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Micro-Raman and FT-IR spectroscopic studies of ceramic shards excavated from ancient Stratonikeia city at Eskihisar village in West–South Turkey

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ABSTRACT

In this study, micro-Raman and Fourier transformed infrared (FT-IR) spectroscopies, X-ray diffraction (XRD) and scanning electron microscope with energy dispersive X-ray (SEM-EDX) were used to characterize the mineralogical structures of pigments of four ceramic fragments in which one of them belongs to Hellenistic period (1st – IVth century BC) and other three ceramic shards belong to Early Rome (IVth century BC- 1st century AD) excavated from Stratonikeia ancient city. In the results of investigations on these four ceramic fragments, the various phases were identified: quartz, kaolinite, albit (or Na-feldspar), calcite, anastase, hematite and magnetite. Furthermore, the obtained findings indicate that firing temperature is about 800–850 °C for all the shards.

1. Introduction

Ancient Stratonikeia city is located at Eskihisar village of Yatağan town of Muğla province in West–South Turkey (Fig. 1). Although the first settle downs in this region became in the period of Bronze Age, the archaeological finds belong to the end of 2000 BC [1]. Stratonikeia was founded at the beginning of Helenistic period by the Seleucid King, Antikhos the first in the honor his wife Stratonike.

The first excavations in the mentioned archaeological site were verified by Prof. Yusuf Baysal and his team in 1977. However, the artifacts have been excavated under the auspices of Pamukkale University, Turkey, since 2008.

It is well-known that the analysis of the archaeological remains is quite important in order to obtain the maximum amount of information on an object for the archaeologists. Therefore, an interdisciplinary approach is asked for the analysis of pigments, clay, structures and mineralogical characterization of ancient ceramic fragments. Micro-Raman spectroscopy is a powerful non-destructive analytical method on ceramics, paintings, mummies and various ancient objects [2–8]. On the other hand, FT-IR spectroscopy as the complement of Raman spectroscopy provides useful information about the firing temperature performed for the production of ceramics [9–12]. Other two complementary methods can be considered as SEM-EDX and X-ray diffraction (XRD) on ceramics [13,14].

The purpose of this study is to present for the first time the results of IR and micro-Raman spectra of four ancient ceramic shards by considering data obtained from XRD and SEM-EDX measurements.

2. Experimental

2.1. Sample preparation

The ceramic fragments excavated from the archaeological sites were cleaned by conventional method. First, they are cleaning by using a brush under water without giving any damage to artifacts and then, were dried in air at room temperature. The specimens studied are described in Table 1.

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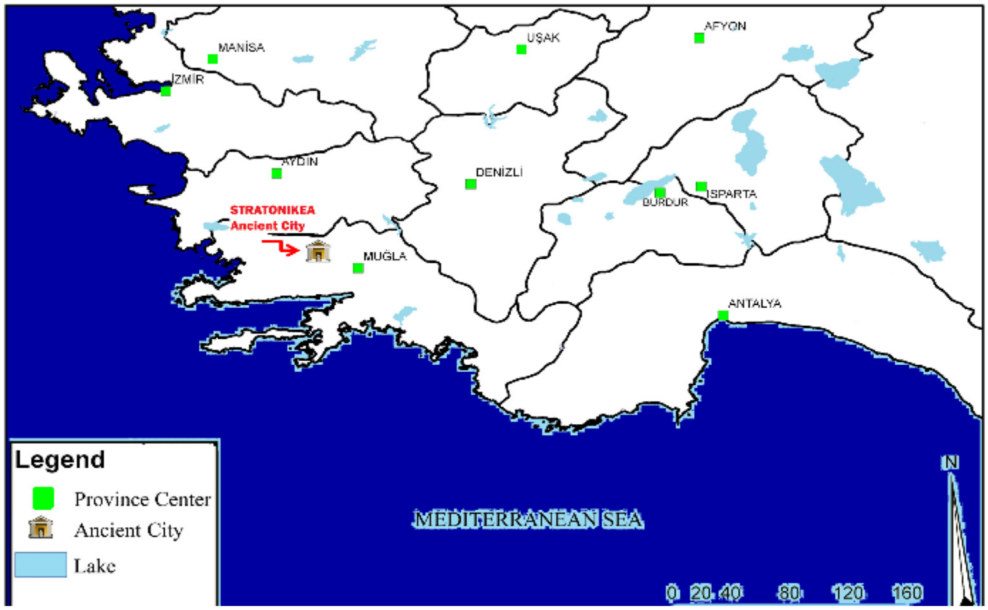

