

Study of Architectural Ceramics of the Qal'a of Bani Hammad

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Abstract. In order to define with precision, the physicochemical characteristics of the architectural ceramic exhumed from the Qal'a of Bani Hammad's site (inscribed on the UNESCO's World Heritage List in 1980), our study was conducted on a set of ceramics. The first analyses were focused on the color of the glazes, by optical absorption spectrophotometry (also called optical absorbance spectroscopy), on the texture by X-Ray diffraction, and on the elemental composition of the various constituent stages of these glazes and their terra cotta, by scanning electron microscopy and X-Ray fluorescence. Indeed, the study combining optical absorbance spectroscopy and X-Ray fluorescence enabled us to determine the origin of the monochromic green coloring of the glazes in some samples. This coloring can be attributed to the presence of the Cu^{2+} chemical element; but these glazes darkened due to the high iron content they contain. This could be explained by an intentional addition of iron, in tin-opacified leaded glazes, baked in an oxidizing atmosphere and put on a limestone-rich marlacious paste.

The chemical and mineralogical analyses of all shards show that their paste is calcareous. This dataset will be exploitable for re-creation experiments. It also represents a first exploitable referential system, for potential comparisons with other samples belonging to other Algerian sites and different periods of time.

Key words: The Qal'a of Bani Hammad; physicochemical characterization of ceramics; optical absorption spectrometry; X-ray fluorescence; X-ray diffraction.

1. Introduction

An Ifriqiyian architecture took shape in the 9th century and the first elements of a ceramic art appeared in Kairouan under the Aghlabids dynasty. Moreover, important urban centers such as Mahdia, the Qal'a of Bani Hammad, Bougie, Tunis, appeared and adopted this new industry.

As for the Qal'a of Bani Hammad, numerous records confirm that it was one of the most dynamic cultural and scientific centers of central Maghreb in the 11th and 12th century. It played an important role of "relay place" after the decline of Kairouan and before the founding of Bougie (Aissani and Amara, 2014); and there is no doubt that local workshops definitely existed. Golvin (1978) even assumed their exact location at the Qal'a of Bani Hammad. The ceramic that we found in this site has an oriental style and continues the Egyptian Aghlabid and Fatimid tradition (Daoulatli, 1978). More specifically, the architectural ceramic is abundantly represented in the Qal'a; the remarkable variety of its production techniques can only stir up regrets, regarding the fact that no systematic study using modern analysis processes has been undertaken, despite promising typological rating tests carried out a long time ago by George Marçais (Golvin, 1978) and refined by other archaeologists.

Although the site perfectly meets the criterion of authenticity defined by the world heritage chart, we defined specific objectives for this study by not selecting "all" the architectural ceramic varieties existing in the Qal'a. Indeed, the purpose of this work is to expose some varieties of the ceramic exhibited in our different museums, to provide a counterpoint regarding the analyzed

ceramics and to contribute, on the first part, to the collection of analytical data supposed to promote historical, archaeological and architectural research, with a focus on the production techniques of the ceramics in question, as well as their diffusion and movement.

2. Sampling and Analytical Methods

A first study of the ceramic (collected from the Qal'a of Bani Hammad's site) took place in 1903 in works dedicated to the old city. It was led by A. Roberto, who provided the reproduction of nine shards, among which eight were in color and mentioned them in the "*Collection of the Archaeological Society of Constantine*". In 1906, Georges Marçais emphasized on the importance of this ceramic, in a comprehensive study on Art in Algeria. In 1908, M. Saladin published and commented on Paul Blanchet's notes, evoking architectural coatings and ceramic partitions found in the ruins. In 1909, General De Beylié put forward in his book on the Qal'a, pictures and engraved drawings, and summarily analyzed this industry.

In 1965, Lucien Golvin provided in his book « Archaeological Research at the Qal'a of the Bani Hammad », pictures and drawings of an important amount of ceramic, exhumed from the site in question. He also gave a descriptive approach on the production techniques used in the past. This same year 1965, Rachid Bourouiba published in a preliminary report about the excavations campaign led by himself, a number of plates about architectural ceramic and ordinary potteries exhumed from these excavations, that took place in September 1964.

