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Building material and production analysis of the industrial lime kiln (19th C.) excavated in the valley of La Panouse, Marseilles.

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Abstract

An archaeological excavation combined with the study of buildings was conducted on the ruins of an industrial lime kiln of the Panouse valley dating from the second half of the nineteenth century and located in the new Calanques National Park. The site is of particular interest since are associated with the furnace, the limestone quarry supplying the raw material and the grappier - made of combustion residues. This massive short flame continuous furnace presents a major development of the activity of lime burner already very present in this valley. The objectives of this study were to identify the different phases of construction of the kiln and its dependencies, understand its working and analyze its production. One hundred samples including renderings and sealing mortars of stone and brick masonry were taken from different parts of the kiln to identify the materials used and their implementation and try to establish a relative chronology of the construction. The marly limestone quarry, the grappier and some lime remains found in the kiln and in the slaking tank were also sampled and analysed.

The lime kiln site of the valley of the Panouse is a major industrial complex set in the hills of Saint-Cyr Carpiagne in the National Park of the Calanques in Marseilles. The valley contains the industrial furnace, the oldest temporary furnaces, quarry, combustible, cooking residues: slag and grappier and annexes : house-tank and stables (Fig. 1).

The massive short flame and continue cooking kiln, shows a major evolution of the lime burners craft that was already present in the hills.

An archaeological evaluation and study of the building were carried out during restoration work of the kiln. This article describes the lime kiln and presents the results of analyzes of mortars and plasters samples of the building, lime production, combustible and cooking residue. It seeks to demonstrate the contribution of physical and chemical material analysis to the knowledge of the building and of the kiln production.



Fig. 1 Valley of the Panouse: quarry, lime kiln and grappier (cl. D'Ovidio A-M)

1 Analysis methods

A particle size study was carried out by dry sieving (mesh size 8 mm to 0.063 mm) (method of H. Jedrzejska) after dissolution of some samples with hydrochloric acid dilute at 10%. The study clarified the particle size distribution and composition of the aggregate for each mesh and allowed to estimate the ratio of calcium carbonate / insoluble aggregate (mass). A mineralogical analysis was carried out by X-ray diffraction to CICRP (XRD Bruker D8 Focus, Co, 35kV-40mA, equipped with a linear detector Lynxeye) on one hundred samples. It was performed on the matrix and aggregate powder. It was supplemented by observations at optical polarizing microscope and by elemental analysis with the Environmental Scanning Electron Microscope (ESEM) on thin sections and rough fragments (SEM: LV, FEI Philips XL30, Mode Low vacuum, equipped with an EAS detector EDAX to make semi-quantification and a BSE detector to see the chemical contrasts) to Pratim pole to Aix-Marseille University.

2 The lime kiln

2.1 Lime kiln building

The archaeological operation consisted of surveys, excavations, study of buildings and archival research. The study of the building is based on detailed observation of construction, materials and their implementation.

In the 18th C. and during the first half of the 19th C., many periodic circular lime kilns installed in the hills of Saint-Cyr Carpiagne were well suited to the production of pure lime. The mid 19th C. is the transition of periodic to perpetual lime kilns and to hydraulic lime. In 1847, a Marseille architect V. Leroy wrote: "In our country, hydraulic lime was manufactured in the only establishment of Mr.

Tocchi and Mr. Villeneuve (at Roquefort-la-Bédoule)” [1]. A decade later many perpetual furnaces are built in the valleys of Vaufrèges and La Panouse. The 25th of July 1857, Antoine Homsy and Felix Gravel got an official authorization for the establishment of "two hydraulic lime kilns in their property located in the valley of the Panouse". The calcareous marl gray Bajocian - Bathonian formations of these hills allows this kind of production.

The industrial lime kiln appears as a solid cube of 14 m in front and 8.70 m high against the hill. This is a short flame and continuous calcination double furnace. In this draw kiln, the limestone was layered with coke and lit. As it burnt through, lime was extracted from the bottom of the kiln and further layers of stone and coke added to the top.

The excavation was conducted in the eastern furnace. It is built like an ellipsoid of revolution elongated, 4 m diameter, 7.5 m high, with a volume of about 64 m³. It is truncated at the top by the exhaust opening with a diameter of 2.20 m for loading the charge and at the base by the draw hole, a rectangular box 1.20 m x 1 m (Fig. 2). It is built in large local limestone rubbles lined with firebricks sealed with clay. The space between the façade and the furnace is filled with small flat stones and earth. The draw arches are gunboat vaults of 7.6 m² and 2.30 to 2.80 m high built in plain bricks (Fig. 3). Special care has been taken for the façade especially the piers and voussoirs of the harp paired arches.

