stereoscopic-microscope Leica Wild M10. To complete the grain identification, thin sections and polished cross sections are prepared on a selection of coats, and are observed within transmitted and reflected light using a digital microscope 3D type Keyence VHX 5000.

Table 1 Buildings and renders description

Building					Facade	Render	Render estimated dating
Name	ID	Typology	City	Date			
Château de Montépilloy	CMP	Castle	Montépilloy	XIIIth, XVth and XVIIth	CMP-F1	CMP-F1-E2	1650
Hôtel Amelot de Bisseuil	HAH	Mansion (Hôtel particulier)	Paris	1660	HAH-F1	HAH-F1-EZ2	1660
Hôtel Marquelet de la Noue	HMN	Mansion (Hôtel particulier)	Meaux	1660	HMN-F1	HMN-F1-E2	XVIIIth
Hôtel du Petit Contrôle	HPC	Mansion (Hôtel particulier)	Versailles	1723	HPC-F1	HPC-F1-E3	1723
Château de Méréville	MER	Castle	Méréville	1784	MER-F1	MER-F1-E1	1784
Folie Huvé	FHuV	Mansion	Meudon	1792	FHuV-F1	FHuV-F1-E1	1792
Marché aux bestiaux	MBS	Market	Sceaux	1673	MBS-F1	MBS-F2-E3G	XIXth
						MBS-F2-E3J	XIXth
20 rue Saint Honoré	VHO	Apartment block	Versailles	1830–1880	VHO-F1	VHO-F1-E1	1830–1880
						VHO-F1-E2	1830–1880
						VHO-F1-E3	1830–1880
Château de Goussainville	GVC	Mansion	Goussainville	1860	GVC-F2	GVC-F2-E1	1860
Atelier Lorenzi	ALA	Apartment block	Arcueil	1871	ALA-F1	ALA-F1-E1	1871
					ALA-F2	ALA-F2-E1	1871
Théâtre de la Renaissance	TR	Théâtre	Paris	1873	TR-F1	TR-F1-E1	1873
						TR-F1-E3A	1873

2.3 Scanning electron microscope observations

The images presented are constructed from the emission of secondary electrons realized with a Scanning Electron Microscope JEOL 5600LV. The analyses are carried out in ‘High Vacuum’ mode (15 kV and 20 kV acceleration voltage, 38 mm working distance).

2.4 X-ray diffraction (XRD)

An X-ray diffractometry is performed on each coat with a Bruker D8 Advance diffractometer (Co anode, LinxEye Super Speed detector). A representative part of approximately 3 cm^3 is crushed into a powder passing through 100 µm sieve. The standard conditions are as follows: 40 kV, 40 mA, 5–64° 2θ, acquisition time 1 s, step width 0.02°. The interpretation is obtained by comparison with the JCPDS database diffractograms, sections. 1–83. Quantification of identified crystalline phases is estimated using a Rietveld refinement (program TOPAS, Bruker).

2.5 Calcimetry

To corroborate the calcite contents obtained by the Rietveld refinement, the coats without dolomite are tested in duplicate with a volumetric calcimeter (Bernard calcimeter). In this method, the carbonates are treated with excess acid, and the carbon dioxide emitted by the reaction is determined volumetrically [19]. A sample of the coat is crushed into powder passing through a 100 µm sieve, and then dried until constant weight. The calcite content previously estimated on each sample with the Rietveld refinement is used to calculate the quantity of powder to be sampled: as a matter of fact, the sample should contain approximately 0.4 g of pure calcite to get reliable and reproducible calcimetric results. The powder is then dissolved in hydrochloric acid at 14.5% with the addition of 5% of calcium acetate. With the temperature and the atmospheric pressure known, the ideal gas law allows the estimation of the molar content of carbon dioxide. As calcite is the only carbonate in the selected coats, a calcite content estimation is possible. For each coat that does not contain dolomite, the calcimeter measurement is done, in duplicate.

2.6 Mercury intrusion porosimetry

On a selection of coats, a 1 cm^3 sample is cut and dried at 40 °C until constant weight. If the coat contains large grains, only the matrix is sampled. Mercury Intrusion Porosimetry (MIP) is performed with an Autopore IV 9500 porosimeter, controlled by the software AutoPore IV version 1.07.

3 Results and discussion

3.1 First observations and identification of the Parisan typology

The observation of cross sections shows that the Parisan renders is composed of 2–3 layers containing grains of different sizes dispersed into a white matrix (Fig. 1). The observation of the layers, their position in the render, their thickness and the grain sizes, allows a categorization of the coats [15]: the first layer is the so-called leveling coat which is meant to equalize the support irregularities (rubble or brick masonry/timber-framed wall) in order to even the surface out. The second layer, called scratch coat, may be quite thick (1–6 cm) and usually contains grains up to 10 mm large. The finishing coat is the final and therefore superficial coat of the render and contains grains smaller than those of the scratch coat. The Parisian typology is characterized by the systematic presence of one scratch coat and one finishing coat. The leveling coat is present if necessary on account of the surface irregularities of the substrate. All coats are not pigmented, and the render surface is always smooth.

