CHARACTERIZATION OF HISTORIC MOSAIC AT PFEIFFER-KRAL SEPULCHER, JABLONEC NAD NISOU: A STUDY OF THE MORTAR AND TESSERAE ORIGIN

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The art of mosaic in the Czech lands in the 19th century generally reflects the religious sentiment of the artists and their customers. As they are very expensive and require a particular technique and technology, the mosaics are not commonly used as a decorative part of architecture. Exceptions may be found in the decoration of churches or municipal monuments. They are also specifically used on the sepulchers of significant families. Before the start of restoration work on Jablonec nad Nisou mosaic historically used materials were studied. The characterization of mortar and glass tesserae helps to substitute, as closely as possible, the missing original ones. The work on material characterization concludes in high probability that Italian glass tesserae were mounted into "old Italian mortar" style.

INTRODUCTION

The technique of mosaic is well known from the ancient Greek culture (6th century B.C.). Having come probably from the Middle East, it famously developed and spread throughout the Roman Empire. Historic mosaic floors now embellish the floors of villas and churches in Italy, North Africa, Middle East, Spain and southern France [1, 2].

Ornamental and figurative decoration was first composed of stone pebbles of different colors; later, small stone and glass cubes (tesserae) were prepared by cutting, hacking and chopping and fixed in calcareous mortars. Over time, especially on the Isle of Murano in Venice, various glass technologies were developed, among others also the technology of the catted mosaic glass cubes so-called "smalti". The wide range of colored glass opened new possibilities for the mosaic renaissance in Italy; the knowledge subsequently spread all over Europe [3-6].

An example of a monumental glass mosaic in Central Europe can be found on the southern façade of Saint Vitus Cathedral in Prague, Czech Republic. This medieval mosaic – "The Last Judgment" – was implemented on the "Golden Gate" entrance during under Charles IV (1316-1378), Holy Roman Emperor and King of Bohemia.

The object of the presented article is a specific exception – the private sepulcher of the Pfeiffer-Kral family in Jablonec nad Nisou. The Pfeiffer family played an important role in the development of Czech glass industry from the beginning of the 19th century in the region of North Bohemia and mainly in the town of Jablonec nad Nisou. In 1839, Adolf Pfeiffer after the death of his father Josef, assumed the main responsibility for the family enterprise. His personal aim was the production of glassware, namely costume jewelry, pearls and buttons, small religious objects and also decorative bedecked parts of lamps as well as metallic jewelry. Adolf Pfeiffer invested efforts into the development of aventurine glass and namely into the technology of aventurine pearls. This type of production had been developed by Italian glass masters in Venetia. In 1847, A. Pfeiffer invited Italian glass makers to his glass factories in Jablonec and with their participation he developed the Czech variation of "smalti" mosaic technique and the specific glass technology of "millefiori" in Jablonec.

It is well known that the Pfeiffer family demanded only "*the best products and the best artisanal works available on the market*" [7]. This is proven by the family sepulcher; the construction is done in the early Art Nouveau style. Unfortunately, there is no documentation on the construction or the participation of the artists in the decoration of this private monument. The successors of the German-speaking family, the owners of several glass factories, forests and other property, were entirely excluded from society after the Second World War and finally exiled from the country. The anti-German mood in the area along the Czech borders, annexed by Nazi Germany in 1937, was extremely strong; consequently, many historical documents disappeared and many historical truths were modified.

The lack of information should be compensated by the analyses of glass tesserae and analyses of the mortar used on this sepulcher before any restoration work begins. The main aims of the presented work are:

The issue of the origin of glass tesserae. Were the glass tesserae fabricated in Italy or are they a product of Czech glass factories?

The issue of the mortar type and its identification. The identification of the mortar composition is an important aim of this work.

EXPERIMENTAL

Restoration background

Description of the monument

The decorative mosaic (opus musivum) of the sepulcher niche runs along the top of the round bronze relief. The strip is approximately 5 meters long and 1 meter wide. The decorative mosaic is composed of eight segments, repeating a vegetable decorative band interlocking green thistle flowers, haulms and foliage motifs bordered by a gold line over the blue background. Each segment of the mosaic is axisymmetric. The mosaic was made from glass tesserae and mounted without visible joints between them. The pure Art Nouveau decoration could be attributed to the work of Italian masters or to the Neuhauser master atelier in Innsbruck, Austria.