Fig. 1. The location of ancient Stratonikeia city in South–West Turkey (map with Assoc. Prof. Kadir Temuçin's courtesy).

Table 1
Data on ancient ceramic fragments.

Sample labeling	Description
STR-1	A shard of a large bowl (ca.IVth – 1st century BC. in Hellenistic period) excavated from ancient Stratonikeia city. STR-1a and STR-1b denote bright red and pale red regions on the outer side of the shard, respectively and STR-1c indicates a region on inner side of the mentioned shard.
STR-2	A shard of a plate excavated from Stratonikeia city (ca. IVth century B.C-1st century A.D in early Roman period). STR-2a and STR-2b show dark red and brownish yellow regions on the outer side, respectively, and STR-2c denotes dark brown region on the inner side of the shard.
STR-3	A fragment of a plate excavated from Stratonikeia (ca. IVth century B.C-1st century A.D in early Roman period). Outer side is from pale red (STR-3a) and dark brown coating (STR-3b). Inner side is dark brown coating (STR-3c).
STR-4	A fragment of a plate excavated from Stratonikeia (ca. IVth century BC -1st century A.D in early Roman period.). Outer side is dark brown coating (STR-4a) and inner side is black coating (STR-4b).

The photographs of analyzed samples are shown in Fig. 2. In these photographs the labeled circles show the parts of the ceramic shards from where the measurements are verified.

2.2. Instruments

2.2.1. X-ray diffractometer

XRD patterns were recorded using a X'Pert PRO (PW 3040/60 Model) powder diffractometer with CuKα (1.54060 A, 40 mA, 45 kV) at 0,02 steps at the rate of 0,5 per second over range $5 < 2\theta < 75$.

2.2.2. Scanning electron microscope (SEM)

The SEM images and energy dispersive X-ray (EDX) graphics of the pigments on the surfaces of ceramic shards studied in this work were monitored by VEGA-II LSU Variable Pressure Scanning Electron Microscope.

2.2.3. Micro (μ) –Raman spectroscopy

Micro-Raman spectra of the samples were recorded at room temperature using a Jasco NRS-3100 Laser Raman Spectrometer equipped with the CCD detector cooled at −50 °C. The excitation source was a diode laser, operating at 785 nm and scan number was 10.

2.2.4. Infrared spectroscopy

The samples were compressed into self-supporting pellets and introduced into an IR cell equipped with KBr windows. IR measurements at room temperature were performed on a Perkin–Elmer Spectrum One FT-IR (Fourier Transformed Infrared) Spectrometer with a resolution of 4 cm^{-1} in the transmission mode.

3. Results and discussion

3.1. XRD patterns

XRD analysis provides the recognition of the mineral characterization of ceramic potteries and the composition of the crystallographic phases which are related to their provenance [15]. XRD patterns of the specimens which are described in Table 1and shown in Fig. 2 are listed in Table 2.

As seen in Table 2, the existence of quartz (α-SiO_2) mineral is available for all of the samples. However, for the ceramic shard STR-1 the major primary mineral present in the sample is kaolinite [$Al_2Si_2O_5(OH)_5$] and the secondary mineral present is quartz and the accessory minerals present in the sample are the biotite which is a mica group mineral at the approximate chemical formula [$K(Mg,Fe)_3AlSi_3O_{10}(F,OH)_2$] and gypsum [$CaSO_4.2H_2O$].