The Parisian render is found on urban or prestigious buildings (castels, mansions, hôtels particuliers, etc.). This render typology seems to follow the traditional technology of gypsum renders construction described by A–C. d'Aviler [16] (Volume 2 item "Plâtre" page 795), in the Encyclopédie de Diderot et d'Alambert [20] (Volume XII item "Plâtre" page 753) or by J-R Lucotte [17] the first to describe the gypsum renders applications in detail. The render is meant to consists of at least 2 non-pigmented coats that we identified as a scratch coat, and a finishing coat (Fig. 1). According to these sources, the scratch coat is prepared with a powder obtained by sieving the material through a basket, the smaller grains being removed from the powder by another sieving through a "sas", a sieve made of horse hair. The treatises [16, 17] or the encyclopedia of Diderot et d'Alambert [20] do not specify the size mesh of the "sas", neither the ones of the basket.

The finishing coat is prepared with the fraction of the powder passing through the "sas". This coat has a smooth surface, which is obtained by a specific action on the coat surface while it is still not completely set: the coat is "coupé" ("cut") with the sharp side of the Berthelée trowel.

According to the dating estimations of the "Parisian" renders, this fabrication process appears remained the same since the XVIth until the end of the XIXth century. The earliest renders most probably follow know-hows which were either transmitted orally or on treatise we have not identified.

In the Paris Area, a lot of buildings or renovation works are dating from the XIXth century; the sampled renders from this period are the most numerous. In addition, the number of sampled Parisian renders is not

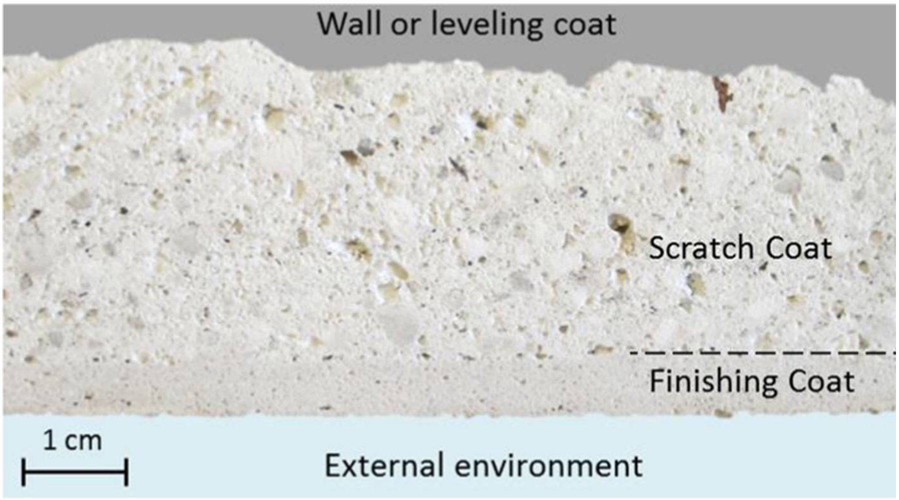

Fig. 1 Render cross section with two coat types. Render TR-F1-E3A, sampled from the building "Théâtre de la Renaissance" in Paris

necessarily representative of the original proportion of this render typology in the historical buildings. The best preserved and best documented buildings of the corpus are the prestigious ones, which are usually with a Parisian render. This must be taken into account to assess the representativeness of the corpus, particularly with regard to dating and typologies.

3.2 Stratigraphy

Observations tend to confirm the use of powders with a specific granulometry for the production of each type of coat. The scratch coat systematically contains coarser grains than the finishing coat, the grain size of which is less than 0.5 mm (Fig. 2).

With the exception of one render, the scratch coat is thicker than the finishing coat (Fig. 3). The scratch coats are 25 mm thick on average with a significant variation between coats, the standard deviation is 10.9 mm. For the finishing coats, the particular case (HMN-F1-E2) is excluded from the calculations. Then, the average thickness of a finishing coat is 6 mm with a standard deviation of 2.6.

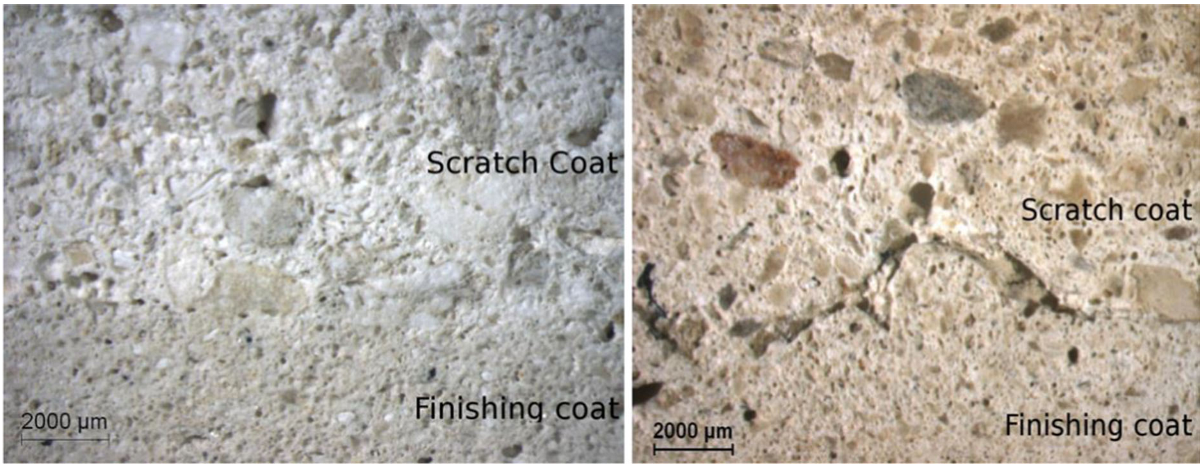

Fig. 2 Interface scratch coat-finishing coat observed on a cross section under the stereo-microscope, "Théâtre de la Renaissance" render TR-F1-E3A (left), "Hôtel Amelot de Bisseuil" render HAH-F1-EZ2 (right)

