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AN INVESTIGATION OF THE CERAMIC TECHNOLOGY OF A LATE IZNIK CERAMIC PRODUCTION (XVIIITH CENTURY AD)

Ayed BEN AMARA, Max SCHVOERER, Maïa CUIN

Centre de Recherche en Physique Appliquée à l'Archéologie, Université de Bordeaux 3/
CNRS (IRAMAT, UMR5060), Maison de l'Archéologie, 33607 - Pessac Cedex, France

Mohamed Baji BEN MAMI

Institut National du Patrimoine, 4, Place du Château, Tunis, Tunisie.

Corresponding author: BEN AMARA A. (E-mail: ayed1@caramail.com)

1. AIMS OF THE RESEARCH

This paper describes the results of a physical study on a late Iznik ceramic production in order to investigate the ceramic technology of the 17th century Ottoman workshops. More particularly it will help to determine the specificity of a "red Iznik colour" dating from this time (Ben Amara, 2002). This colour made the originality of the Iznik ceramic workshops from the second half of the 16th century to the end of the 17th century. The first known examples of the "red Iznik colour" appeared around 1550 on the *mihrab* of Suleymanie mosque in Istanbul (Soustiel, 1985; Süslü, 1995; Altun, 1997).

Our main objective is the characterisation of the nature and composition of the different parts of the ceramic architectural ornaments. These quantitative data will help us to find technological processes used to produce these ceramics. Secondly, we will compare our data with bibliographical data relative to the first production (Tite, 1989).

2. MATERIALS

We have worked on four fragments of tiles presenting polychrome decoration (red, blue, green and black). These samples are of a late Iznik glazed production and decorate the walls of the Sidi Mehrez Mosque in Tunis, built by M'Hammed Bey in 1692. Bibliographical data (Daoulatli, 1979; Ben Mami, 2000) tell us these tiles were an ottoman production and were imported from Iznik workshops. Actually, Tunisia was an Ottoman province as early as 1574. Iznik (ancient Nicaea) is a town located on the main trade

routes linking Middle East to Balkans through Anatolia since the 4th century AD. Its name is synonymous with the brilliant period of ceramic production during the Ottoman era.

3. METHODOLOGIES

The description of the texture was carried out with scanning electron microscopy (SEM). Observations under scanning electron microscopy (JEOL JSM 820) were carried out on a thick blade detached by sawing, perpendicularly to the surface of the sample.

The elementary composition of the various layers was determined by energy-dispersive X-ray spectrometry with the system Link AN10000, coupled with the SEM. The analysis was made on five distinct zones of about $1.08 \mu\text{m} \times 0.88 \mu\text{m}$ for the ceramic support and of $108 \mu\text{m} \times 88 \mu\text{m}$ for the glazes and decoration. In accordance with the use, contents are expressed in percentage of weight of oxides.

Crystalline phases were identified by X-rays diffraction. We used a powder diffractometer (Siemens, Krystalloflex D500 with copper anticathode); the explored angular range was between 5° and 60° (for 2σ) and the lines of the diffractograms obtained were allotted by comparison with the A.S.T.M. (American Society for Testing and Materials) reference card.

Raman spectrometry analysis was used for a more precise characterisation of the "red Iznik colour", with the help of Dr. Pham V Huong (Laboratory of Molecular Physicochemistry, CNRS-University of Bordeaux 1). The spectra were determined by micro-Raman spectrometry (Dilor, model LABRAM) using 514.5 nm and 632.8 nm laser emission. The energy of the laser was always maintained very weak (lower than 10mW) to avoid any deterioration of the dyes. The spectra resolution was about 1nm.

4. EXPERIMENTAL DATA AND RESULTS

4.1. Texture and elementary composition

The ceramic material is made up of various layers: a pink-beige ceramic body on which is applied a white slip, used as a background for the decoration, and recovered by a transparent glaze. A SEM photomicrograph (fig. 1) of a section through a red-painted region shows (from top to bottom) the different parts of this complex material:

1. The transparent glaze, which thickness varies between 200 and 250 μm , covers the entire surface. It is a lead glaze (46.2 - 53.5% PbO). The sodium and potassium contents are relatively weak: 3.6 to 4.5 % Na₂O and lower than 0.6 % K₂O. This glaze is slightly coloured in green with copper Cu²⁺ (about 0.3 % CuO).