Besides, seven samples of the architectural ceramic, used for the construction and the interior and exterior decoration of the Qal'a's palaces, were selected for this study. A quick examination of the physical characteristics of these samples enabled us to choose the appropriate investigation methods to solve the problem and, to define the analytical modes. These samples were submitted to the following tests:

First, the surface of each shard was photographed and observed thanks to a binocular magnifier. Then, millimetric chips removed from each shard were examined by using a scanning electron microscopy (PHILIPS.SEM 505 model).

In order to identify the chromogen agents responsible for the coloring of the glazes, at the level of the enameled samples, a double-ray UV-visible optical absorption spectrometer (CARY 500 de Varian model) was used with the spread-reflection mode, directly on the sample for the glaze and in section for the terra cotta; the scanned spectral range vary between 350 nm and 800 nm.

The study of the glaze's elementary composition was performed by using the backscattering electron mode, thanks to the X-Ray fluorescence paired up with the scattering energy scanning electron microscopy (JEOL JSM6360LV model). Results are in oxides mass percentage and the measures were carried out on cross-sections of glazes, previously polished and metallized with a thin carbon layer, on selected areas.

The study of the terra cotta's elementary composition was performed with an X-fluorescence spectrometer (SRS 3400 – BRUKER – AXS model). Results are in oxides mass percentage and the measures were carried out on pearls, with a small amount of terra cotta.

The study of the terra cotta's mineralogical composition was performed with an X-ray diffractometer (PANalytical: XPERT-PRO model).

During the surface collection, the KBHEC01 sample was found at the level of the Lake palace, and KBHEC06 was also collected from a ground paving made of terra cotta bricks; the whole of the bricks was put in opus picatum at the level of this same palace. Nevertheless, the KBHEC05, KBHEC08, KBHEC09, KBHEC10 and KBHEC12 samples were collected at the level of the Manar palace.

2.1. Presentation of the studied material

The KBHEC01 sample: it represents a stalactite debris molded and enameled in opaque white on the four faces; the terra cotta is beige orange, compact and contains some thin and abundant inclusions. The piece has a parallelepiped shape with four levels; on each side, we notice ribs.

The KBHEC05 sample: this one is a fragment from a rounded tile, produced by the modelling technique; the terra cotta is red-rosy, compact and contains a thick and abundant grog grease remover.

The KBHEC06 sample: It is a brick, produced by using the modelling technique, with a red color, compact and it contains a thick and abundant grog grease remover.

The KBHEC08 sample: it is a merlon debris molded in a leaf shape, which low surface is enameled in a dull green that has almost all disappeared. The paste of this shard is beige yellow, compact and contains some thick and abundant inclusions. This merlon represented a frieze element that decorated the Hammadid palaces' frontage.

The KBHEC09 sample: It presents a six-point star-shaped ceramic marquetry, molded and enameled in a bottle-green color on its superior surface. The paste of this marquetry is red-rosy.

The KBHEC10 sample: this one is a fragment from a cross-shaped marquetry, used as ground paving, molded and enameled in white on its superior surface. The paste of this marquetry is beige yellow, compact and it contains some very thin and abundant inclusions.

The KBHEC12 sample: it represents a triangle-shaped ceramic marquetry debris, molded and enameled in green on its superior surface. The paste of this marquetry is beige yellow, compact and contains some very thin and abundant inclusions.

All the samples indicated above are dated given the current state of knowledge, between the 5th and 6th Hijri century (11th – 12th century after J.C).

3. Experimental results and Discussion

3.1. Study of the surface's states

To the naked eye and by using the binocular magnifier, we notice that the surface state of these samples is characterized by a more or less significant loss of color. All these surfaces have some scratches caused by a mechanical degradation. One of the samples, KBHEC10, also exhibited a "crazing". These cracks emerge in the glaze during the cooling, coming from traction pressures due to differences in terms of thermal distention coefficient between the ceramic paste and the glaze. It is not uncommon to observe them on the surface of a glaze (Munier, 1957). All samples appear to be in good condition. They all have relatively homogeneous surfaces. With the exception of the KBHEC08 sample, which almost lost all of its green glaze; the remaining part appears in a more advanced state of degradation.

