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Pozzolanic mortars for the conservation of old masonry structures

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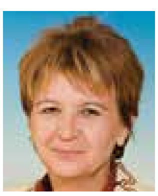

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Professional paper

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Pozzolanic mortars for the conservation of old masonry structures

The design of repair mortars started from the characterization of the original mortar samples preserved in small areas in the floors of the Dungeon Tower, Bač Fortress. Two materials (waste brick and clay material) were investigated in order to select the pozzolanic material which later was a component of the repair mortar. Based on the compatibility test of the examined old mortars and of the newly developed mortars, slaked lime based, the mortar with the ground waste brick as a pozzolanic material was selected for the conservation treatment of the Dungeon Tower.

Key words:
clay, pozzolan, conservation, medieval fortress, slaked lime, compatibility

Stručni rad

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Pucolanski mortovi za konzervatorsku obradu starih zidanih konstrukcija

Projektiranje sanacijskih mortova počelo je karakterizacijom uzoraka starog morta sačuvanih na malim prostorima podova donžonske kule u bačkoj tvrđavi. Ispitana su dva materijala (stara cigla i glineni materijal) da bi se odabrao pucolanski materijal koji je kasnije upotrijebljen kao komponenta sanacijskog morta. Na temelju analize kompatibilnosti ispitanih starih mortova i novoprojektiranih mortova baziranih na gašenom vapnu, za konzervatorsku sanaciju donžonske kule odabran je mort u kojem se kao pucolanski materijal koristi drobljena otpadna cigla.

Ključne riječi:
glina, pucolan, konzerviranje, srednjovjekovna tvrđava, gašeno vapno, kompatibilnost

Fachbericht

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Puzzolanischer Mörtel für die Erhaltung alter Mauerwerkskonstruktionen

Die Zusammenstellung von Reparaturmörteln ist mit der Charakterisierung von Mörtelproben des bestehenden Füllmittels begonnen worden, das in begrenzten Bereichen des Kerkerturms der Bač Festung erhalten geblieben war. Zwei verschiedene Stoffe (Ziegelschutt und Tonerde) sind untersucht worden, um das Puzzolanmaterial zu wählen, das als Bestandteil des Reparaturmörtels verwendet werden sollte. Auf Kompatibilitätsproben des bestehenden Füllmittels und der neu entwickelten Mörtel beruhend, ist ein Mörtel aus Löschkalk mit zerkleinertem Ziegelschutt als Puzzolanmaterial gewählt und für die Instandsetzung des Kerkerturms angewandt worden.

Schlüsselwörter:
Ton, Puzzolane, Erhaltung, mittelalterliche Festung, Löschkalk, Kompatibilität

1. Introduction

In order to restore the loss of cohesion or the loss of adhesion, conservation strategies are always the best option in case of minor repairs, filling of lacunae, crack repair, and consolidation of old materials. Nevertheless, if defects are severe, such as the general desegregation or large detachment of materials, it may be necessary to substitute old materials, either partially or completely. In the case of mortars, it is essential to design materials which possess good compatibility with the existing backing (stone, bricks, old mortar) and with the fragments of the old mortar, and that are durable enough to resist environmental conditions [1]. The technique used for the application/placing of newly developed mortars, as well as curing conditions, are also significant for the overall performance of the structure that is being preserved.

The basic elements of this work are the principles of authenticity (examination and understanding of the composition and characteristics of old historical mortars) and compatibility (development of mortars with similar characteristics), as well as the fact that there has been a lack of adequate commercial products on the market. The main subject of investigations and conservation efforts presented in this work is the Dungeon Tower of the Bač Fortress. The starting point of the work is related to the interior design of the tower. The rehabilitation principles of the monument had to be respected because the monument is to become a tourist and educational centre with an exhibition. The original mortar in the tower was preserved in small fragments and it constituted the basis for laboratory testing. The aim of these investigations was to develop new compatible materials with the following characteristics: aesthetic appearance and colour meeting high criteria, suitable plasticity that would enable the use of traditional methods of preparation and placing, as well as adequate mechanical properties that would make the material resistant to wear caused by numerous visitors, and relatively stable in existing microclimatic conditions.