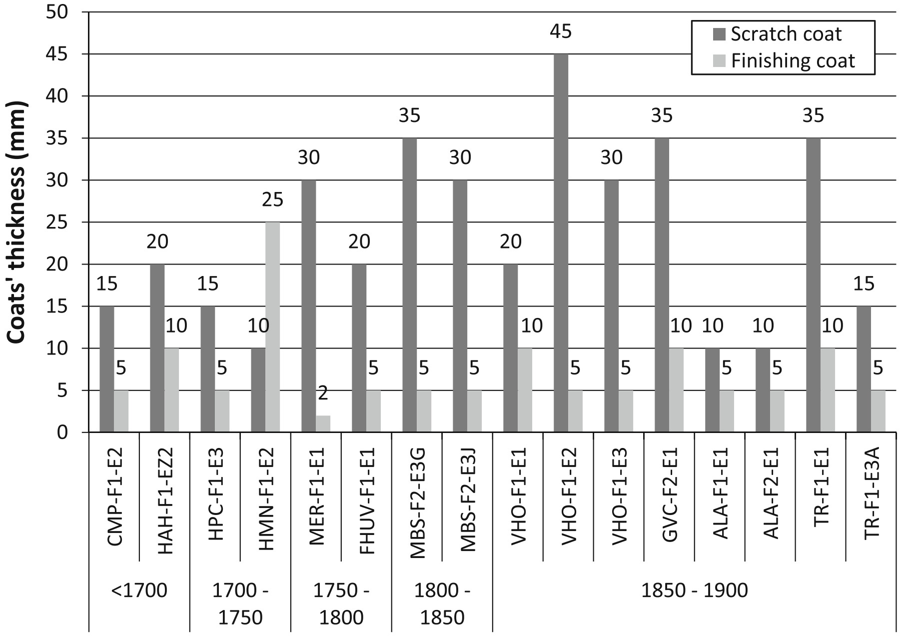

Fig. 3 Thickness of each renders' coat, according to their dating

3.3 Coats composition

Scanning Electron Microscope observations of the coat matrix shows a characteristic microstructure formed by entanglement of needle shape gypsum crystals (Fig. 4). The crystals are less than 1 µm in thick and around ten microns long.

The gypsum matrix is characterized by the presence of spherical cavities corresponding to air bubbles trapped into the paste (Fig. 5). This feature is common and quite representative of the gypsum renders.

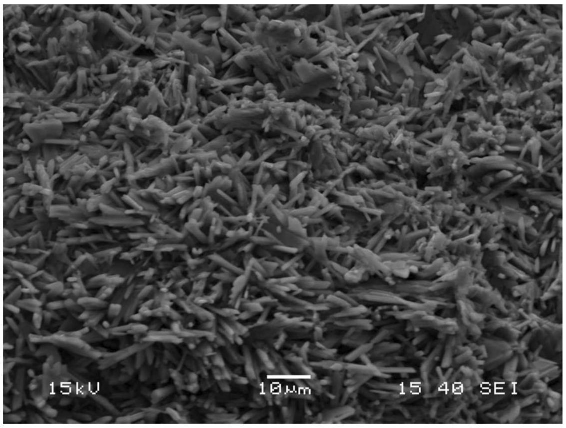

Fig. 4 SEM observation of the gypsum matrix in the finishing coat of HAH-F1-EZ2

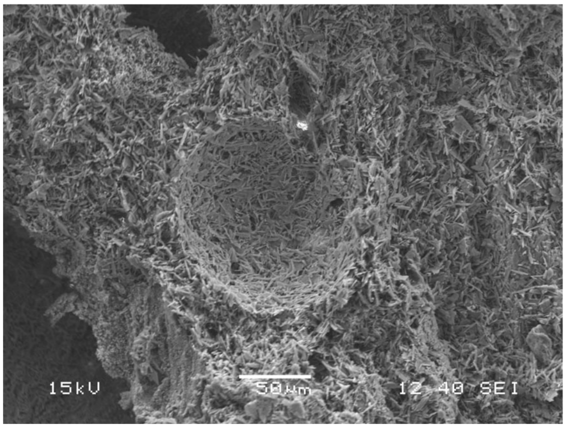

Fig. 5 SEM observation of an air bubble in the gypsum matrix in the scratch coat of HAH-F1-E2

The microstructure of gypsum renders is known to show variations that can provide important information about the fabrication process and exposure conditions of the renders: the gypsum crystals could have more flattened or less elongated shapes in relation to their crystallization kinetics, especially when some additives have been used as retarders during the casting (citric acid, tartaric acid…) [6, 13, 21]. The crystal morphology may also be affected by the wet/dry cycles due to their exposure to the weather [6]. The homogeneity of crystal size and shape suggests that none of these factors have affected the microstructure of the samples.