Macroscopically, KBHEC01 presents on its glazed part a defect of withdrawal. In the current state of knowledge, the glaze withdraws from the shard during the fusion and reveals spots where the clay is bared (Fig. 1). This phenomenon can affect a glaze put on a poorly dusted biscuit. Moreover, a frequent cause of this phenomenon is the crazing and cracking of the raw glaze. This case is more frequent in very matt glazes and viscous glazes during the fusion (Rhodes, 1976).

On a fresh break at the level of the terra cotta contained in the KBHEC09 sample, we observe the presence of undecomposed limestone or gypsum in the shard's pores (Fig. 2). The observation under the binocular magnifier of a polished section at the level of the interface zone, between the glaze and the terra cotta support of the same sample, reveals the presence of a yellow border

(strip) (Fig. 3), particularly visible at the level of the green glaze; which could correspond to devitrification crystals. This leads us to assume that the glaze was put on earth.

At the level of the KBHEC10's terra cotta and on a polished section (fig. 4), the paste presents a beige color; it contains mauve luminescent grains, surrounded by a large amount of orange grains. This color could correspond to the respective presence of quartz crystals and undissolved calcite. The observation under the magnifier, at the level of the terra cotta contained in the KBHEC12 sample reveals characteristics similar to those contained in the previous sample: the beige terra cotta contains mauve luminescent grains, surrounded by a large amount of orange grains. At this scale, no other characteristic is discernible.

The observation using the backscattering electron mode shows the heterogeneity of the glaze contained in the KBHEC08 sample, its thickness of about $350\mu\text{m}$ (fig. 5) as well as the presence of numerous variable-size bubbles. The bubbles were probably formed during the degassing phase of the terra cotta, or during the decomposition of the glazed mix during the baking. Besides, KBHEC12 (fig 6) reveals on its glazed surface tiny needles, which could represent residues coming from a vegetal ash or from a contamination of the environment, such as biological microorganisms.

3.2. Characteristics of the glazes

3.2.1. Measure of the glazes color

The physicochemical origin of the coloring of the glaze is accessible analytically, by trying to identify metallic cations playing the role of chromogen agents. The registered optical absorption spectrum can thus be compared with reference absorption spectra.



Fig. 1. Defect of withdrawal of the stalactite enamel KBHEC01



Fig. 2. Observation on a fresh break at the level of the terra cotta, KBHEC09

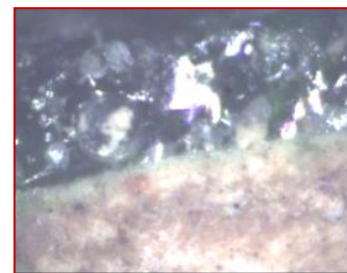


Fig. 3. The glaze/terra cotta interface zone, KBHEC09



Fig. 4. Polished section at the level of the paste of KBHEC10



Fig. 5. Micrography using the MEB of the glaze/terra cotta interface zone of KBHEC08

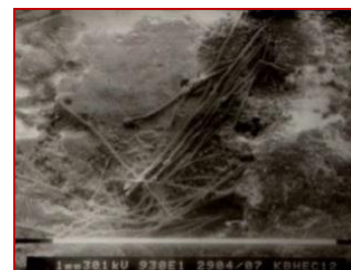


Fig. 6. Micrography using the MEB of the glazed surface of KBHEC12

The spectra obtained on the white glaze contained in KBHEC01 and KBHEC10 respectively present a low absorption between 350 and 410 nm and between 350 and 450 nm, which corresponds to the violet area. Afterwards, the absorption is very low and constant between 450

and 800 nm. The spectra show that the obtained color is not a perfect white but an off-white; and this, due to the absorption in the violets, as well as the beginning of the blues present in the glazes of the two samples. These spectra do not allow us to reveal the chromogen elements responsible for the coloring (Richard, 2000).

The optical absorption spectra of the KBHEC08, 09 and 12 show a first absorption strip between 350 and 500 to 520 nm (corresponding to the visible and blues area), and a second starting from 550 nm. A large strip varying from 550 and 800 nm is normally attributed to the Cu^{2+} , which gives the glaze a green color when it gets leaded (Rhodes, 1976).