1.1. The Bač Fortress – values and history of conservation efforts

The Bač Fortress is classified as a *fortress on water*, with the defence system adapted to marshy land. It consists of a fortified castle and suburb located on a meander of the river Mostonga (the Danube tributary). The fortified complex is situated in the vicinity of the town of Bač, on its west side, Figure 1. The place where the fortress is located is a significant archaeological site populated uninterruptedly since the late Neolithic period. The first building phase dates back to the period between 1338 -1342, and the Hungarian King Charles Robert of Anjou. Intensive building activities started in the mid 15th century, when new warfare techniques were adopted, and the southern boundary was strengthened against Turkish incursions. However, the Turks did conquer the fortress in 1529, and they used it until the year of liberation, 1686. During the Rakoczy uprising (1704), the fortress was devastated, never to be restored again. Although its physical integrity has substantially been lost, the preserved elements point to a sophisticated architecture of the fortification school of High Gothic style, with some elements of the early Italian Renaissance.

The value and significance of the Bač Fortress was fully recognised in modern times, and it was listed as a protected monument in 1948. This meant the end of its further decay, and plunder of its material [3]. The reconstruction of the fortified castle remains began in 1960 with the works on the Dungeon Tower, the best preserved structure in the fortress. The entire perimeter of the final upper part of the tower was missing, and the reconstruction work was determined by the existing data, respecting the original design [4]. The rehabilitation was interrupted in 1962, before the interior of

Figure 1. Panoramic view of Bač with the fortress at the left side and the Dungeon Tower marked with a circle [2]

the tower was finished, but the work was resumed in 2003. The conservation-restoration work including five floors, Figure 2., was undertaken with highest urgency. Due to the absence of architectural data, characteristics of materials were particularly important. The final decision considering the composition of these materials for the floors and walls was made based on the results of multidisciplinary work.