The actual state before the restoration work

The glass tesserae were partially dislocated from the mortar and in some places totally missing (Figure 1). The mortar degradation is reflected in the mortar relieving from the wall. The weathering is a consequence of long-term humidity caused by the absence of the roof maintenance. The glass tesserae tested by visual observation did not exhibit any visible imperfections. The tesserae were only grayed, slightly covered by grease deposits on the surfaces, but the tesserae containing gold leaves under the thin glass coat were visibly damaged, some of them even without the protective coating layer, the so-called "cartellina" glass. The cartellinas were relieved just like an uncovered golden leaf by a simple finger touch.

Restoration work

The degree and extent of mortar degradation led to the decision to transfer the mosaic. The first step was to make a perfect photographic documentation. After that, the transfer started by copying in detail the ornamental segment by the specification of each mosaic tesserae color onto transparent sheets. The adhesive Dispercoll D2 was used to fix two layers of protecting gauze. Before moving the mosaic, it was necessary to prepare a plaster support which exactly copied the form of the sepulcher arch. The plaster support was reinforced by fencing and tissue and its construction was secured by the wooden slates. This plaster negative "mold" will be used in later work – the preparation of the positive copy of the rounded arch onto which the mosaic will be finally transferred.

Mosaic transfer

The mosaic mortar was reinforced by copper bars which were brass nailed to the sepulcher construction. The penetrating water generated salt efflorescence, which caused decomposition and mortar degradation, resulting in the deallocation of the tesserae. The undamaged part of the mortar conserved its compactness and fixed the tesserae excellently until the transfer. The mosaic was divided into 18 parts depending on the state of mortar and its reinforcement. In some cases, tesserae fell from the damaged mortar, but all of them were recollected and will be used during the restoration work.

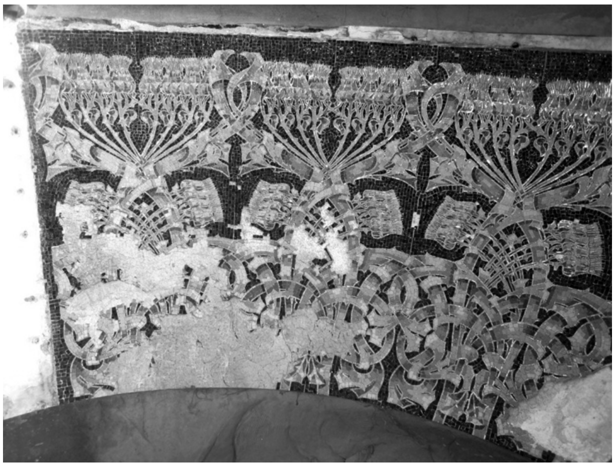

Figure 1. The condition of the mosaic before the transfer (one and half decorative segments).

The current condition

The mosaic decomposed into 18 parts was transferred to the restorer's atelier and all tesserae were mechanically cleaned to remove the rest of the mortar. The samples of the mortar and colored tesserae were studied in laboratories to resolve the main issues of the work – the mortar and glass origin.

Methods

Non-destructive X-ray fluorescence (XRF) spectrometry (Spectro IQ, Kleve, Germany) was used; the target material was palladium and the target angle from the central ray was 90°. The focal point was a 1 × 1 mm square, and the maximum anode dissipation was 50 Watts with 10-cfm forced-air cooling. The instrument is equipped with the Barkla HOPG crystal. The tested samples were prepared by the pressed-pellet method:

The 4.0 g of material (a particle size of 15 - 20 μm) were mixed for 10 minutes with 0.9 g of the binding additive (HWC Hoechst wax, Germany). The pressing power was 80 kN. In the case of the tesserae, the material was tested without pretreatment.

The X-ray powder diffraction data were collected at room temperature with an X'Pert PRO θ-θ powder diffractometer with parafocusing Bragg-Brentano geometry using CuK_α radiation (λ = 1.5418 Å, U = 40 kV, I = 30 mA). The data were scanned with an ultrafast detector X'Celerator over the angular range of 5-60° (2θ) with a step size of 0.0167° (2θ) and a counting time of 20.32 $s \cdot step^{-1}$. The data were evaluated and the mineral phases identified in the HighScore Plus software package.

The measurement of the infrared spectra was performed by the Nicolet 7600 spectrometer (Thermo Nicolet Instruments Co., Madison, USA) with a DTGS detector and a KBr-ray distributor. The parameters of the measurement were the following: the number of the spectrum accumulation was 128, a resolution of 2 cm^{-1}, KBr-tablet transmission and Happ-Genzel apodization. The spectra were evaluated by the Omnic 7.3 computer program.