3.2.2. Elementary composition of the glazes

For the white glazes contained in the KBHEC01 and KBHEC10 samples, they represent potassic alkaline-leaded glazes (Table 1), which white opaque aspect is essentially due to the presence of tin ($\text{SnO}_2 = 10.95 \pm 1.58$ in *KBHEC01* and 17.13 ± 1.58 in *KBHEC10*), in the form of cassiterite crystals (SnO_2), highlighted by X-Ray diffraction and performed on a sample of white glaze from the above samples.

It is also possible that the unmelted quartz crystals in the white glaze reinforce the effect of the tin oxide in the opacification phenomenon. Indeed, we know that the opacity of the pre-Islamic glazes is sometimes related to the only presence of quartz crystals and feldspar (Mason, 1997).

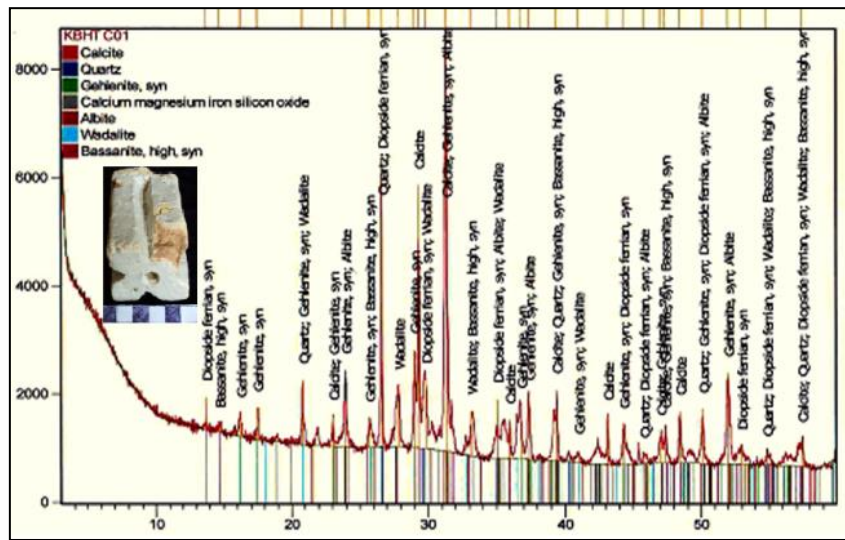
We also observe the presence of iron in non-negligible content ($\text{Fe}_2\text{O}_3 = 2.55\%$ for *KBHEC01* and 4.49% for *KBHEC10*), which may explain the white color with a yellow tendency of glazes contained in the two samples, because 1% of iron oxide is enough to dimly dye a glaze. Besides, this high content in iron could also come from an intentional addition of it, in order to change few properties of the glaze. Sometime a matt glaze will get bright and shiny by adding 2 to 3% of iron (Picon, 1973). But the presence of lime in high content (05.56% in CaO) minimized the effect of the iron oxide on the glaze's color.

On the other hand, the non-negligible content in aluminum (4.40% of Al_2O_3 for *KBHEC10*) could explain that there was an intentional addition of aluminum as well, as alumina increases the viscosity of the glaze and the adhesion to the ceramic substrate.

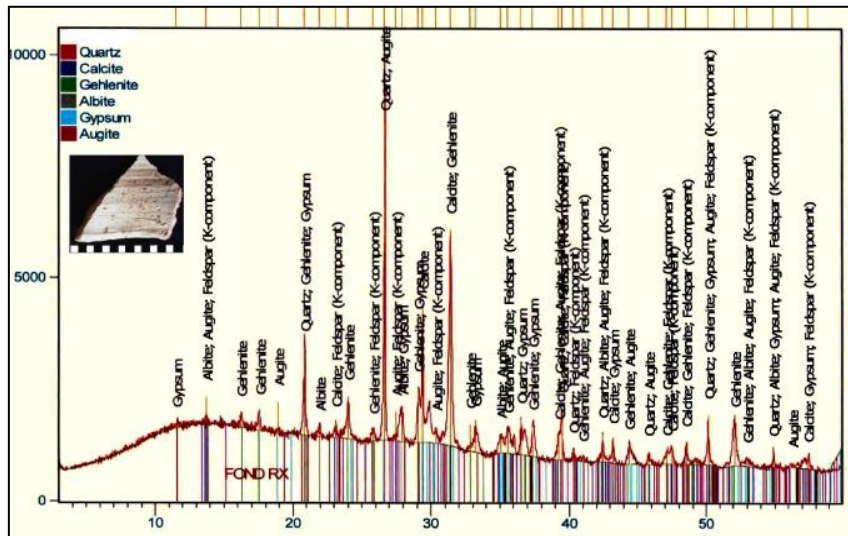
The elementary analysis of the green glazes from the KBHEC08, KBHEC09 and KBHEC12 samples (Table 1) show that they are leaded and that the tin (with the respective content of $\text{SnO}_2 = 11.63\%$, 10.05% et 10.86%) explains the opacification of the glazes.