Table 2 summarizes the mineral composition obtained by the Rietveld refinement method on each coat types. With a proportion higher than 85%, gypsum is definitely the main component of the scratch and finishing coats. On average, calcite and dolomite constitute respectively 7% and 5% of the scratch coats and both of them, 3% of the finishing coats. The gypsum rocks in the Paris Basin subsoil naturally contain calcite. The purest gypsum quarries in the Paris area can contain less than 2% of calcite (first stratum in Cormeilles-en-Parisis [22]) but in some quarry sites and in some stratum the calcite content can reach 13% [23, 24]. All the coats, both scratch coats and finishing coats, having more than 7% dolomite are part of a single render sampled on a building located in the South-Western part of the Paris region. We believe that this particularity may be linked to a local gypsum or mineral resources or specific construction technique.

Dolomite contents lower than 3% are difficult to estimate due to the proximity of the gypsum and dolomite XRD peaks and the relative littleness of the dolomite peaks in comparison to the gypsum ones. With the cobalt anode, the main dolomite peak appears at $2\theta = 36.14°$ (*d*-spacing 2.886 Å) and one of the gypsum peaks appears at $2\ \theta = 36.35°$ (*d*-spacing 2.87 Å). With a dolomite content around 3% and a gypsum content around 85%, the dolomite main peak is about 5 times smaller than the neighbor gypsum peak.

Variations inside the same type of coat are high. Considering the values of the standard deviations, average differences between the scratch and the finishing coats are not clearly significant. But, if one considers the two coats of the same render, clear differences appear in their calcite content. This phase is always more abundant in the scratch coat than in the finishing coat. The same observation can be made for the dolomite containing renders. In order to evaluate more precisely the calcite content, we duplicated its quantification using the calcimetric method. This was done only on samples that did not contain dolomite. We found out that calcite contents measured by the calcimeter are close to the ones obtained by Rietveld refinement (Fig. 6).

3.4 Grains identification

The same types of grains are found in the scratch coat and the finishing coat, only the grain size is different.

Visual observations at naked eyes or under the microscope show two main types of grains in the coats: black grains and saccharoidal grains, i.e., grains having a microstructure similar to the one of brown sugar.

The black grains are remnants of combustible materials. In most layers, these grains are coal, recognizable by their typical wood microstructure (Fig. 7, left). There is one exception, the “Théâtre de la Renaissance” render TR-F1-E3A, wherein the black grains appear to be porous, bright and hard. The combustible used here seems to be coke (Fig. 7, right). Black grains will be referred as combustible residues.

The saccharoidal grains are constituted of gypsum crystals agglomerates. Each gypsum crystal has a lenticular shape with a diameter of several hundreds of micrometers (Fig. 8). Micrite (microcrystalline

Table 2 Proportion of crystallized phases of each coat type according to Rietveld refinement method

		Crystallized mineral content				
		Gypsum (%)	Calcite (%)	Dolomite (%)	Quartz (%)	Anhydrite (%)
Scratch coat	Average	85	7	5	2	1
	Standard deviation	7	3	5	3	1
	Median	85	7	4	2	0
Finishing coat	Average	91	3	3	1	1
	Standard deviation	4	1	3	1	1
	Median	91	3	2	1	0

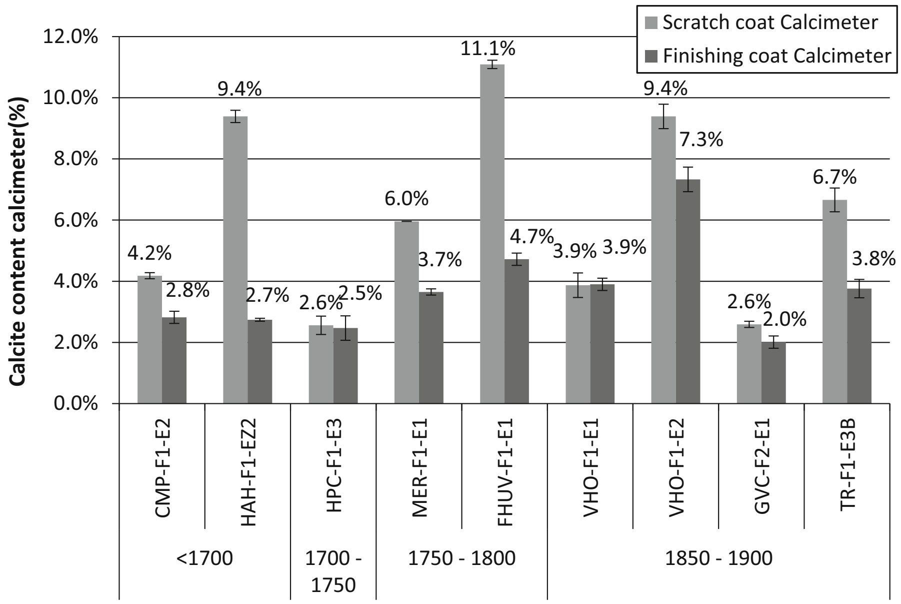

Fig. 6 Calcite contents of renders' coats, measured by calcimeter, according to their dating