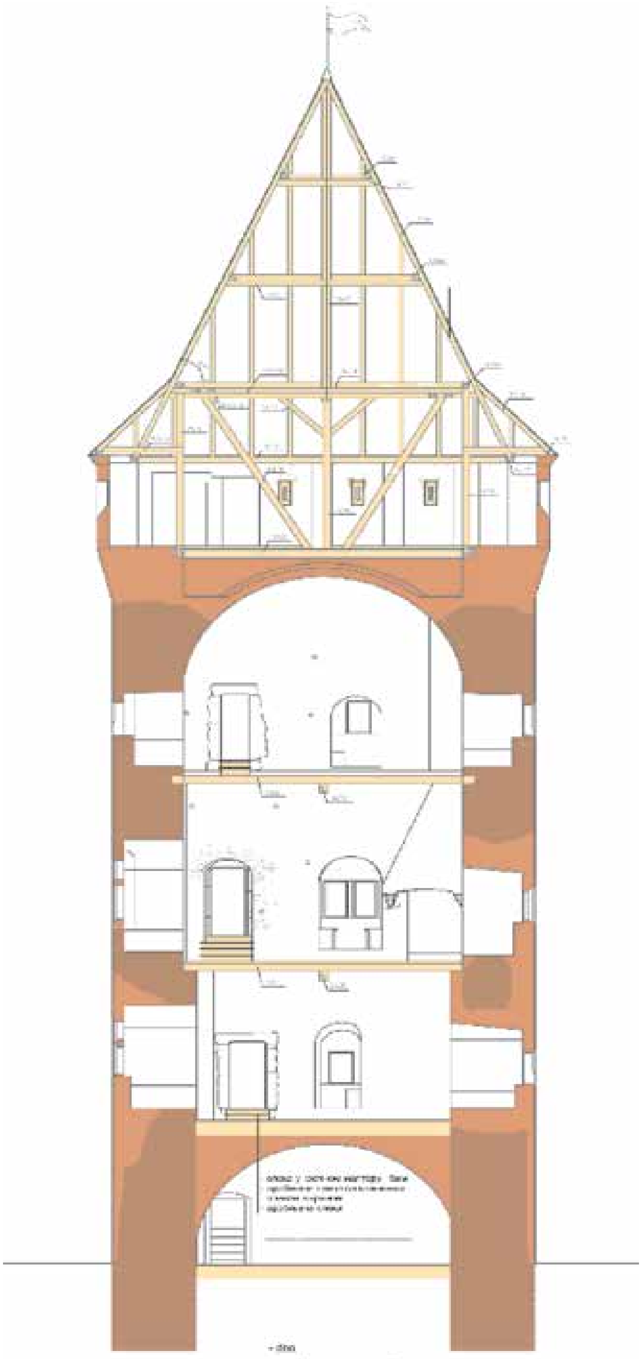

Figure 2. Dungeon Tower, conservatory intervention - cross section [4] (Light areas - 2003 - 2007., middle light areas - 1960-1962. and dark areas - authentic structures)

1.2. Methodology

In order to obtain results that could lead to a qualitative development of newly designed mortars, the investigation work was concentrated on four aspects: laboratory investigation of old mortars from floors of the Dungeon Tower, development of new mortars in laboratory conditions, *in situ* application of developed mortars with a sustainable conservation procedure, and examination of the applied mortars after a determined period of time (4 years), Figure 3. This methodology was set up by a multidisciplinary research team consisting of archaeologists, architects and civil engineers from the Regional Institute for Protection of Cultural Monuments, Petrovaradin, and researchers specializing in the materials science and microbiology from the Faculty of Technology, University of Novi Sad.

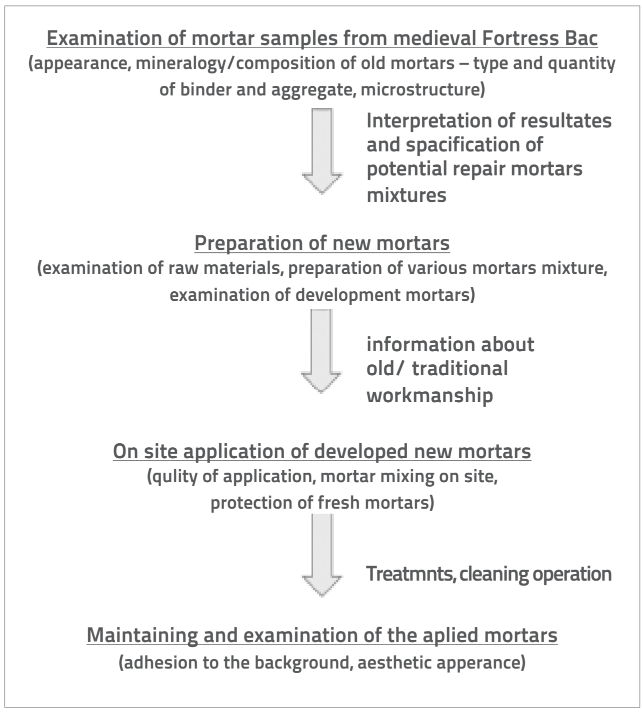

Figure 3. Schematic view of methodology adopted for newly developed mortars

Methods and techniques

Basic microstructural features and mineralogical and chemical compositions of historical and newly developed mortar samples were determined by X-Ray Diffraction, Optical and Scanning Electron microscopy (SEM), Differential Thermal Analysis (DTA), and Differential Scanning Calorimetry (DSC). These techniques are widely applied for the study of mineral phases and chemical composition of ceramic and mortar samples [5].