The sample surface was studied by Scan Electron Microscopy (SEM). The sample was Au-coated on a K550X Emitech sputter coater in an Argon atmosphere and examined on a Quanta 450 scanning electron microscope. The high vacuum mode was used and the images were taken on the Everhartd-Thornley detector (ETD) in a secondary electron mode. On the same microscope the X-ray qualitative microanalysis of chemical elements was made with an energy dispersive spectrometer (EDS) silicon drift Si(Li) Apollo detector with a FET preamplifier. The data acquisition was done using the EDAX TSL OIM software with ZAF corrections.

RESULTS AND DISCUSSION

The identification of the materials used in the historic mosaic was divided into two parts – the first was the identification of the mortar in which the tesserae were mounted. The XRD, XRF and FTIR analyses were used and the microstructure was observed by the SEM – EDAX analysis.

The second part was devoted to the XRF chemical analyses of the glass used comparing the results with genuine Venetian tesserae.

Mortar

The laboratory of the IRSM, Academy of Sciences, tested the mortar from this mosaic. It was found that also in highly weathered mortar it is possible to observe particular brow-red ceramic shards. Figure 2 depicts particles up to 100 μm. Much smaller powdered particles are present everywhere in the mortar.

The technique of adding low-fired potshards (roof tiles, broken bricks, etc.) is well known from the Roman times. The Italian expression for this material is "cocciopesto" [8]. The addition of milled ceramic shatters considerably changes the quality of the mortar, increasing its durability by adding to it the hydraulic properties which were used in the construction of water tanks, cistern, municipal bath and so on [9-11]. Czech references mention this type of additives in 1839 (J. S. Presl [12]) and in 1863 (F. Kubert [13]). The technology of ceramic additives to the calcareous mortars was not unknown – it was described in the historical publications from the half of the 19th century has described.

The analysis acquired by the electron microscopy (Figure 3), at a selected point "A" (a chosen point containing predominantly powdered ceramic mass) has the following elemental composition (see Table 1),

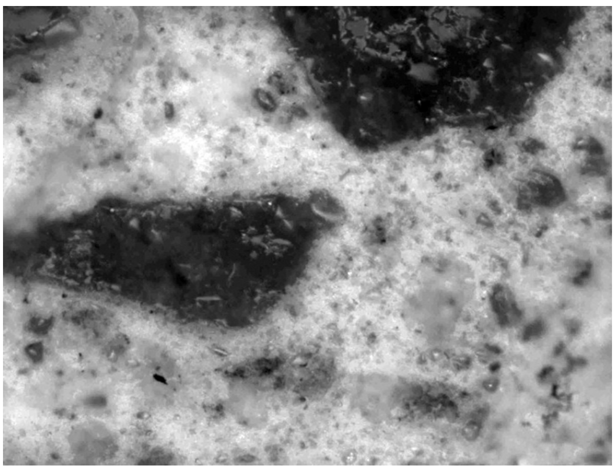

Figure 2. The surface of the mortar with visible particles of potshards (dark places).

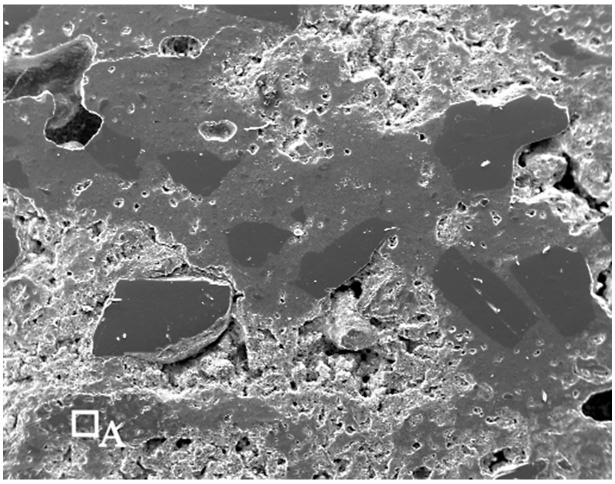

Figure 3. The surface of the mortar sample examined by SEM (point A).

Table 1. The participation of the elements at “point A” as revealed by SEM- EDAX analysis.

Element	C	O	Na	Mg	Al	Si	K	Ca	Ti	Fe
%	7.20	29.97	0.94	0.68	15.24	34.42	1.88	4.98	0.53	4.14

which corresponds to the common clayed material.

The SEM-EDAX analysis (Figure 3, point A) has shown the predominant participation of the alumina-silicates with the participation of the calcium-potassium content. This means that the composition corresponds to the most common clayed material used in the brick and roof-tile production in Central Europe. This basic clayed material is usually accompanied by a certain participation of feldspar and micas, corresponding in the presented analysis to the content of sodium and magnesium, which of course could appear in carbonates too. The red color is easily explained by ferrous oxides.