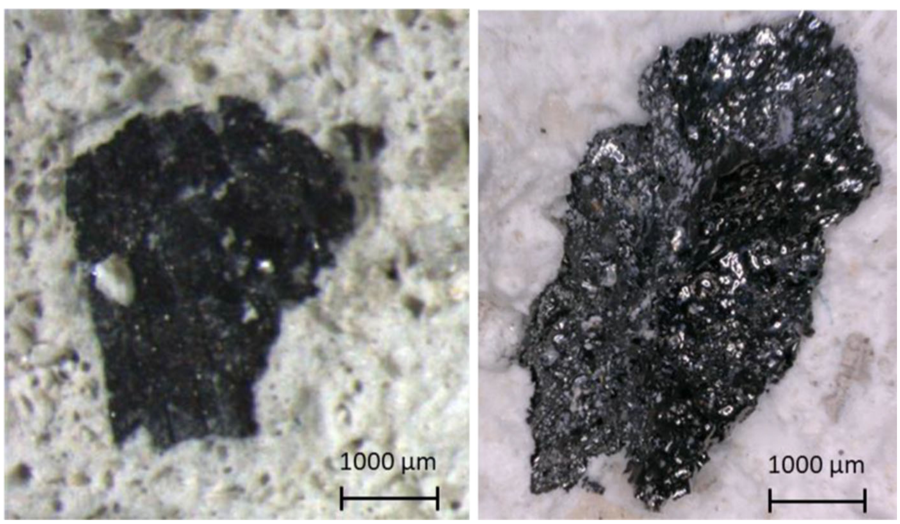

Fig. 7 Combustible residues from the scratch coat of MBS-F2-E3G (left) and TR-F1-E3A (right). The first one is a coal fragment, the second one is a coke fragment

calcite) can be found between the gypsum crystals. These grains will be referred as saccharoidal gypsum agglomerates, even if some micrite is also present.

Scanning Electron Microscope observations allows a clear differentiation between the gypsum matrix and the saccharoidal gypsum agglomerates even if the crystal nature is the same. The gypsum crystals in the saccharoidal particles are much larger than the 10 µm needle shape gypsum crystals in the matrix, they are not elongated and their cleavage faces are easily recognizable under the optical microscope (Fig. 9).

Difficult to differentiate from the matrix, even under optical microscope observations, polarizing microscope observation of thin sections allows to clearly identify anhydrite II particles (Fig. 10). The anhydrite II under unanalyzed polarized light is colorless and transparent. The relief is weak, however, slightly higher than that of gypsum. Under cross-

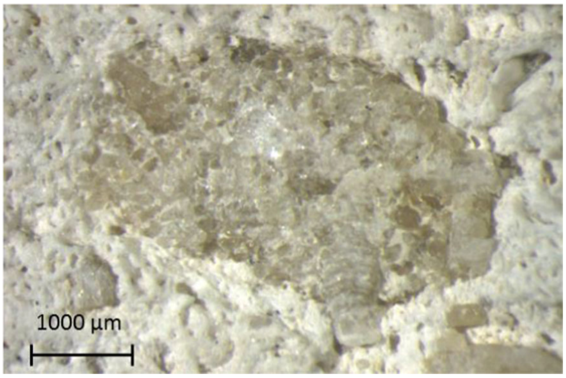

Fig. 8 Saccharoidal gypsum agglomerate from the scratch coat of MBS-F2-E3J

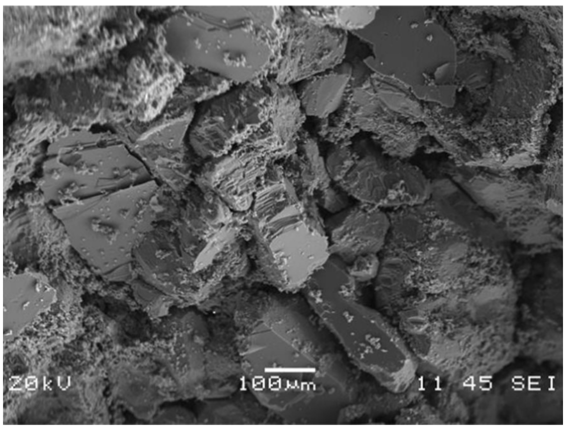

Fig. 9 SEM observation of a saccharoidal gypsum agglomerate in the scratch coat of TR-F1-E3A

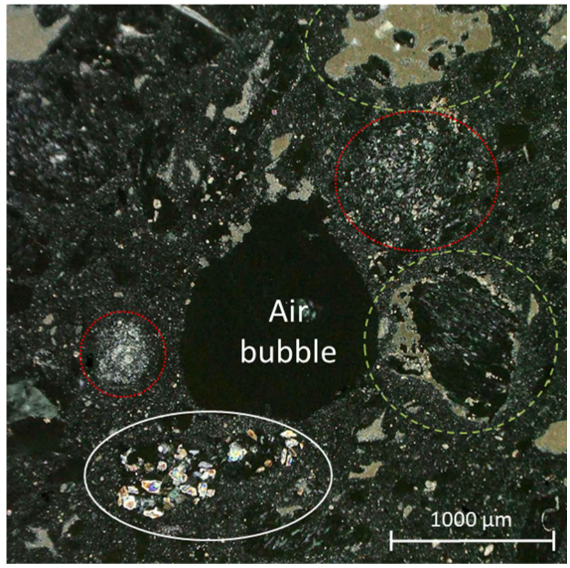

Fig. 10 Thin section image of the scratch coat of TR-F1-E3A under cross-polarized light. Three types of grains can be observed: a polycrystalline grain of highly birefringent anhydrite II (white circle), two polycrystalline gypsum grains with a gray birefringence (red dotted circles), and micritic calcite with its typical brownish color, surrounding gypsum monocrystals (green dashed circles). (Color figure online)