The mineralogy of the examined materials (historical mortar and raw materials) was determined by **X – ray** diffraction (XRD) with a Diffractometer PW 1050, PHILIPS, CuKα radiation source. The morphology and chemical composition was studied by means of the scanning electron microscopy. The SEM observation of the studied samples was made with an Electron microscope JSM – 6460LV, JEOL. The examined samples were sputter-coated with gold, using the BAL – TEC SCD 005 instrument (180 s/30mA/50mm distance). The thermal analysis (DTA with TG and DSC) was used to obtain a more detailed information about the mineral composition of pozzolanic materials contained in ancient mortars. DTA were recorded in the static atmospheric air, with a thermal analyzer STA 503, BAHR in range from 20 to up to 1000 °C, with the heating rate of 10 °C / min. The samples were packed in alumina crucibles. The referent material was an empty alumina crucible. The DSC was carried out using the Scanning Calorimeter Q20 V23. 10 Build 79, NY (USA), from ambient temperature up to 500°C, with the heating rate of 2 °C/min.

The pozzolanic activity of materials selected for development of the newly designed mortars was determined directly by measuring the calcium ions consumption (volumetric method), and indirectly by the determination of electrical conductivity. The examination of characteristics of mortar applied in the Dungeon Tower was done by means of the Portable Optical Microscopy.

The volumetric method for assessment of pozzolanic activity was conducted by measuring consumption of Ca^{2+} ions in the saturated lime solution (in contact with pozzolanic materials) prepared at room temperature, at time periods determined in advance. The lime solution was filtered and analysed by a titration procedure with the complexometric EDTA and murexide indicator. This indicator was used because of its ability to change colour from pink to purple. The consumption of Ca^{2+} ions of the investigated pozzolanic materials could be directly connected with the pozzolanic activity of the examined materials (removal of Ca^{2+} from solution could be attributed to pozzolanic activity) [6].

The pozzolanic activity examination via electrical conductivity measurements was performed by using the Conductance meter MA 5962. It is known that the degree of pozzolanic reaction is proportional to the reduction of electrical conductivity. The loss of electrical conductivity presents the difference between the initial and the final value of the electrical conductivity of the test suspension (saturated solution of $Ca(OH)_2$ with the presence of the examined pozzolanic materials). It represents the degree of chemical reaction between pozzolanic materials and $Ca(OH)_2$ available in the test solution. Mechanical properties of the newly developed mortars, such as flexural strength, were determined on prismatic test specimens 10 mm x 10 mm x 60 mm in size, at 3, 7, and 28 days. Prepared mortar sample specimens were tested with the Toyoseiki, AT-L-118B instrument, Tokyo, Japan.

2. Experimental

2.1. Old mortars

The sampling of the old mortar from the floors (finishing layer) of the Dungeon Tower was carried out taking into account minor damage in the sampling area. The quantity of sample specimens enabled the planned analysis: determination of the binder/aggregate ratio, and microstructure and structure analysis (Optical and Scanning electron microscopy, XRD). Obtained results, which constitute the basis for development of new mortar compositions, are presented in Table 1 and Figure 4.

Table 1. Characteristics of old mortars [7]

Sample	TB M1: Floor mortar- finishing layer
Picture	
Overview of the analysis	X-Ray Diffraction, Optical and Scanning Electron Microscopy, Differential Thermal Analysis and Differential Scanning Calorimetry
Main characteristics of the examined historical mortar samples - finishing mortar	System based on quartz, clay minerals (chlorite), feldspar, mica, carbonates (calcite, dolomite), with brick fragments about 1 mm in size; SEM analysis indicated the presence of mica, Figure 4; lime/aggregate mass. ratio = 1/3 (sieving procedures)