Table 1. Qal'a of Bani Hammad. Elementary analysis by scattering energy X-Rays fluorescence, of the white and green glaze, converted in oxides mass percentage of studied samples. Nd : not determined.

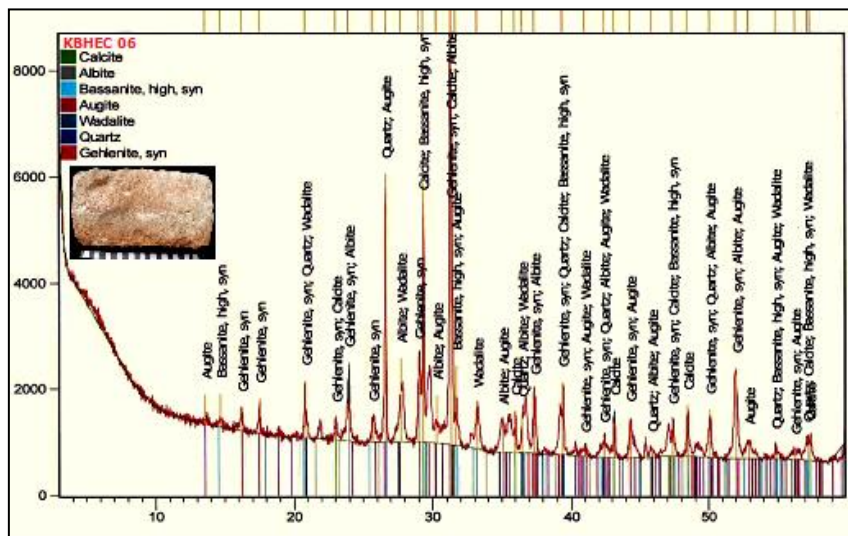
Oxides	KBHEC01	KBHEC10	KBHEC 08	KBHEC09	KBHEC12
	White glaze	White glaze	Green glaze	Green glaze	Green glaze
SiO_2	51.32	32.77	36.44	21.57	23.93
Al_2O_3	01.72	04.40	04.43	01.26	03.15
CaO	02.59	05.56	05.85	02.03	03.44
MgO	nd	03.75	01.00	00.23	03.08
Na_2O	00.84	00.59	00.73	nd	00.85
K_2O	02.79	03.60	00.60	00.58	00.58
PbO	25.49	23.46	26.91	27.79	22.77
SnO_2	10.95	17.13	11.63	10.05	10.86
Fe_2O_3	02.55	04.49	01.74	04.23	01.46
CuO	nd	nd	08.93	28.85	25.83
ZnO	nd	00.90	nd	nd	nd
P_2O_5	01.74	00.34	00.45	nd	nd
C	nd	03.02	01.29	03.11	04.05
Cl	nd	nd	nd	00.30	nd



