



Spectroscopic analysis of XIV century wall paintings from Patriarchate of Peć Monastery, Serbia



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ABSTRACT

The Church of the Holy Mother of God Hodegetria in Peć is decorated with wall paintings that date from the beginning of the 14th century. In terms of style they correspond to Byzantine wall paintings from the epoch of Paleologos. The painting technique and pigment palette has been examined on micro fragments in thin cross-sections by means of optical microscopy (OM), scanning electron microscopy-energy dispersive X-ray spectroscopy (SEM-EDS) and micro-Raman spectroscopy. Use of the fresco technique and two supporting plaster layers was noted on the majority of samples, while in large blue painted areas, a combination of fresco and secco techniques was used. The SEM-EDS results showed the presence of Ca as the main component of plaster besides the traces of Si and Mg. In some samples egg white as a binder was identified. The paint film is often multilayered. Twelve pigments were identified, mainly natural earth pigments such as red ochre, yellow ochre and green earth. A mixture of pigments was used for attaining desirable optical and aesthetical impressions. As decay product only weddelite was detected in many preparatory and painted samples.

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1. Introduction

The Patriarchate of Peć is a complex of medieval churches, near the Kosovo town of Peć, along the river Pećka Bistrica at the mouth of Rugovska Klisura canyon. It consists of four churches connected with narthex, built from 13th to 14th century (Fig. 1a). Since then it was a center of the Serbian Orthodox Church and the seat of Serbian Archbishops and Patriarchs. The Church of the Holy Apostles of Peć Patriarchate was built by Archbishop Arsenije I (St. Sava's successor) in the third decade of the 13th century, when the Bishopric seat was transferred from Žiča Monastery to Peć. Between 1321 and 1324, Church of St. Demetrius along the northern wall was built. Later, Archbishop Danilo II built the Church of the Holy Mother of God Hodegetria ('one who shows the way') along the southern wall of the Church of the Holy Apostles. In the 1330s, he also built a narthex with a tower as well as the small Church of St. Nicholas along the southern wall of the Church of the Holy Mother of God Hodegetria [1].

The base of the Church Hodegetria has a shape of a developed inscribed cross. The octagonal lower dome is placed on free pillars, which separate the temple into three parts, down, by vertical axis. Walls made of stone and bricks were initially lime plastered, and then

painted with sacral decorations on fresh lime. There are two beautiful gothic windows on the altar apse and south wall.

Almost all wall paintings of this church were executed between 1334/1335 and 1337 [1]. The life of the Mother of God is presented on the walls. Among others, there are also "Grand holidays", a series dedicated to John the Precursor and a portrait of founder, Danilo II with the model of the church in his hands. Two frescoes, portraits of St Danilo II after his death and St. Ermolay, that form the second layer of the frescoes, may originate from the later decades of the 14th century. The wall paintings were made by three groups of unknown artists that followed the principles of Paleologus Renaissance with elongated figurations and fin artistic details and more vivid coloration than in earlier decades [2–4].

Big renovation work of the complex was done during the 16th century, now visible on fresco paintings in narthex. During a second renovation in the 17th century most of the frescoes from the Church of St. Apostles were repainted as well as some parts of the Church St. Demetrius. On the other occasion during the 17th century all devastated frescoes from Church of St. Nicholas were over painted by famous fresco painter Radul. Only in the Church Hodegetria the intact original fresco paintings are preserved. An entire history of the styles of medieval wall paintings in Serbia from the 13th till 18th century can be seen in this unique cultural and religious monument.

In the year 2006, monastery complex Peć Patriarchate was inscribed into the UNESCO World heritage in danger list due to the difficulties in

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Fig. 1. a) Image of the Peć Patriarchate complex, Serbia; b) dome of the Church Hodegetria; c) detail of a wall painting with the efflorescence; d) detail of a wall painting with craquelures.

its management and conservation, stemming from the region's political instability. Since then, the complex is under permanent conservation. In the restoration campaign in year 2014, the conservation work on fresco paintings and the renovation of the roof of the church Hodegetria was done.

In the present study, undertaken before the 2014 restoration, micro-fragments of the original painting material dated from the beginning of the 14th century from the church Hodegetria were analyzed by a combination of micro Raman and Fourier transform infrared (FTIR) spectroscopy, optical microscopy (OM) and scanning electron microscopy-energy in order to recommend the proper cleaning solutions and consolidation procedures dispersive X-ray spectroscopy (SEM-EDS). The aim of our research was to investigate the painting technique and characterize the chromatic palette of these wall paintings. The painting technique and pigment composition obtained in this study were used in the conservation procedure, in order to recommend the proper cleaning solutions and consolidation procedures.

2. Experimental

2.1. Samples

Before restoration, micro samples were extracted with a scalpel from colored areas of a wall painting; some of them were analyzed directly, while the majority were prepared as cross sections by their embedding into an epoxy polymeric resin Araldite 2020, than polishing with grinding paper (mesh 500, 800 and 1000). The samples were subjected to examination by OM, SEM-EDS, FTIR and micro-Raman spectroscopy. The description of the samples is given in Table S1 (Supporting information).

2.2. Instrumentation

Prior to the spectroscopic analysis, cross-sections of the samples taken from the wall paintings were observed under OM. Cross-sections of investigated samples were recorded by an Olympus Camedia digital camera mounted on an Olympus BX51M microscope equipped with UV lamp Olympus U-RFL-T and U-MWUS3 and U-MWBS3 filters. Magnifications were 50 \times , 100 \times and 200 \times .

In order to perform SEM-EDS analyses, all investigated cross sections of investigated samples were coated with carbon. Major and minor element oxides were analyzed by an energy-dispersive X-Max Large Area Analytical Silicon Drifted spectrometer (Oxford) coupled with scanning electron microscope JSM-6610 LV. The analyses were done under acceleration potential of 15 kV, a beam current of 20 nA and a spot size of 1 μm . Appropriate internal and external standards were used for the analyses. The detection limit was 0.1%.

FTIR spectra of the powdered samples, dispersed in potassium bromide and compressed into pellets, were recorded in the range of 4000–400 cm^{-1} at 64 scans per spectrum at 2 cm^{-1} resolution using an Avatar 370 FTIR Spectrometer (Thermo Nicolet).

The Raman spectra were recorded from as-collected fragments or from epoxy-embedded polished cross sections on a DXR Raman Microscope (Termo Scientific). The 532 nm line of a diode-pumped solid state high brightness laser was used as the exciting radiation and the power of illumination at the sample surface ranged between 2 and 10 mW. Collection of the scattered light was made through an Olympus microscope with infinity-corrected confocal optics, 25 μm pinhole aperture, standard working distance objective 50 \times , grating of 1800 lines/mm and resolution of 2 cm^{-1} . Acquisition time was 10 s with 10 scans. The laser spot diameter on the sample was 1 μm . Thermo Scientific OMNIC software was used for spectra collection and manipulation.

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