In this context, the average composition of the mortar is more important. A milled sample with a grain size below 20μm was used for the identification of the chemical composition by XRF analysis. Mineralogy was defined by XRD analysis. The samples were also analyzed by FTIR spectroscopy with the aim to find chained structures corresponding to the pretended “pozzolanic” effect of the powdered ceramic material in the calcareous matrix. Table 2 shows the chemical composition of an average mortar sample (from 6 sampling points) containing also the loss on ignition (LOI).

Table 2. The chemical composition of the mortar.

Oxide	MgO	Al_2O_3	SiO_2	SO_3	CaO	TiO_2	Fe_2O_3	LOI
Wt. %	1.01	4.33	13.35	2.25	41.87	0.29	2.07	33.45

The content of calcium oxide (recalculated from XRF analysis) ensures that the main element of the mortar was calcium carbonate admixed with clayed material. The content of sulfur-oxide signals the weathering and formation of gypsum ($CaSO_4.2H_2O$), a typical product known on the surfaces of calcareous mortars and sculptures originating from calcite ($CaCO_3$). The approximated calculated content (omitting micas and ferric/titanium oxides) of the components of the mortar (according to the stechiometric ratios and using the LOI data):

Calcite ....75.0 wt. %

Gypsum ....4.0 wt. %

Clay (ceramic)...8.5 wt. %

Quartz sand ....10.0 wt. %
(with high probability a part of the ceramic body)

The analysis of the sample by XRD supports the previously calculated composition and Figure 4 shows the predominance of calcite, complemented by quartz (SiO_2) and gypsum.

When the chemical analysis and the XRD pattern of “pozzolanic” materials were compared with chemical analysis was observed that the main problem is to find and identify the crystal phases containing an aluminum ion. More than 4 wt. % (Table 2) of alumina in its natural state forms alumina-silicates, in our case the clayed material of ceramic shards. The form and type of the connection between these two elements (Al and Si) was studied by FTIR analysis. Figure 5 presents the results of the FTIR analysis of the mortar from the Jablonec mosaic.

The FTIR analysis detects a vibration band at 459 cm^{-1} and also a band at 1038 cm^{-1}, which belonged to the chained structures of Si–O–Al. This confirmed the presence of aluminum on one side and its connection to the silicon by an oxygen bridge.

The recognition of the vibrations defined by CO_3 bonding is depicted in Figure 4. This bonding corresponds to the calcium carbonate. $CaSO_4$ bands were also

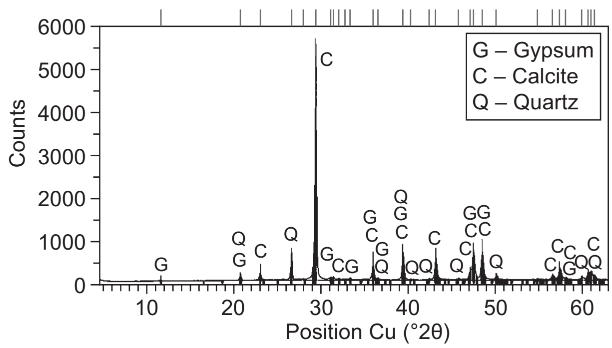

Figure 4. The XRD pattern of the mortar sample from the Jablonec mosaic.

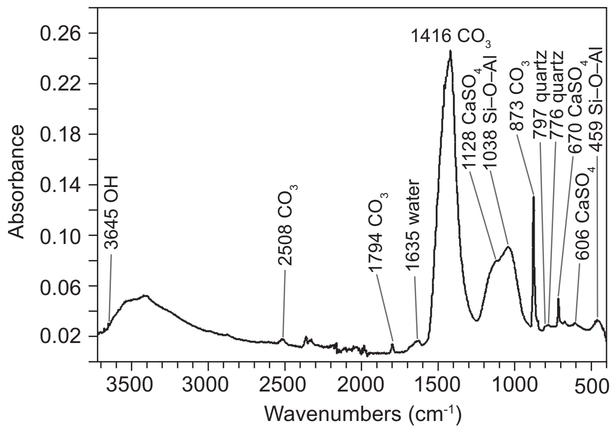

Figure 5. The pattern of the FTIR analysis of the Jablonec mortar.

clearly defined. The confirmation of gypsum appearance is supported also by the identification of the bands corresponding to the water and OH groups.