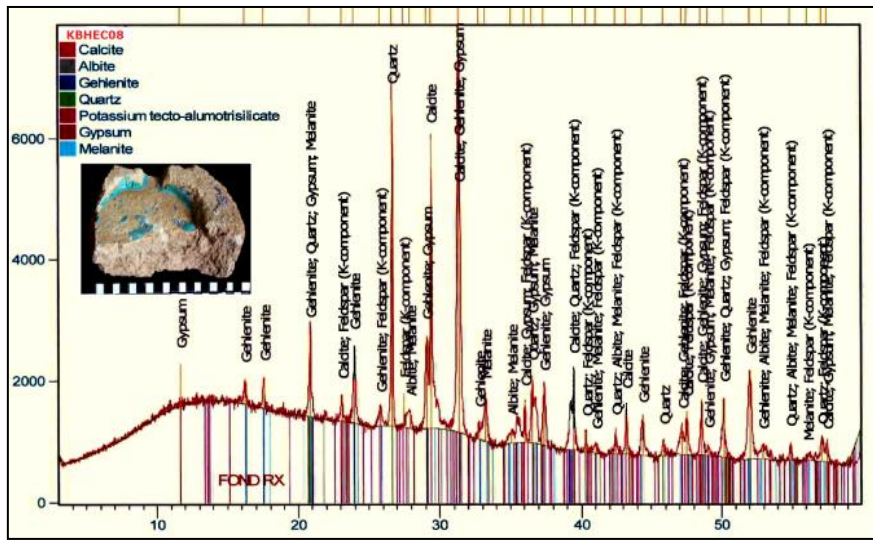
(a)



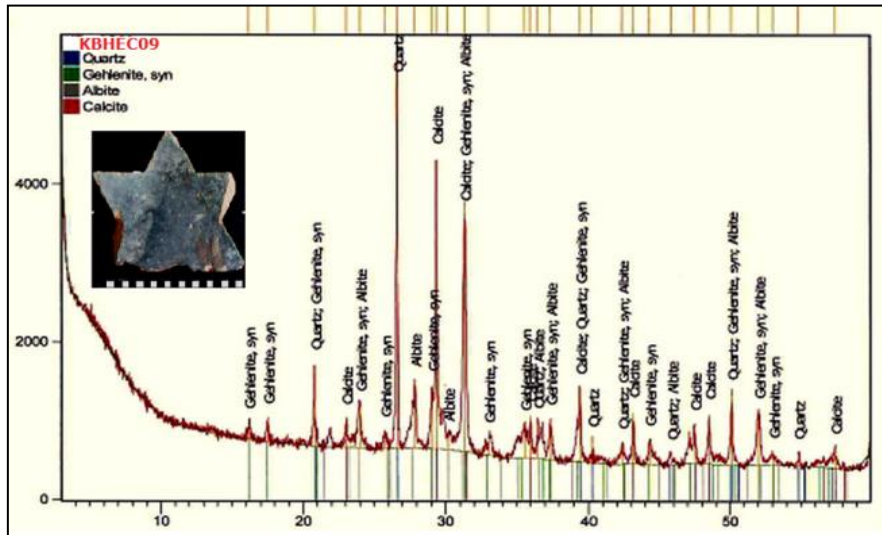
(b)



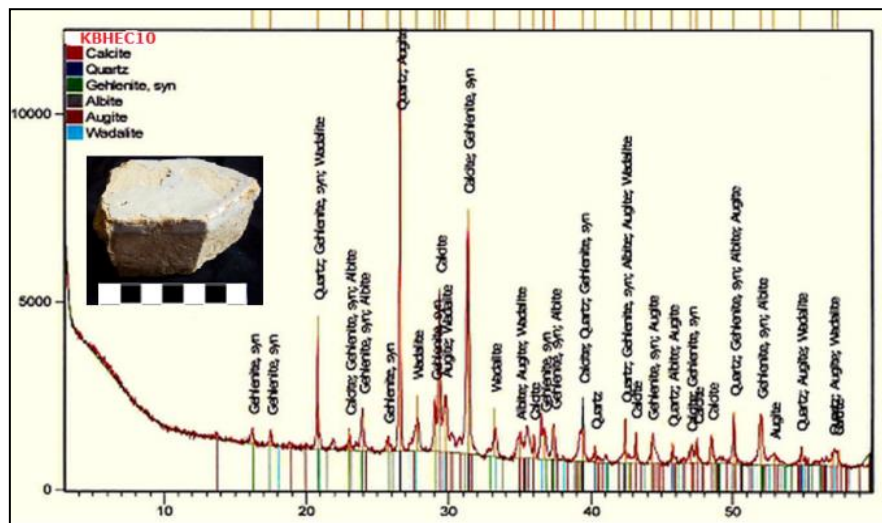
(c)