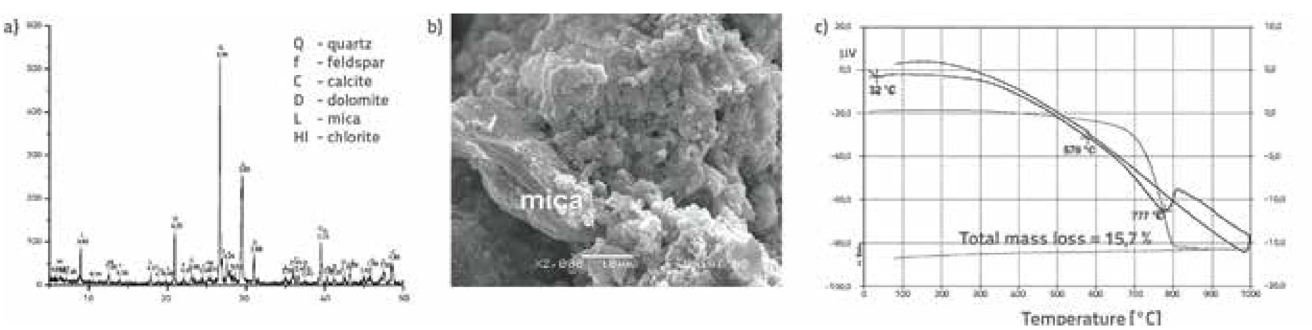

Figure 4. Results of historical mortar sample analysis: a) X-Ray Diffraction; b) Scanning Electron Microscopy; c) Differential Thermal and Thermo-Gravimetric Analysis [7]

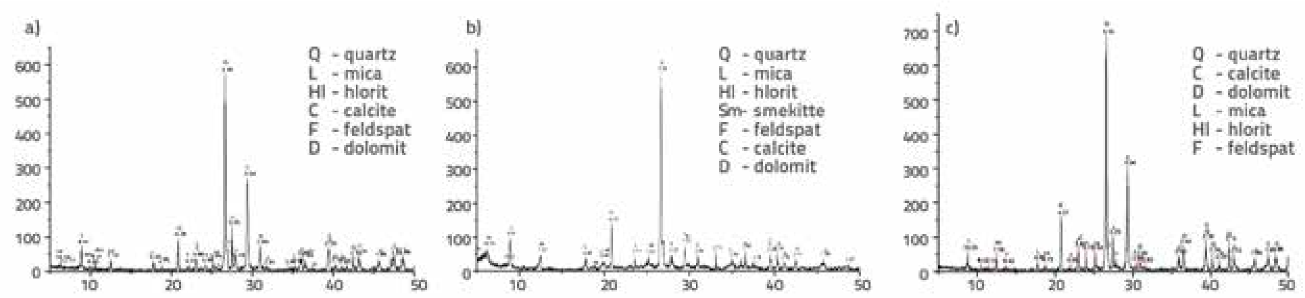

Figure 5. XRD analysis: a) local clay (used for the production of Pozz1 and Pozz2); b) crushed old brick (Pozz3); c) crushed brick fragments from the historic mortar sample TBM1

2.2. New mortars, composition and analysis in laboratory conditions

An important starting point for the design of repair/ replacement mortars was the characterization of the old mortar and its historical context [7]. Based on results obtained by examination of the historical mortar TB M1 sample, and taking into account the fact that the lime mortar was the most widely used material in historical-artistic constructions, the newly designed mortars (DM1, DM2 and DM3) are lime - based materials [8]. These samples, Table 2., were prepared by mixing the aggregate (local sand) and a commercial hydrated lime with potentially pozzolanic materials (crushed old bricks and local fired clays), Figure 5.

Table 2. Mass ratio of binder, aggregate and pozzoolans of the newly designed mortars DM1-DM3

Mortar samples – DM	binder/aggregate/ pozzolan mass. ratio
DM1 (based on pozzolanic material Pozz1	1/3/1
DM2 (based on pozzolanic material Pozz2	1/3/1.5
DM3 (based on pozzolanic material Pozz3	1/3/3