The Roman and also medieval common ceramic production used relatively low firing temperature (about 800°C) provided shards of high "pozzolanic" quality [14, 15]. The studied mortar is based on a calcareous matrix with admixed, low-fired, ceramic powder. The explanation of this phenomenon comes with the studies of the aluminum coordination to the oxygen. The natural state of the double-layered alumina-silicate means regular connections between the tetrahedrons of silicon and the octahedrons of aluminum. After firing at low temperatures, which means close to 800°C, the aluminum ions change their position being surrounded by four oxygens only. Both groups involved form tetrahedrons. In the alkali aqueous condition, a chained structure is created. Two misbalanced alumina ions must be equilibrated by two positive charges of alkalis or alkaline-earth metals [15, 16].

The two chained structures $Si–O–Al^{(-)}$ are balanced by one positively charged ion of calcium. These chained structures containing calcium form shorter or longer "fibers" in the calcareous structure of the mortar. These so-called "fibres" are amorphous and consequently hardly identifiable by XRD studies. The above-mentioned "pozzolanic" character is explained by the partial geopolymerization of alumino[1]-silicates in the calcareous matrix. The resistance and hardness of these mortars has been proven over centuries.

[1] *According to the JACS (Journal of American Ceramic Society), the alumina-silicate appears in the crystal form of silicates; when there is no exactly defined structure, the alumino-silicate must be used.*

Tesserae

Even though the first microscopic observation on glass tesserae was positive, with no damage on the surfaces, the first XRF analyses failed, detecting practically only silicon and lead (Table 3 – unpolished blue tesserae). Prepared, well-polished surfaces were later studied by the same method (the superficial layer of 0.4 mm was eliminated). The results are summarized in Table 3. (The elemental composition was recalculated to oxides.)

The characteristic of the glass through the chemical analyses shows natrium-calcium based glass, softened by the lead oxide. The found cuprum-ferric for the color green and cobalt-ferric for the blue tesserae is a common combination used in colored glass production. Such combinations are similar to the glass tesserae produced in Italy (Venetian glass factories), which are presented in Table 4.

Comparing the chemical composition of the sepulcher glass tesserae (beginning of 19^{th} century) with tesserae glass studies [3, 4] of Venice's ones could give the answer. Especially in case of dark green [3] there are similarities: Glass was formed through the sodium-calcite silicates softened by the flux lead oxide, the content of coloring oxides (Cu and Fe) combination also match with previously presented data (Table 3).

CONCLUSION

In this paper, the results of mortar and tesserae samples from Pfeiffer-Kral Sepulcher at Jablonec nad Nisou were discussed. The characterization of these historical materials is fundamental for planned restoring work.

The binder analyses detected a mortar type as an Italian "cocciopesto" style, commonly used also behind the Alps in 19^{th} century but have not confirmed the direct Italian origin of the mortar. It is impossible to define the origin of the mortar materials but the average chemical analyses of taken samples show the combination of lime and current potshards which were certainly available locally.

The presented chemical analyses of glass tesserae have proved the composition of sodium-calcium based glass with a rather high content of lead oxide and a

Table 3. The chemical analyses of the green and blue tesserae from sepulcher mosaic.

Oxide	Na_2O	Al_2O_3	SiO_2	K_2O	CaO	MnO	Fe_2O_3	CuO	Sb_2O_5	PbO	Bi
Green	17.76	1.12	57.84	0.83	3.65	0.29	0.37	1.35	1.77	12.14	0.18
Blue	19.66	0.52	63.88	0.55	4.92	–	0.22	0.24 CoO	0.50	7.28	0.10
Blue (unpolished)	0.29	0.77	78.08	0.71	7.07	0.02	0.33	0.44 CoO	0.42	10.34	0.12

Table 4. The chemical composition of the historical tesserae from Venice [3, 4].

Oxide	Na_2O	Al_2O_3	SiO_2	K_2O	CaO	MnO	Fe_2O_3	CuO	Sb_2O_5	PbO	SnO_2
Green 10 [4]	11.25	1.96	52.84	3.93	6.44	1.09	0.97	2.38	0.75	13.33	1.80
Green F6 [4]	9.16	1.15	49.11	4.44	1.26	0.03	0.28	2.35	9.83	19.23	9.83
Green 9G [4]	10.75	4.48	52.75	4.80	5.59	1.39	2.23	4.29	0.02	5.61	4.09
Dark green [3]	12.85	2.49	64.00	0.82	7.82	0.12	0.48	1.48	–	6.12	1.91

comparison with published results has shown a similarity with historical Italian glass tesserae. These results lead to the conclusion – tesserae were imported from Venice.

The characterization of the used materials showed that mortar type could be substituted from the present local market but the missing tesserae must be imported from the original provenience. The restorer will be in a similar situation as architect of the sepulcher – Italian tesserae mounted to the “Italian style” mortar but combined from the local market materials.

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