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Case study

Pink discoloration on frescoes from Hurezi Monastery, Romania

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ABSTRACT

A multianalitical approach based on optical microscopy (OM), scanning electron microscopy (SEM), X-ray diffraction, grain size distribution and microbiological methods has been applied to characterize pink discoloration on the surface of both original painting and lime-mortar infillings of the frescoes of the refectory from the Hurezi Monastery, Romania. Polarized microscopy, the study of the cross-sections, X-ray diffraction and grain size distribution pointed out the characteristics of materials and led to a better analysis of composition and the availability to be colonized. Thick layers of bacteria developed around and into enlarged pores led to the alteration of substrate pore sizes and changes of moisture circulation. Acting simultaneously with efflorescences, disaggregation and fragmentation of the mortar and pictorial layer take place. Microbial origin of pink discoloration detected by OM and SEM was confirmed by culture based methods. The present study points out the analytical methods for identification of pink aesthetical damage of mural painting and its biological origin.

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1. Research aims

The present work reports the microbial origin of the pink discoloration found in the refectory of the Hurezi Monastery from Romania, on the original frescoes, on the surface of the original and infilling mortars. This is essential for mapping biodeterioration and damages, as well as for the development of treatment for the mural painting. We propose analyzing pink discoloration through analytical techniques, which highlight the significance of multidisciplinary investigation for establishing the diagnosis. Results will be also used for an in progress work concerning the development of the mortars for restoration of pink discolored mural painting.

2. Introduction

Microbial colonization of mural paintings is a complex process that shows the interaction of microorganisms with the substratum and environment [1].

Although the pink discoloration has been pointed out in literature on stone, wall paintings, building and burial-related material from monuments in central and southern Europe [2–4], information on its retrieval on the historical monuments from Romania are lacking. Pink discoloration affects monuments exposed to different climatic conditions with constructional problems that enable water infiltration [2]. Salt efflorescences produce additional pressure in the pores and on the surface, leading to cracking and detachment of the materials [5]. Köcher and Müller [6] proved that moderately halophilic bacterium *Halobacillus halophilus* produces carotenoids to protect the cells against photo-oxidative damage and salt stress, representing a strategy to survive in extreme environments. This way, the original color of the pictorial layer is changed and pink spots are visible on the mortar. Imperi et al. [7] found widespread change in the color of superficial layer of frescoes that turned pink, consequent to particularly dry and hot summer season. Binders like casein, vegetable debris and organic particulate matter were proved to sustain microbial growth [8]. The rosy discoloration was found to be also produced by phototrophs as chlorophyta and cyanobacteria [9].

The Monastery of Hurezi is one of the main and largest monastic ensembles in southern Romania. In early eighteen centuries, a refectory (Fig. 1a) was built in the western side of its axis and in

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Fig. 1. General view of the refectory and the distribution of pink discoloration on the northern wall: a: the entrance; b: niche; c: northern wall.

1705, it was decorated with frescoes following Byzantine tradition. Later on, the eastern and western walls of the refectory, as well as the inner side of the niches have been covered by massive over paintings on the lower side of the walls. The last restoration of the refectory took place in the 70s, when large areas of lacunae at the lower part of the walls have been treated by mortar infilling. On the northern wall of the refectory, an irregular pink discoloration is visible on the surface of the infillings (Fig. 1b), going up to the original frescoes (Fig. 1c).

3. Materials and methods

Grain size distribution of siliceous aggregate was established after its separation from the binder. The aggregate separation was performed through wet chemical separation method [10] and the grain size analysis was done both in *arricio* and *intonaco*. The remained material on sieves (0.55 mm, 0.71 mm and 1 mm) washed and dried was weighed and the material percentage of each particle size class was calculated. The northern wall with infilling mortar colored in pink and white was sampled at 120 cm above the ground.

X-ray diffraction analysis was performed using a Shimadzu XRD 6000 diffractometer, with Ni-filtered CuKα radiation (λ = 1.5406 Å), with scan step of 0.02°. The total amount of sample used was 1 g of powder.