2.3. Analysis of pozzolanic materials

Two representatives of the newly formed pozzolanic materials (Pozz1 and Pozz2) were produced by calcination of a local clay. The pozzolanic material Pozz3 was a crushed brick material (waste material) that does not meet criteria for a construction material. For the production of pozzolanic materials (Pozz1 and Pozz2), the calcination temperature of clay material was determined based on the structure of historic brick fragments included in the composition of the old mortar, and based on mineralogical composition of the local clay, Figure 5. The presence of minerals such as mica, chlorite, and smectite, and the amount of calcite and dolomite, Figure 5a., represented the first indicator that the calcination temperature of the old crushed brick was around 800 °C. The solidification process of the partially formed liquid phase enabled formation of the material with good pozzolanic characteristics [9]. The thermal treatment of the future pozzolanic materials, Pozz1 and Pozz2, was defined based on this fact, and mineralogical properties of the local clay. The calcination was conducted in laboratory conditions (heating rate 10 °C/h; 2.5h at Tmax 960 °C), but different procedures were used for production of each of the two pozzolanic materials. The clay for the production of the pozzolanic material Pozz1 was first dried and then ground and calcined, while in the case of the pozzolanic material Pozz2, the clay was first dried, and then calcined and ground. Even the materials that are commonly not regarded as having good pozzolanic activity can have a good reactivity with

Table 3. Electrical conductivity [msec/cm^3] of the lime-water solution after addition of pozzolanic materials

Time	Pozz1*	Pozz2*	Pozz3*	Pure lime-water solution	Referent lime-solution with quartz sand
0 min.	38	38	38	38	38
3 min.	30	36	36	38	37
15 min.	29	32	32	38	37
45 min.	27	30	32	38	37
90 min.	27	28	30	38	37
7 days	26	27	27	38	37
Δ (7 days)	12	11	11	0	1
* with lime-water solution					

Ca^{+2} ions after the grinding process. This is due to the fact that they possess a large specific surface area that is needed for such reactions [10]. In the case of the studied materials, the mentioned production procedures (drying/calcination/grinding), were set up in order to estimate the impact of the grinding process on pozzolanic activity of the obtained materials.

Electrical conductivity of the lime-water systems containing pozzolanic materials (Pozz1, Pozz2 and Pozz3) was measured in defined periods of time. The corresponding results are shown in Table 3.

By comparing the results of the analyzed materials (Pozz1, Pozz2 and Pozz3) with the reference solution, it can be concluded that all of the examined samples are pozzolanic active materials, Table 3. In the first minutes, the most reactive system was the pozzolanic material Pozz 1, while after 7 days, all systems were more or less equally reactive. Taking into account that the materials Pozz1 and Pozz2 were treated at the same calcination temperature (Tmax 960 °C), the increased reaction with calcium ions in the case of Pozz1, in the first minutes of the reaction, was the consequence of the grinding procedure [6].

Use of volumetric method in pozzolanic activity assessment. The consumption of Ca^{2+} ions by the investigated pozzolanic materials is shown in Table 4.

Table 4. Results of volumetric method for the pozzolanic activity assessment [Δm (Ca^{2+}) (g/10ml)*10^{-4}]

Time	Pozz1	Pozz2	Pozz3
5 min.	0.06675	0.18250	/
30 min.	0.36675	0.18250	6.467
60 min.	0.63332	0.40000	6.733
24 h	0.80000	0.42670	6.867

The results presented in Table 4 show that the pozzolanic material Pozz3 has the highest consumption of calcium ions in the saturated solution of calcium hydroxide. Such high values are the consequence of the hydration process and the binding of calcium ions by the pozzolanic active material. The pozzolanic materials Pozz1 and Pozz2 could be classified as the materials with lower pozzolanic activity than the pozzolanic material Pozz3.

Mechanical characteristics of the prepared mortar samples

The mortar samples DM1, DM2 and DM3 were prepared by mixing the binder, aggregate and pozzolanic material in the ratios given in Table 2. After the moulding procedure, the moulds with specimens were placed in a moist room (90 ± 1% relative humidity and 20 ± 1 °C) [11]. After 24 h they were removed from the moulds and cured for 3, 7 and 28 days, in the above specified conditions. Their flexural strength values were determined after the curing. Flexural strength results (Table 5.) showed that flexural strength values of samples containing crushed old brick (Pozz3) are higher when compared to the thermally treated local clay (Pozz1 and Pozz2).

