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An assessment study of Islamic period tiles (Timurid, Ottoman, Safavid and Qajar) from 12th to 19th centuries to trace the glaze technology

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SUMMARY:

Raw materials determine the final composition and properties of a fired ceramic. Distinctive minor (Ca, K, Al, Mg, etc.) and trace elements (Bi, Rb, Sr, Zr, etc.) of the geological context, which give significant signal, permit distinguishing the provenance and the phases characteristic of the firing procedures, thus differentiating different productions of tiles. In many cases, the most sophisticated part of the process is the formulation and fabrication of the glaze, especially the colored areas. Complete tiles, which are coming from identified historic buildings and exhibited in the museum showrooms are better documented concerning their origins rather than the unregistered fragmentary sherds. Therefore, a detailed analytical examination is required for these undefined tiles to identify where they are produced. Systematic studies with spectroscopic methods will lead to a statistical work in addition to the material culture and art historical research in the Islamic period (focused on the 12th and 19th centuries) ceramic art.

In this study, three techniques, Raman, portable X-ray fluorescence (pXRF) and scanning electron microscopy-energy dispersive (SEM-EDS) spectrometry were applied to reveal the technological characteristics of the glazes and pigments. Raman analysis which characterizes micro- and nano-structures of colored and transparent glazes, opacified or not, was applied to sherds mostly collected before the 1960s, preserved currently at the Louvre Museum¹. These tiles were originated in Anatolia, the Caucasus, Iran, and Central Asia, which are, for most of them, characterized by the application of black lines to separate coloured areas. Similar fragmented sherds which are preserved in the reserves of Topkapi Palace Museum in Istanbul were also analysed with Raman spectroscopy if either they were produced in the Ottoman territory or if outside^{2,3}. Chemical analysis of glazes and colored areas were performed with pXRF for the sherds of the Louvre Museum (29 samples) and SEM-EDS for the Topkapi Palace fragments (4 samples). Figure 1 shows the similar sherds from Louvre Museum and Topkapi Palace Museum analysed for comparison. The reliability of pXRF measurements was formerly revealed when compared with the results carried out with SEM-EDS⁴.

Raman measurements of the collection of Musée du Louvre were carried out in the laboratory with a blue laser excitation and/or on the conservation site with a mobile device equipped with green laser¹. The samples from Topkapi Palace Museum were analysed only with green laser in the laboratory^{2,3}. Comparison with XRF, SEM-EDS and IB composition measurement is made⁵. Three types of glazes were identified (Fig.2a): (i) a lead-rich glaze analogous to that of Byzantine, Zirid to Hafsid and al-Andalus productions, (ii) a lead-alkali glaze typical of the Ottoman productions of Iznik-Kütahya, and (iii) a mixed (poor lead/lead-free)-alkali glaze typical of Safavid productions.

Further comparison is made with the data collected with a different mobile XRF instrument on Edirne Mosque tiles⁶ (Edirne was the first Ottoman capital) and with those recorded on sherds excavated from Ottoman Iznik kilns⁷ (Fig.2b).

The colour depends on the precise composition of the glaze. The identification is not only based on the signature of the stretching mode of the SiO_4 tetrahedron (position of the wavenumber of the component(s)) but also on the position and shape of the continuous luminescence of the Raman background, characteristic of the raw materials used. For the determination of the luminescence, a wider spectral window up to 4000 cm^{-1} must be recorded with Raman, while the glaze signature only covers a spectral range between 300 and 1200 cm^{-1} . Lead-tin yellow, Fe-rich, Mn-rich and Cr-rich black pigments and opacifiers made of cassiterite and wollastonite were also identified in the colored areas. The Raman results (type of glaze and deduced processing temperature) were discussed in the light of the binary and ternary scattering plots showing oxide composition determined in the previous works (Fig.3) and the microstructure examination on the polished section (defining single or multistep firing cycles).



Figure 1. Representative tile sherds produced between 12th and 19th centuries from Louvre Museum (Paris, a-d, i) and Topkapi Palace Museum (Istanbul, e-h) collections.

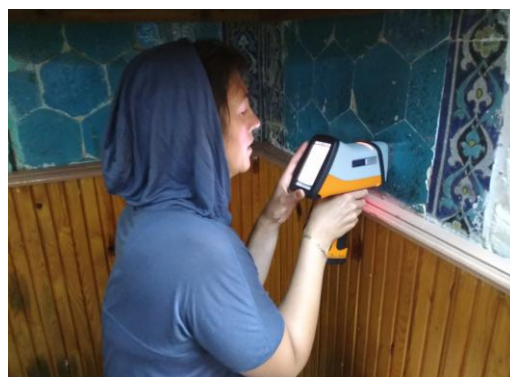
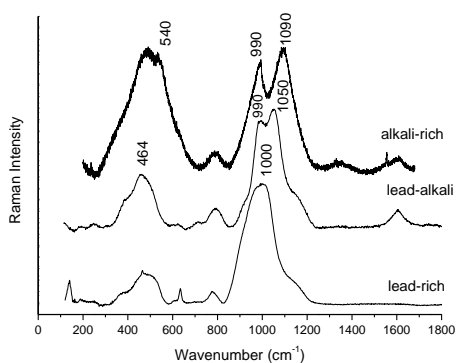


Figure 2. (a) Base-line subtracted representative Raman spectra showing different types of glazes (lead-rich, lead-alkali, and alkali-rich). The baseline has been subtracted in order to compare the bending to stretching band area ratio. (b) On-site measurement with pXRF in Edirne Şah Melek Paşa Mosque.

Continuity was highlighted on the one hand between the tiles of Bursa, Edirne, and Istanbul (Ottoman Iznik-like production) and on the other hand between those of Samarkand area (Timurid) and Iran. Finally, the criteria determined from the study were applied to object from the art-market to identify artefacts.

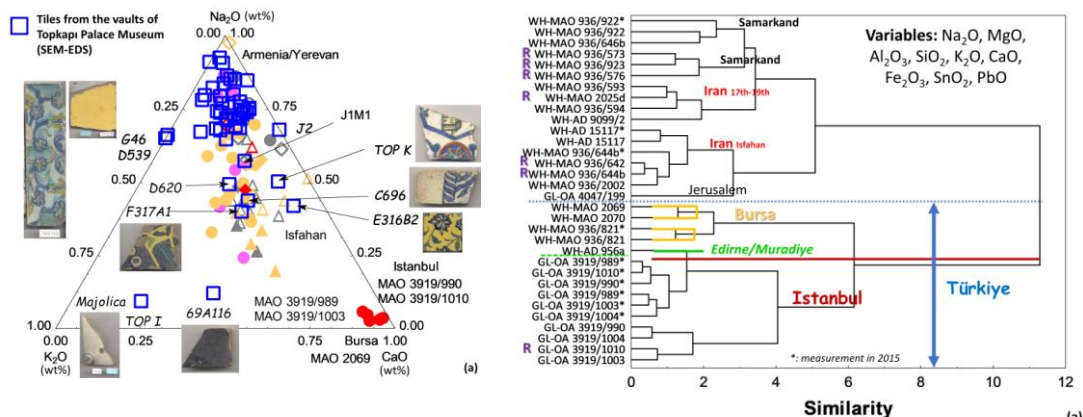


Figure 3. (a) Ternary scattering plot of fluxes (Na₂O, K₂O, CaO) and (b) hierarchical similarity dendrogram built from the variables (wt%) found in the white and transparent glazes.

KEY-WORDS: Raman spectroscopy, pXRF, Islamic period, glaze, pigments

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