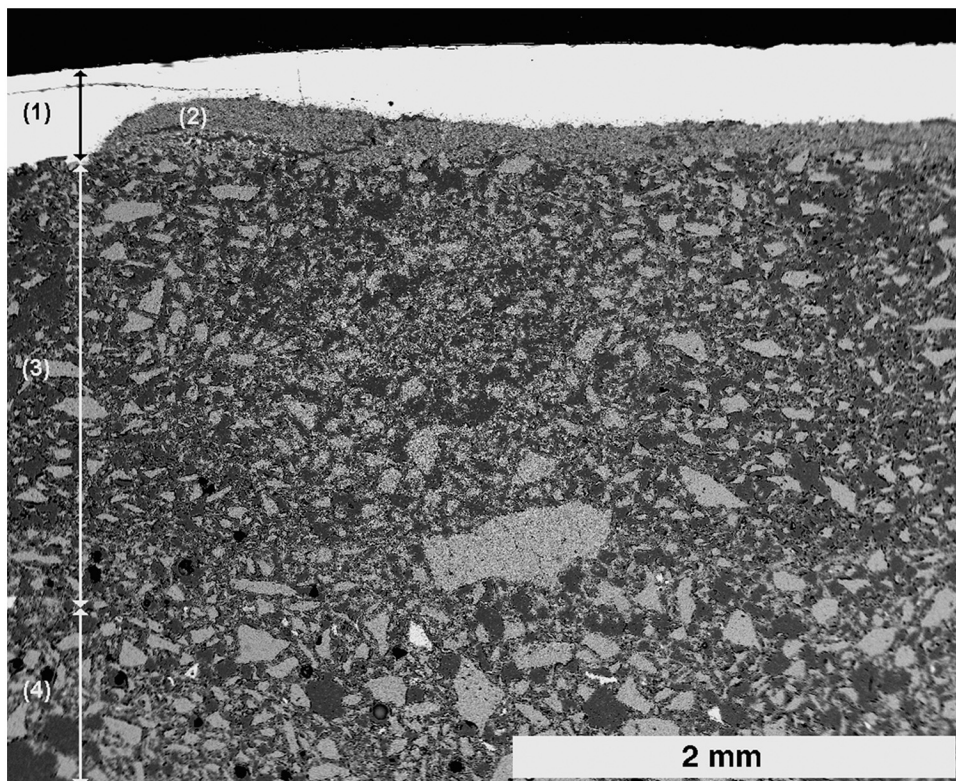


Figure 1. A SEM photomicrograph (BSE image) of a section through a red-painted region, showing (from top to bottom), the transparent glaze (1), the “red Iznik colour” (2), the white slip (3) and the ceramic body (4).

2. The “red Iznik colour” is very rich in silicon (71.3 – 81.4% SiO_2) and also contains iron (5.6 - 8.1% Fe_2O_3). We have detected also aluminium (3.9 - 5.3% Al_2O_3), lead (1.6 - 5.7% PbO), sodium (2.5 - 2.9% Na_2O) and calcium (1.8 - 2.9% in CaO). The presence of these elements could be due to the penetration of the glaze in “red Iznik colour”. Analysis by X-ray diffraction shows the presence of quartz (SiO_2) and hematite (Fe_2O_3).

3. The white slip is very rich in silicon (89.5 - 92.7% SiO_2) and the analysis by X-ray diffraction only reveals the presence of quartz. Other elements are present in small quantity, mainly calcium (1.9 - 2.8% CaO), aluminium (1.6 - 1.7% Al_2O_3), iron (0.7 - 1.4% Fe_2O_3), sodium (0.7 - 1.1% Na_2O) and magnesium (0.8 - 0.9 % MgO).