Activities related to the extraction of lime were protected by an adjacent hangar of 170 m². Indications of the presence of the roof were retained as the beam embedding holes in front and tiles broken ground. Moreover, inside the hangar, the excavation allowed the discovery of lime extinction basin of 7.24 m².

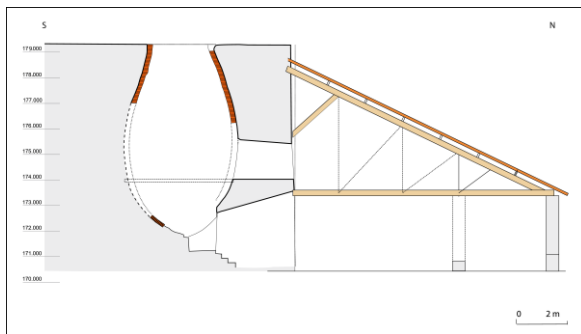


Fig. 2 Drawing of the lime kiln and restitution of the roof of the hangar (Sillano B, Inrap, Delpalillo D)



Fig. 3 Draw arch with layer of coal (cl. D'Ovidio A-M)

2.2 *Lime kiln transformations*

From 1870 to 1881 the property of the lime kiln knew several mutations. It was first sold to an architect and building contractor Hilaire Curtil and a banker Jacques Maroni and then to two other owners in 1877 and 1881. These mutations

can be the cause of changes observed in the hangar. It was divided into three areas by hollow brick walls and received a finishing plaster. The eastern brick wall destroyed the lime extinction bassin. Overmore, a small technical room was built (Fig. 4). Upper windows were pierced in the front of the kiln.

Three hypotheses are proposed to explain these transformations. The first suggested by the introduction of chimney flues within two furnaces, would be the use of the building as a habitat. The second would be the transformation of the kiln in "artificial hatchery" whose archives mention the presence in the valley in 1881. Finally, another hypothesis corresponds to a change in the functioning of the kiln. Originally built as a short flame running kiln, it would have been transformed by the new owner in long flame kiln with a fireplace supplied with coal by an upper opening.



Fig. 4 Hangar excavation : technical room, lime extinction basin and brick wall
(cl. D'Ovidio A-M)

3 Contribution of physicochemical analyzes to better knowledge of the building and to the construction phasing

The study of the construction was completed by the sampling of mortar masonry, coatings and sealing mortar. Their analysis provides information on building materials and their implementation. It allows to connect the structures and helps to establish a relative chronology.

3.1 The mortars of the original masonry

Two types of mortars are linked to the original masonry: earth mortar and clay sealing of the bricks of the furnace.

3.1.1 Use of earth mortar for masonry

The limestone rubble of the kiln and hangar masonry are built with a earth mortar containing a little part of lime and gray limestone gravels. These gravels, related to the geological formation of the relief, could come from the quarry stone mining residue sieving. The mortar is crumbly and the use of lime parsimoniously spotted by the presence of lime amalgam. The slightly dolomitic mortar contains gypsum in small proportions and clays at $d = 10$ and 14 \AA . The ratio of calcium carbonate / aggregate is about 1:1. However this type of analysis does not determine the amount of lime as the local earth included in the percentage of calcium carbonate is calcareous. It is assumed that this lime was produced by temporary circular ovens present in the valley and recorded in the Napoleonic cadastre of 1827. Lime residues used for the building of the kiln were sampled on the substrate at the foot of the eastern wall of the draw arch and the staircase. The binder is based on pure calcite corresponding to the use of a pure lime in the first phase of construction of the kiln.

3.1.2 Brick sealing clay of the furnace

The original bricks of the furnace were sealed with an earth rich in quartz, clayey and also containing dolomite, calcite, hematite and feldspars traces.

3.2 Mortars corresponding to building changes

Several types of mortars are identified as belonging to the transformation phases of the building: the gypsum plaster and the finishing coatings based on dolomitic sand.

3.2.1 The use of white gypsum plaster for the brick walls and the ceilings

In the late 19th C., the white gypsum plaster is used for making the hollow brick walls and the canisse ceilings. The gypsum plaster of these walls and of the walls of the small technical room was identified by XRD. Moreover, a mound of fragments of grooved plaster collapsed at the foot of the north wall of the technical room evokes a canisse ceiling coated with gypsum plaster in the Marseilles tradition. Moreover, Victor Leroy mentioned: the "white gypsum plaster of Roquevaire of good quality is used for sealing that require high toughness and for whitewashing" [1].