Table 5. Flexural strength results for developed mortar samples DM1, DM2 and DM3 [MPa]

Time	DM1	DM2	DM3
3 days	0.05	0.09	0.15
7 day	0.46	0.28	0.26
28 days	0.73	0.35	0.99

The sample compatibility assessment is one of the fundamental aspects on which the design of a repair mortar should rely on. Regarding the obtained results, the compatibility was checked in the context of joining the old and the new materials, thus enabling their mutual interaction and cohesion.

Based on the carbonization/hydration process, a contact zone was formed between two types of materials, fragments of the old mortar and the newly developed mortar pastes, Figure 6. The morphology of these zones was analyzed with a stereo-optical microscope, Figure 7.

Figure 6. Photographs of the compatibility assessment; old mortar TB M1 (central part of the sample) and new peripheral zones of the developed mortars: a) DM 1; b) DM 2; c) DM3

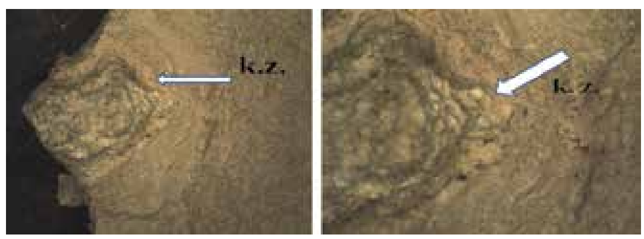

Figure 7. Contact zone *(k.z.) development between the old mortar sample TBM1 and the newly developed mortar DM 3 formed with the pozzolanic material Pozz3

The morphology analysis of the contact zones shows that the formed area possesses a satisfied aesthetic value (without cracks) only in the case of the mortar DM 3, which was produced based on the pozzolanic material Pozz3. Taking into account the composition of this mortar sample (the presence of old crushed brick), the formation of a material with good hydraulic properties could have been expected [12]. These expectations were proven by the obtained results (Tables 3-5) and (Figure 7). Evidently, these characteristics distinguish the mortar sample DM3 from DM1 and DM2 as a potential finishing mortar for in situ application in the area of the Dungeon Tower floor.

2.4. In situ application and selection of final composition of newly developed mortars

Occasionally, laboratory tests do not directly correspond to real on site situations, which can result in various problems. Besides the chemical compatibility of the old and new materials, new materials had to meet numerous requirements with regard to conservation principles. Based on this fact, additional four compositions of mortar samples, M1-M4, were produced, Table 6. One sample represented the basic mortar - M1, while the other three samples (M2, M3 and M4) were floor finishing mortars with similar compositions. The binder/aggregate mass ratio in the prepared sample was 1:3 (as in the case of DM3), except for the mortar M1 where this ratio was 1:5. The binder used for all prepared mortars was a traditional slaked lime, while the origin of the crushed brick was the same as for laboratory samples (grain size under 300 μm), The marble and sifted river sand (from the Danube) were used as aggregates in the composition of the newly designed Ms mortars.

Table 6. Compositions of Ms mortar samples prepared for in situ application

Test samples	M1	M2	M3	M4
Slaked lime	1	1	1	1
Sand	2	1,5	1	1
Crushed brick	-	0,5	0,5	1
Red marble	2	0,5	0,5	0,5
Ground red marble	1	0,5	1	0,5