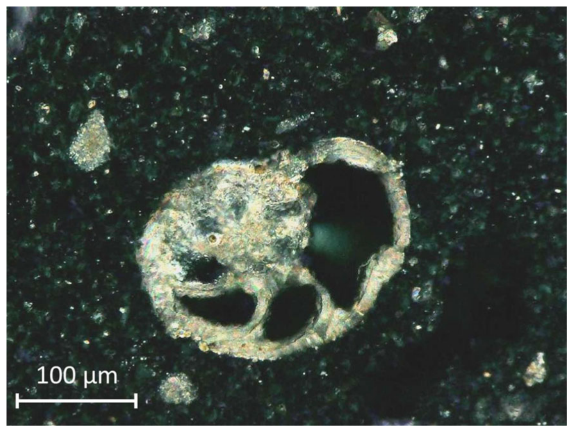

Fig. 11 Thin section image of the scratch coat of TR-F1-E3A under cross-polarized light. Observation of Miliolidae in the gypsum matrix

polarized light, the birefringence is high, with vivid second order shades. Anhydrite II is not frequently encountered (less than 1% on average identified with the XRD analysis) and the larger crystals are less than 1 mm.

In the following figure (Fig. 10), gypsum crystals, sometime surrounded by micrite, are also visible. The lenticular shape of the gypsum grains, their size and their association mode is clearly the same as the one of the saccharoidal gypsum agglomerates.

Calcite and dolomite residues are also present as dispersed particles in the gypsum matrix. Fossilized shells of micro-organisms such as Miliolidae have been found in TR-F1-E3A (Fig. 11). These microfossils are typical of some Lutetian limestone of the Paris Basin (the so-called"calcaires à milioles"). This means that at least a part of the calcite in this coat originates from crushed limestone, probably accidentally added due to their rare occurrence, only two microfossils have been found in this render (50 cm^2 were observed).

The scratch coat of FHUV-F1-E1 contains rounded gravels 1 cm large. This coat has the highest calcite content. The XRD analysis of the gravels shows a mineral composition made of quartz and calcite.

Those huge gravels have clearly been added deliberately in the coat recipe. This is the only coat that seems to be the case.

Quartz can occasionally be found, with content equal or lower than 4%. The presence of this mineral seems to be accidental, due to impurities added during the powder manufacturing process. Only one render (CMP-F1-E2) has a higher quartz content: 14 and 5% in respectively the scratch and finishing coat. In this render, silica sand seems to have been added to the scratch coat on purpose.

3.5 Porous medium properties

Mercury Intrusion Porosimetry allows the estimation of the accessible porosity and the pore size distribution. In all coats analyzed with this technique, the pore size distribution is unimodal: the major part of the mercury penetrates in the sample by pores with closely grouped opening sizes. This threshold radius ranges from 0.6 to 1.7 µm depending on the coat. This poral distribution is commonly found in pure gypsum plasterwork [25]. The accessible porosity ranges from 34 to 49 vol% depending on the type of coat (Table 3).

No correlation can be made between the accessible porosity and the threshold radius. No significant differences or tendencies are observed between the scratch coats and the finishing coats. But the variations between the scratch coats are smaller than the variation between the finishing coats. For the scratch coats, the porosity varies from 38 to 46%. For the finishing coats, the porosity varies between 34 and 49%.

The water/powder ratio (W/P) used during the mixing determines the final porosity of a gypsum coat [26]. If we suppose that pure hemihydrate ($CaSO_4 \cdot H_2O$) is used to make a coat, the W/P stoichiometric ratio should be equal to 0.186. In reality, to obtain a suitable rheology for practical use, the W/P ratio must be much higher in the fresh paste. The volume occupied by this residual water in the fresh paste is considered to be exactly equal to the porous space volume of the material in its final state, i.e., once set and after the evaporation of the residual water. Considering this hypothesis, the porosity of old gypsum render gives information about the water/powder ratio used [27].

The real W/P ratio with a powder containing anhydrite II, underburnt gypsum or impurities is different from this theoretical estimated W/P ratio. But according to the composition usually found in the traditional powder (composed by more than 70% of hemihydrate [10, 11]) the difference between a W/P calculated from the porosity and the real W/P ratio is estimated to be less than 5%.

Table 3 Porous characteristics of each analyzed coat and estimation of the mixing ratio

Identification Building-Facade-Render	Type of coat	Accessible porosity (vol%)	Threshold radius (µm)
HAH-F1-EZ2	Finishing coat	47	1.06
	Scratch coat	40	0.95
HPC-F1-E3	Finishing coat	35	1.07
	Scratch coat	41	0.95
FHUV-F1-E1	Finishing coat	45	0.68
	Scratch coat	38	1.07
MBS-F2-E3J	Finishing coat	49	1.68
	Scratch coat	46	1.68
TR-F1-E3A	Finishing coat	34	0.6
	Scratch coat	41	1.33
Finishing coat	Average	42	1.08
	Standard deviation	18	0.41
Scratch coat	Average	41	1.28
	Standard deviation	3	0.39
Total	Average	42	1.2
	Standard deviation	5	0.38

Modern coarse calcined gypsum powders are mixed with Water/Powder ratio around 0.6 or 0.7 for the casting of renders. These W/P ratios lead to a final porosity of 45% to 50%. Some coats of the old renders in our corpus have a much smaller porosity. It seems that they have been made with a significant smaller W/P ratio.