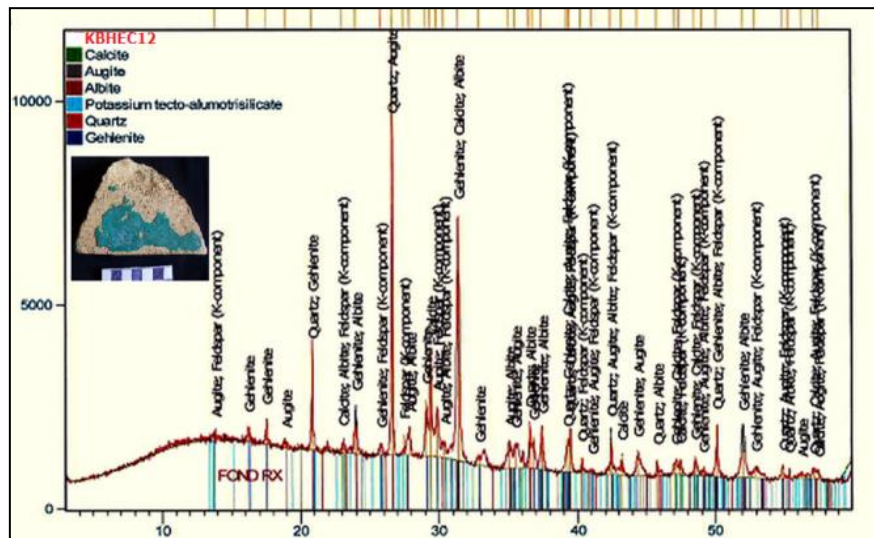
(d)



(e)



(f)



(g)

Fig. 7. Qal'a des Bani Hammad, X-Ray diffraction spectrum of terra cotta from the KBHEC01(a), KBHEC05 (b), KBHEC06 (c), KBHEC08 (d), KBHEC09 (e), KBHEC10 (f), KBHEC12 (g) samples.

The X-Ray diffraction enables us to highlight the mineralogical composition and thus, it is possible to define temperature zones. The Gehlenite (silico-aluminatocalcium) appears at about 800°C and reaches its maximum at around 900°C. The cohesion of the limestone pastes results from the formation of the Gehlenite, when the baking does not exceed 900°C. However, at the highest temperature the cohesion role is mainly played by the Anorthite. The Diopside is formed after the Gehlenite. But the fact that we discovered an important phase of unseparated calcite allows us to venture the hypothesis that the substrate would have been baked at around 850 to 900°C with a short length of baking time, which produced an incomplete separation of the calcite. It is important to mention as well that once baked, the shard shouldn't contain calcite. The separation tension of the calcite is about 1 atmosphere at 900°C, but the separation becomes appreciable from 700°C in limestone clays (Picon, 1973).

Nevertheless, it is not rare to discover calcite in archaeological ceramics. There could be three reasons:

- a- The baking temperature remained at a lower level to bring the complete separation of the calcite, from which a part remained unchanged.
- b- The baking temperature, although it was high enough to lead to a complete separation of the calcite, was not maintained a sufficient time so the quicklime (CaO) resulting from the decomposition of the carbonate (CaCO_3) could get mixed with the other constituents of the paste. In these conditions there will remain a certain proportion of lime in the shard. But this lime, sooner or later, will come back to a carbonate by fixing the carbonic gas (CO_2) of the air (Picon, 1973).
- c- It results from a sediment (the action in a landfill place), which means that the calcite come from the water contained in landfill soils.

4. Conclusions

This study of characterization, that concerns few samples of architectural ceramic collected at the level of the Qal'a of Bani Hammad, enables us to know the nature of clays in this region better and to conclude that:

- The ceramic material updated at the Qal'a constitutes the major argument of a reasoning revealing the existence of an important handicraft of ceramic that developed in the area of this Hammadid site.

- The similarity regarding the composition, between the samples of the analyzed ceramics, stipulates that the production workshops used the same baking method, which temperature can vary from 850 to 900°C.

5. References

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