4. The elementary composition of the ceramic body is relatively close to that of the slip: 96.5 - 93.2 % SiO_2 , 1.7 - 1.3% Al_2O_3 , 0.6% Fe_2O_3 and 1.0 - 0.9 % MgO .

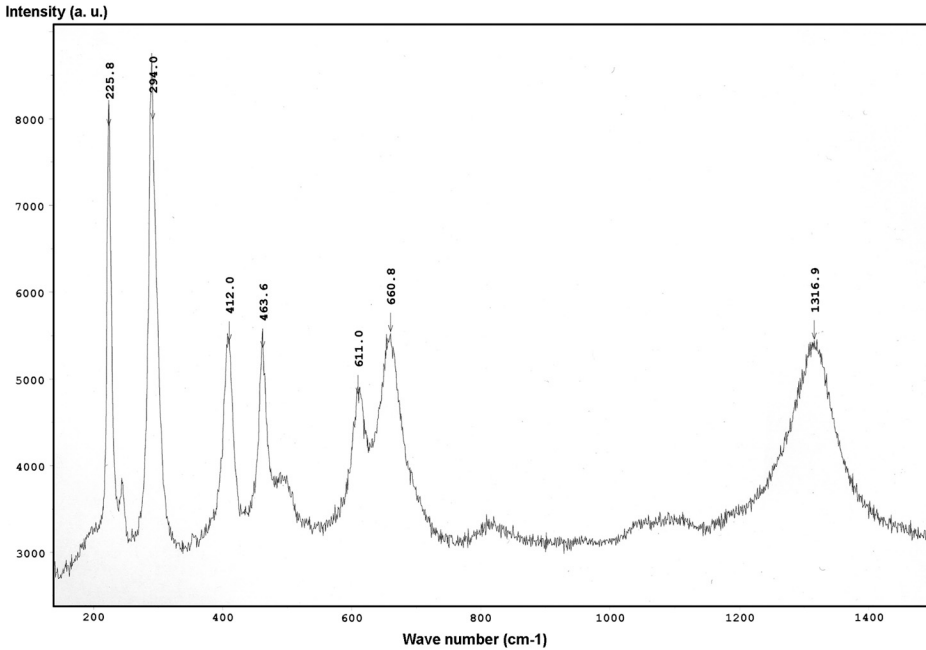


Figure 2. Raman spectrum of red grain present in the red decoration. The line at 463.6 cm^{-1} is specific of quartz. The others lines correspond to hematite (Fe_2O_3).

We note that the red decoration is different from the first Iznik production. Actually, it tends to be brown-red coloured. This colour is composed of differently coloured grains (red, black and orange). The analysis carried out on red grains gave a Raman spectrum (fig. 2) which one could assign to hematite (Edwards and Tait, 1998). Among the identified Raman lines, it is necessary to note the presence of another line at 463.6 cm^{-1} , independent of the hematite, which we identified as being quartz. The relative intensity of this line is variable and seems to be correlated to the colour of the analyzed grain. When the red grain is clearer, the line of quartz is more intense. This makes us consider the use of ferruginous sand. The presence of small black grains in the red colour, identified by Raman spectrometry as amorphous carbon (fig. 3), contributes to darken the colour of the red decoration.

The other colours of decoration are:

Blue: The elementary composition of the blue-painted region is close to that of the transparent glaze. The blue decoration is obtained with a cobalt dye.

Green: The elementary composition of the green-painted area is also close to that of the transparent glaze. One notices the presence of copper (Cu^{2+}) in greater proportion in this region (2.1 - 0.7% CuO) compared to the transparent glaze (about 0.3% CuO).