Other factors than the W/P ratio, like weathering alterations, could explain the relative small porosity in some old coats. The porosity of a neo-formed gypsum matrix, made of entanglement of needle shape gypsum crystals is correlated to the density of the microstructure. Some authors [6] attribute the dense microstructure to dissolution and recrystallization processes caused by wet-dry cycles taking place during long-term weathering. Such processes, in order to induce an overall decrease in porosity of several percent, would result in a significant reduction in the render's apparent volume, and would greatly affect its appearance. In this study, all coatings were sampled in a sheltered area, as it was necessary to ensure that they were in good conservation conditions. Their surface was smooth and devoid of any features characteristic of dissolution by water. In addition, if dissolution–recrystallization had occurred, the finishing coat should have been more affected than the scratch coat. In our study, most of the finishing coats are more porous than the scratch coats (3 out of 5 cases). The coats' low porosity does not appear to be mainly due to these processes.

To explain the small porosity of old gypsum renders our main hypothesis is based on the old calcined gypsum powder characteristics who allowed the use of small W/P ratios.

In a previous study, some traditional gypsum renders were reconstituted with powder made from gypsum rock using artisanal calcination processes and coarse grinding [28]. The suitable Water/Powder ratio used was around 0.45, the final porosity was 37%, which is in line with the data of our corpus.

This tends to prove that the manufacturing process and/or the implementation of the traditional gypsum renders are responsible for their low porosity. Two main explanations can be proposed.

The traditional calcined gypsum powder is not composed of pure hemihydrate but also contain anhydrite and underburnt gypsum (saccharoidal gypsum agglomerates). Theoretically, to a given W/P ratio, an addition of 10% of anhydrite II in the powder reduces the final porosity by 1%. Saccharoidal gypsum agglomerates are less porous than the needle shapes gypsum crystal matrix, if they are in large quantities they can contribute to reducing the render's global porosity. But the content of underburnt gypsum in old calcined gypsum powder is estimated by some authors to 10% [10, 11], which contribute to reducing the final global porosity by less than 2%.

From our point of view, the main factor to explain the small porosity of some old gypsum renders is the use of a small W/P ratio which is possible thanks to the granulometry of the powder. The traditional calcined gypsum powder is made by crushing the heated gypsum block after the calcination with a wooden staff [16, 17]. The powder obtained is coarse with a relatively small specific surface compared to modern calcined gypsum powders [28] and then, it can be mixed with a small W/P ratio while maintaining a suitable workability. Additives could also have been used, acting as water reducers.

3.6 Reconstitution of the manufacturing process

The results obtained in this study should be interpreted in relation to the historical documentation that we can have about the traditional manufacturing process. Our analysis formally confirm some of the widespread accepted knowledge but also contradict some preconceptions about gypsum renders.

Old architectural treatises or encyclopedia [16, 17, 20] describe the theoretical fabrication process of the calcined gypsum powder in the Paris area and the renders' implementation on sites. But some of them are in contradiction between them, and a gap could occur between the theoretical recommendations and the site reality [8]. Many studies in the 1950s [10, 11] proposed analyses of the calcined gypsum obtained in traditional kilns. In the second half of the twentieth century, most suppositions were made about the implementation and the composition of traditional gypsum renders based on few studies. The main issue in the reconstitution of the traditional fabrication process of gypsum render has been whether or not air lime and aggregates were added. The present study based on a representative corpus brings out some precious data to understand the traditional calcined gypsum powder-making process and the implementation of the traditional "Parisian" gypsum renders typology.

3.7 Traditional calcination process

Traditional kilns produced a calcined gypsum powder where various calcium sulfate phases coexisted due to the heterogeneous temperature inside the kilns [10, 11]. Our analysis confirms this heterogeneity by the identification of underburnt gypsum (saccharoidal gypsum agglomerates) and anhydrite II in the renders. The underburnt gypsum presence indicates an incomplete calcination of gypsum rock at a temperature less than 120 °C. While anhydrite II presence means a gypsum calcination process at more than 350 °C. Thus, in a traditional kiln, the temperature varies from less than 120 °C to more than 350 °C depending on the zones.

The identification of the combustible residues also gives valuable information about the calcination process. The fuel used in traditional kilns, such as the "four culée", was wood, coal or coke. In a "four culée" and usually in traditional kilns, the gypsum blocks are directly exposed to the burning fuel. Remnants of combustible materials can still be found after the calcination: most renders still contain coal or coke. The presence of coke inside a coat means that the render was cast during the nineteenth century or the beginning of the twentieth century.

3.7.1 Consequences of the traditional grinding process

After the calcination of the gypsum rocks, the traditional grinding was done with a wooden stick [16, 17, 20]. This grinding method led to a coarse powder with content variations between the different granular fractions of powder: logically, the harder the minerals, the coarser the particles were supposed to be. Therefore, the coarser fraction of the powder was expected to contain more minerals such as calcite or dolomite, minerals unaffected by the calcination, which have a higher hardness than the hemihydrate or anhydrite originating from gypsum.

3.7.2 Implementation process

The stratigraphy observations, the grain sizes differences between the scratch coats and the finishing coats are in accordance with the old architectural treatises [16, 17, 20]. The finishing coats seem to have been made with grain size fractions smaller than 0.5 mm. While the scratch coats can contain grains larger than several centimeters. These grain size observations make it possible to estimate the mesh size of the "sas" at about a half or a third of a millimeter. Some reconstitutions of a sieving with a basket [28] give a powder with a maximum grain size of 2 cm or 3 cm similar to the observations in the scratch coat of old renders.