The samples have been embedded in polyester resin and cross-sectioned with IsoMet Low Speed Saw, submitted to wet polishing and glossing using the instrument Phoenix Beta Grinder/Polishe. The sections have been observed at optical microscope Carl Zeiss AXIO IMAGER A1m endowed with soft and video camera. The analysis was performed on polished sections and thin sections, in polarized light, with cross Nicols and parallel Nicols. The dimension of the field visualized in figures is of 700 × 520 μm.

Microphotographs of the pink discoloration had been obtained *in situ*, using a Dino-Lite microscope on the northern wall, mostly on the pictorial layer. For the infilling mortar samples, microphotographs were obtained in laboratory with Nikon AZ100 microscope and with scanning electron microscope (SEM) JEOL JSPM 5200 (Japan), in order to identify bacteria and their types of arrangements.

The northern wall was sampled at 150 cm above the ground getting 15 samples for microbiological analysis. The infilling mortar samples were taken using a little hammer and a chisel; the average sizes of the fragments never exceeded 2 cm for all analyses. Samples were collected under aseptic conditions into sterile boxes and stored at 4 °C until processing. For microbiological analysis, the samples were weighed (g), the decimal dilutions were inoculated on solid media supplemented with 10%, respectively 20% NaCl [11] and incubated at 28 °C for 35 days. Pure cultures were obtained using depletion loop technique. Tolerance test was performed in liquid media with different NaCl concentrations.

4. Results and discussion

4.1. Microclimate monitoring

HOBO LCD data loggers positioned at a height of 2.75 m on the southern and northern wall of the refectory monitored temperature and relative humidity (RH) at every 30 minutes. They revealed values of temperature between 2 and 24 °C and relative humidity between 37 and 95%. The highest temperature value was registered in autumn and summer and the lowest in winter. In case of RH, the entire year the highest value was of 80–95%.

4.2. Technical analysis of mural painting

The frescoes of Hurezi refectory are executed in the Byzantine tradition of 'true fresco': the support of the painting is composed of two distinct layers of mortar (*arriccio* and *intonaco*). The *arriccio*, applied on brick masonry, consists of calcium carbonate (calcite) as binder and river sand (quartz, potassic feldspar and muscovite) in proportion of about 65% (by mass) as mineral aggregate. The *intonaco*, consists of calcium carbonate as binder, 6–5% (by mass) river sand as mineral aggregate and short hemp fibres as vegetal aggregate. The river sand grains are more frequently angular and sparsely round, coloured in ochre grey, in size up to 2 mm and belong to grain size fraction under 0.5 mm.

Light microscopy performed on cross-sections and thin sections on the pink infilling mortar indicated a pink coloured porous mass in the *intonaco* layer (Fig. 2a), produced by bacteria; both layers contain granules of aggregate and cementing calcite (binder) with a fine granulation (Fig. 2a and b).

The aggregate composition consists of: sub/angular-rounded fragments of quartz with diameters of 50 μm up to 250 μm (Fig. 2b), sub-angular-rounded fragments of quartzite with dimensions up to 500 μm (Fig. 2c and d), sub-angular fragments of feldspars with diameters up to 250 μm (Fig. 2d). The granulation is below 2 mm, with a weight of 41–68% of fine fraction below 0.5 mm.

X-ray diffraction analysis put into evidence for *arriccio* layer compounds of binder – lime (calcite 29.43 Å, 39.47 Å – PDF 83-0578) and of aggregate – river sand (quartz 26.75 Å, 21.00 Å – PDF 83-0539, potassium feldspar 27.60 Å, 21.01 Å – PDF 76-0826, muscovite 8.89 – PDF 06-0263) and a salt of sodium carbonate [$Na_3H(CO_3)_2 \bullet 2H_2O$ – throne 33.88 Å, 29.07 Å – PDF 78-1064] as a product of degradation. We suppose that trona was developed due to soluble alkali coming from the cement used for consolidation of the building in 1970.

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