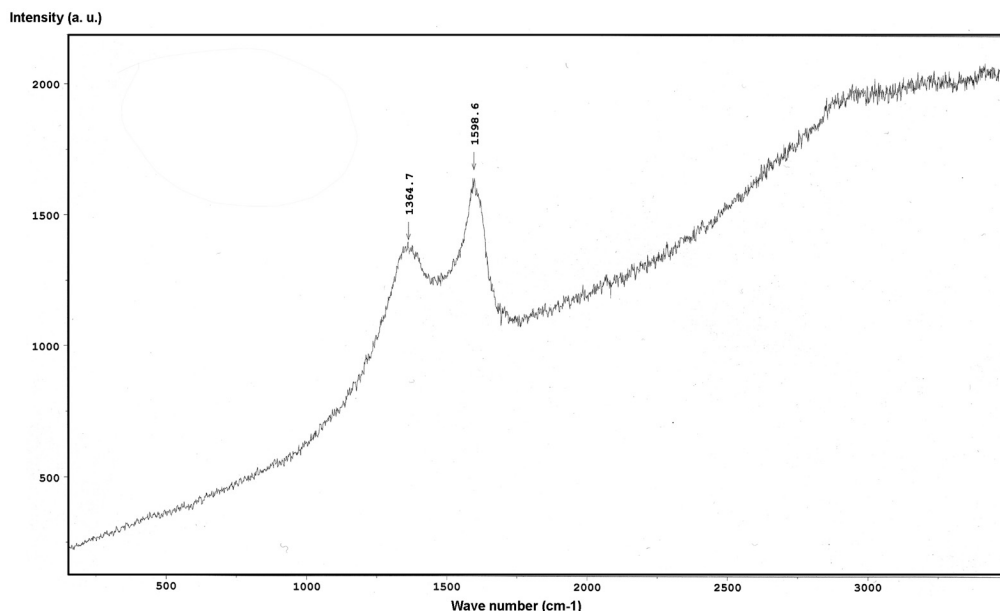


Figure 3. Raman spectrum of black grain present in the red decoration. We note two wide lines at 1364.7 and 1598.6 cm^{-1} characteristic of amorphous carbon (David *et al.*, 2001).

Black: It was carried out using a material made up of quartz and chromites. Copper is not a component of chromites $[(\text{Fe}, \text{Mg})(\text{Cr}, \text{Al}, \text{Fe})_2\text{O}_4]$. Its presence in the crystals would be due to the aptitude of chromites for capturing copper present in the transparent glaze (Tite, 1989).

4.2. Comparison with the first Iznik production

The comparison of these results with bibliographical data relative to the first productions (Tite, 1989) shows some differences which could be at the origin of the decline of this ceramics:

- The transparent glaze is a lead glaze whereas it was lead-soda glaze in the first productions (20 - 40% PbO).
- The amount of clay and soda-lead frit is more important in the first production.
- Concerning the “red Iznik colour”, the first production was made of iron-rich quartz-frit mixture whereas the late Iznik production was made with ferruginous sand.

5. CONCLUSION

The physical-chemical properties of the various parts of this complex material were determined.

The Raman spectrometry analysis proves that the studied “red Iznik colour” is composed of ferruginous sand and amorphous carbon. This is completely different from the famous red Iznik colour of the first production.

The comparison of our results with the first Iznik production shows many differences which can explain the “technological decline” of the late production. The main difference is the nature of the glaze. That “choice” supposes the change of firing conditions, glaze viscosity and preparation mode of the glazing mixture.

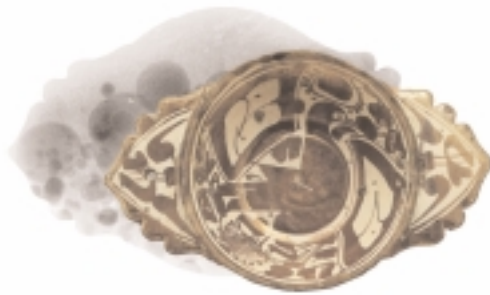
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34th INTERNATIONAL SYMPOSIUM ON ARCHAEOOMETRY

3-7 May 2004

Zaragoza, Spain



Institución «Fernando el Católico» (C.S.I.C.)
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CREDITS

PREFACE

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INDEX

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The logo on the front cover represents a piece of lustre pottery from Muel (16th century), Museum of Zaragoza. The shadow is a TEM image of Cu nanocrystals forming the lustre layer.

START