In this study, it was also observed differences in the mineral contents between the scratch coat and the finishing coat in the same render. The scratch coats made with the coarser grains are richer in dolomite and calcite compared to the finishing coats made with the smallest grains.

It can be attributed to the sieving of the powder used to cast the coats, as the traditional calcined gypsum powder is expected to have a heterogenous composition depending on its grains size.

To test this hypothesis, it is sufficient to reconstitute a render made out of powders with two different particle size distributions following the recipe described in old architectural treatises or encyclopedia (eighteenth century) [16, 17, 20]. This is what we did in a previous study [28]. We calcined gypsum blocks in the traditional way, then grinded them with a stick, and finally sieved the powder using the basket/sas method. We could show that in the experimental render made from these sieved powders, the scratch layer is indeed richer in calcite than the finishing layer. [29].

The calcite content in the old gypsum renders is similar to the one in the gypsum quarries in the Paris area (less than 10%). The matrix is constituted by needle gypsum crystals, only few calcite particles have been observed. As observed (Fig. 10) calcite is mainly present under a micritic form around gypsum crystals, corroborating the main origin of calcite as impurity of the raw material. In the same way, the quartz content in most of the old gypsum renders (less than 4%) indicate than in the large majority of cases, no aggregates were added during the implementation. Saccharoidal gypsum agglomerates, which are underburnt gypsum brought by the powder, act as the role of aggregates but they are not intentionally added.

There are two exceptions: the scratch coat of FHUV-F1-E1 which contains rounded gravels, and the render CMP-F1-E2 whose the scratch coat and the finishing coat contains respectively 14% and 5% of quartz. Additions of aggregates or filler were not recommended in the old treatises but it could be used to reduce the cost of materials. This practice was a controversial [8]. Some impurities could have been added involuntarily, for example during the grinding process which was usually made directly on the ground in the quarry, next to the kiln or even in the construction site [8].

The accessible porosity measurements also provide valuable information about the implementation process. As the accessible porosity can be linked to the Water/Powder mixing ratio, W/P doesn't seem to be specific to a type of coat: the ratio used for the finishing coat is not necessarily bigger than the one used for the scratch coat. The porosity values suggest than some of the coats were mixed with a relatively low W/P ratio, around 0.45 for the less porous coat (with a porosity of 34%).

In contradiction with some widespread ideas, our work shows than the traditional "Parisian" gypsum renders typology seems to have usually been manufactured only with the coarse powder mixed with water, without any lime or aggregates. The powder was a coarse granulometry and was composed by various calcium sulfate phases and impurities and was only made from gypsum rocks calcinated in the traditional kilns, such as the "four culée".

3.8 Gypsum renders durability in outdoor environment

As gypsum is soluble in water (2 g/L at 20 °C), its use outdoor is not commonly admitted. Modern practices often prefer to replace gypsum renders by a gypsum-lime mortar, or even a pure lime mortar. In our study the good performance of gypsum renders appears to be mainly due to the low porosity, and then dense microstructure compared to modern gypsum materials. A low porosity limits the water transport into the microstructure and gives a higher water resistance [6]. The poral distribution is unimodal, centered around a large pore radius (around 1.2 µm on average). Then, traditional gypsum renders don't block the vapor transport, letting water come out. Allowing the render to "breathe" could be the main reason for the traditional finishing treatment, "cut" with Berthelée trowel. This is crucial as wood is usually present in the old building and water stagnation in the walls could be deleterious.

Mechanical properties of gypsum based materials are generally in direct relationship with the porosity: the less porous the gypsum material, the better the mechanical strengths [13]. But more mechanical characterizations are needed.

Anhydrite II reacts slowly with water and can still be observed in several hundred old renders. Its presence in the renders could confer better behavior to water penetration, as it reacts to form more gypsum.

The durability of traditional gypsum renders outdoor is also due to the building architecture [8]. The design of the facade is indispensable to avoid rainwater flowing over the facade: cornices on top, string-courses at every floor, and external window frames [4]. For the prestigious buildings, the facade base is in ashlar preventing the ground water from reaching the gypsum render by capillary action.

4 Conclusion

The traditional gypsum renders are an important part of the cultural heritage of the Paris area. Their qualities allow them to face several centuries. A better understanding of their manufacturing was essential. This study brings confirmation that the making process describe in the archives matches the reality of the prestigious monuments renders. As the previous work [15] showed it, no lime was added. It appears here that generally no aggregates were voluntary added either (even if some exceptions could be encountered). The focus on the Parisian typology highlights the influence of the traditional calcination, grinding and sieving processes on the renders characteristics, particularly the coats' composition and porosity.

Some conservation solutions need to be created, based on these results. But, to develop compatible and durable repair materials, additional work is needed, especially for a better understanding of old renders' mechanical and water transfer properties. In a precedent study [29], we performed measurements of dynamic modulus of elasticity, flexural strength, capillary absorption, and water vapor permeability on reconstitutions of traditional renders. The same kinds of analyses should be performed on the old render samples we have collected. The results will also help for a better comprehension of the interactions with the old structures.

Equally important, some communication for their valorization must be done to preserve the last witnesses of this forgotten artisanal know-how.

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Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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