3.2.2 The finishing render of lime and dolomitic sand

Another aspect illustrating the evolution of the building is the use of a lime-based finishing plaster with dolomitic sand as aggregate. It coated the stonework of the hangar. It also coated the façade of the kiln and the brick piers and arches. Samples from several spots of the building show a similarity in composition allowing to put them in relation. Moreover, observation of the implementation of

these coatings shows a coincidence with the installation of hollow brick walls. Indeed, this dolomitic plaster is spread making a small thickening on the partitions. This is a slightly greyish coating, containing angular dolomitic sand and some coarser rolled silica sand grains. The sand particle size distribution shows two peaks, one at > 0.09 mm and another at 0.425 to 0.25 mm. Calcium carbonate ratio (lime) / aggregate (siliceous and dolomitic) is about one part of lime for one part of sand to two parts of lime for three parts of sand. Dolomitic sand is identified by microscopy in polarized light (and analyzed) on the thin section (Fig. 5). Analysis of the matrix by EDS shows that the binder was a slightly dolomitic calcium lime. The analysis on the angular grains confirmed the dolomitic nature of the sand. Two sources of dolomitic sand are known near the site: the first one is natural covering the slopes of Col de Sabatier and the second one is artificial (crushed stone) extracted from a quarry called Perasso settled in the Toulouse valley since 1840.

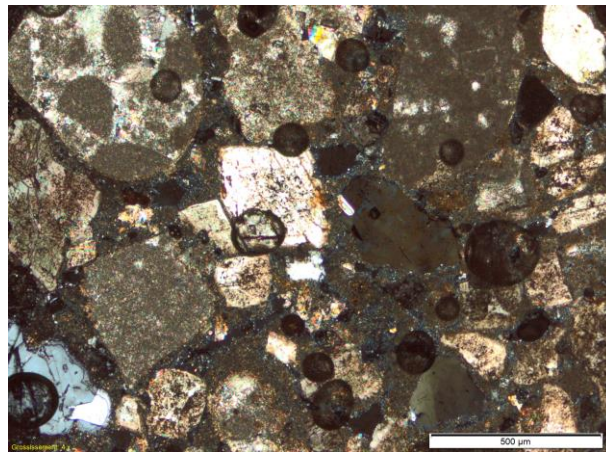


Fig. 5 Sample n° 80, hangar east wall: dolomitic sand, in polarized analyzed light x40 (cl. Bromblet Ph)

3.2.3 The use of hydraulic lime and gray and red gypsum plaster for sealing

The sealing of metal brackets and beams were made with hydraulic lime or gray or red gypsum plaster. From the observation of the stratigraphy of dolomitic coating it can be deduced that the gypsum sealing are latter than the dolomitic coating since the gypsum plaster covers the coating. On the contrary, the seals made with hydraulic lime predate since they are covered by the dolomitic coating. Thus, the western and central metal facade brackets are sealed into the wall with a hydraulic lime. They may have been made at the time of the kiln working. Otherwise, the eastern beams and brackets are sealed with red and gray gypsum plaster. Red gypsum plaster contains clay minerals at $d=10$ to 14\AA . V. Leroy mentioned the red gypsum plaster of Allauch, "most wanted than the grey one, because it is easier to apply under the trowel" and "colored by metal oxides" [1].

3.3 The old restorations: use of hydraulic lime and white gypsum plaster

The mortar study also reveals old restorations, especially at the furnace whose high temperature fluctuations require ongoing maintenance. Two materials are identified for restorations: hydraulic lime and white gypsum plaster. At the top of the furnace, the original bricks of the mark Carbonel lining the wall were replaced by brick Uzes David. They are not sealed to the clay but with hydraulic lime. Louis David factory did not exist at the time of construction of the kiln. It was built shortly after in 1859 and was closed in 1913. The restoration of the furnace is to be considered in this period of time 1859 - 1913. However, in the bottom of the furnace, the bricks were restored with gypsum plaster (Fig 6). The gypsum plaster can withstand high temperatures. The draw arch of the furnace is backfilled from the 1920s, so these restorations are previous.



Fig. 6 Gypsum plaster restorations in the bottom of the furnace (cl. D'Ovidio A-M)

4 Contribution of physicochemical analyzes to better knowledge of the kiln production

Other samples were done on quarry, residues of production: limestone, slag and coal to try to better understand the production of industrial kiln.

4.1 Production: hydraulic lime or pure lime?

Research on the nature of the production of the kiln is made from samples in the nearby quarry of formations Bajocian – Bathonian. It shows the presence of clay at $d = 10$ and 14 \AA associated with dolomite, gypsum and quartz at traces. Other analyzes performed on the residue adhering to the wall of the basin show that the composition of this lime almost exclusively based on calcite and a bit of

dolomite is that of a slightly dolomitic lime (Ca 19.50% At, Si 1.11%, At, Mg 0.75% At). However, the limed soil was made with a hydraulic lime: the SEM analysis gave in the matrix: calcium (11.54% At) and silicium (10.48% At) in equivalent amount (Fig. 7). XRD analysis performed on residues inside the kiln did not show the presence of gehlenite but further SEM analysis would be required. Thus, these analyzes allow us to observe that two types of lime were produced in the industrial furnace: a pure lime and a hydraulic lime.

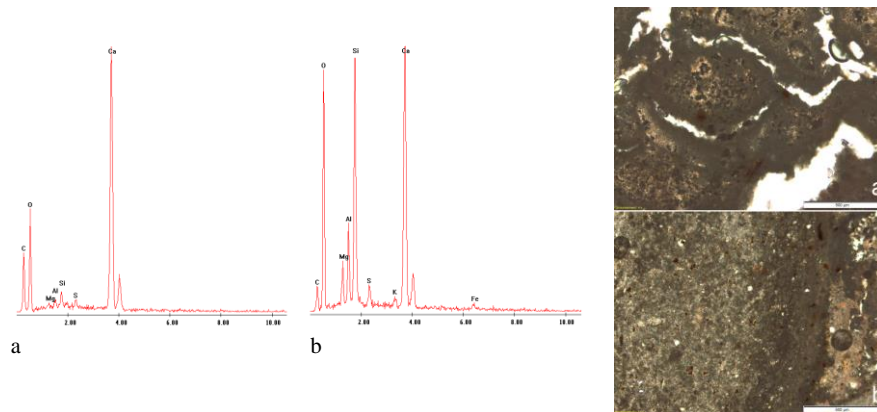


Fig. 7 EDS analysis and microscopy pl x40 of pure lime (lime in the basin) and of hydraulic lime (limed soil)

A second source of rock had to be used for the production of lime, maybe the white limestone of the Upper Oxfordian and Kimmeridgian formations on the northern slope of the valley of La Panouse.

4.2 The combustible

A thick layer of angular blocks of coal was spread in the draw arche of the kiln. It might have been pulled out the furnace before calcination and is a testimony of the end of the kiln use. The volatile matter content of 41% and the amount of carbon (68-72% At) (59 to 61% Wt) evoke the use of lignite. The transition from coal to wood is a key driver of the move towards industrialization of lime manufacture. That corresponds to the period where “new mining companies engage in industrialization methods of extraction and transport of local lignite until Marseille industries”[2].

4.3 The cooking residue: grappier and slag

Two kinds of industrial pollution mark the environment of the kiln: the grappier and slag. A whitish embankment of 72 m long and 12 m wide, 6 m high, with a volume of around 2000 m³ is the grappier. It was formed by the accumulation of cooking residue, of unburnt and overcooked materials. It contains

small grayish limestone not fully transformed into lime, brick fragments collapsed from walls of the furnace and slag. The analysis of samples taken in the grappier gives interesting results. For example, the presence of cristobalite reveals a high cooking temperature. The presence of the gehlenite evokes the production of a hydraulic lime. More systematic sampling from each stratum deposit should be performed to specify the production phasing. Moreover, traces of picket with a pick or crowbar were noticed in the grappier (Fig.8). They could evoke its reuse as cement grappier. This is a technique invented by the french ciment producer Lafarge that appears to have been developed in the second half of the 19th century until the World War I.

Two mounds of black dust including a quantity of slag buttressed either side of the kiln. XRD analysis shows that these slags come from different sources. Some come from the high temperature firing of the lime kiln. This is the case of slag with lime gangue or limestone, or those whose presence of mullite evokes cooking firebricks detached from the wall. However, others are slag glass or metal. These contain hematite, magnetite and olivine (fayalite).

Their presence on the site may be linked to a collect of a second hand combustible on the metalworking industry sites. That had been already noticed by Raoul Blanchard to other nearby kilns (ovens Fangas of Redon in Marseilles) [3]. The lime burners provide lime to these industries and come back with this combustible including slag. The lime burning does not require a high calorific value combustible. The operating period of the kiln of La Panouse corresponds to the Marseilles metallurgy growth period for irons and fonts. The blast furnaces of St. Louis and the Capelette forges are in their growing phase [4]. Lime is used in steel industry. At high temperatures, it reacts with the impurities of metals and contributes to their refining.



Fig. 8 The grappier (cl. D'Ovidio A-M)

In conclusion, this study has provided important elements to the knowledge of the building as the identification of materials as their implementation. Moreover, the study of materials helps the archaeologist of the building in the phasing of the transformations of the building. Finally, the identification of production and its residues is an essential tool for industrial archeology.

Thus, this site presents all of the operating chain of production of lime essential to large Marseilles urban projects and to the development of metallurgy irons and fonts in the second half of the 19th century.

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