Besides chemical, esthetical, physical or mechanical characterization that is generally made in the case of the newly developed materials, the conservators are also concerned about workability aspects [13]. The workability of a chemically compatible mortar can not be guaranteed in advance. Therefore, one or more application tests are often decided upon on site. Some in situ tests were performed to study workability and consistency of prepared mortars (Table 6). Prepared mortar samples were placed on glass plates in form of a truncated cone (cookies of about 9 cm in diameter) by manual vibration in order to assess volume consistency [14]. These steps were significant for defining aesthetic, adhesive, and mechanical properties of the newly designed samples M2, M3 and M4, which were to be used as finishing floor mortars. The mortar sample (M1) with the binder/aggregate ratio 1/5 was planned to be a base mortar that would be used together with M2-M4 finishing mortar samples. Some observations considering the differences between the applied mortars M2-M4 were made after 24 hours of drying in the shade, Figure 8. In the case of the mortar sample M2 the surface was cracked and rough with spots. The mortar sample M3 also had a rough surface, but of a lesser intensity and with fewer crumbles. The last sample, the mortar sample M4, did not have visible cracks, and the surface was smooth as an adequate plasticity with the desired tonal effect was achieved.

Figure 8. Testing workability and consistency of prepared mortars

Taking into consideration laboratory results and results obtained by in situ testing of mortar samples, the M4 sample was finally selected for the conservation treatment of the Dungeon Tower floor area. This mortar satisfied numerous required properties such as: good hardening time and adhesion properties, good aesthetic appearance (colour, texture, surface finish), similarity to the authentic finishing mortar, and good workability without any cracks during the hardening process. After the mortar production and selection, the conservation treatment of the floor was performed. The following procedures were applied: the joints were carefully cleaned (by washing with water, and by removal of all loose parts of the bricks and mortars) and the base mortar M4 was applied, Figure 9.

Figure 9. Appearance of the floor area before and after conservation treatment [4]

2.5. In situ examination of applied mortars after conservation treatment

The behaviour of the applied mortar M4 in real environmental conditions was monitored four years after the application. The treated area in the Donjon Tower was inspected in situ using a stereo-optical microscope, Figure 10. This kind of investigation is a suitable method for the detection of cracks or micro cracks that directly determine the durability and aesthetic performance of the applied mortar. The results are shown in Figure 11.

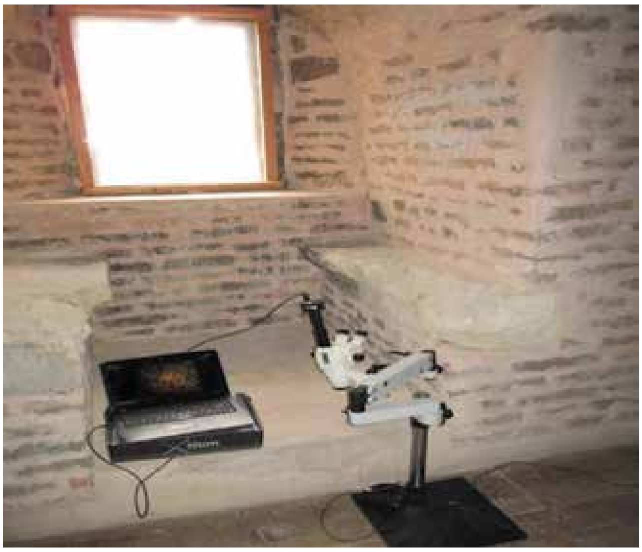

Figure 10. In situ investigation with a stereo-optical microscope

The obtained stereo-optical micrographs, Figure 11, show that the area is a compact zone without any visible cracks or micro cracks. These results suggest that the conservation treatment was successfully completed, and that the applied mortar M4 is characterised by a satisfactory performance, without visible anomalies throughout its exploitation in real-life conditions over the past four years.

Further collaboration between conservators and scientists is expected through mobile training teams that would provide preliminary diagnosis by using non-destructive methods as well as expert advice when needed, in order to prevent inadequate interventions and loss of matter – being an evidence of the authenticity of the tangible heritage [15, 16]. Finally, it should be noted that the authenticity appears as the essential qualifying factor concerning adequacy of conservation treatments. As pointed out in The Nara Document on Authenticity "authenticity judgments may be linked to the worth of a great variety of sources of information. Aspects of these sources may include form and design, materials and substance, use and function, traditions and techniques" [17].

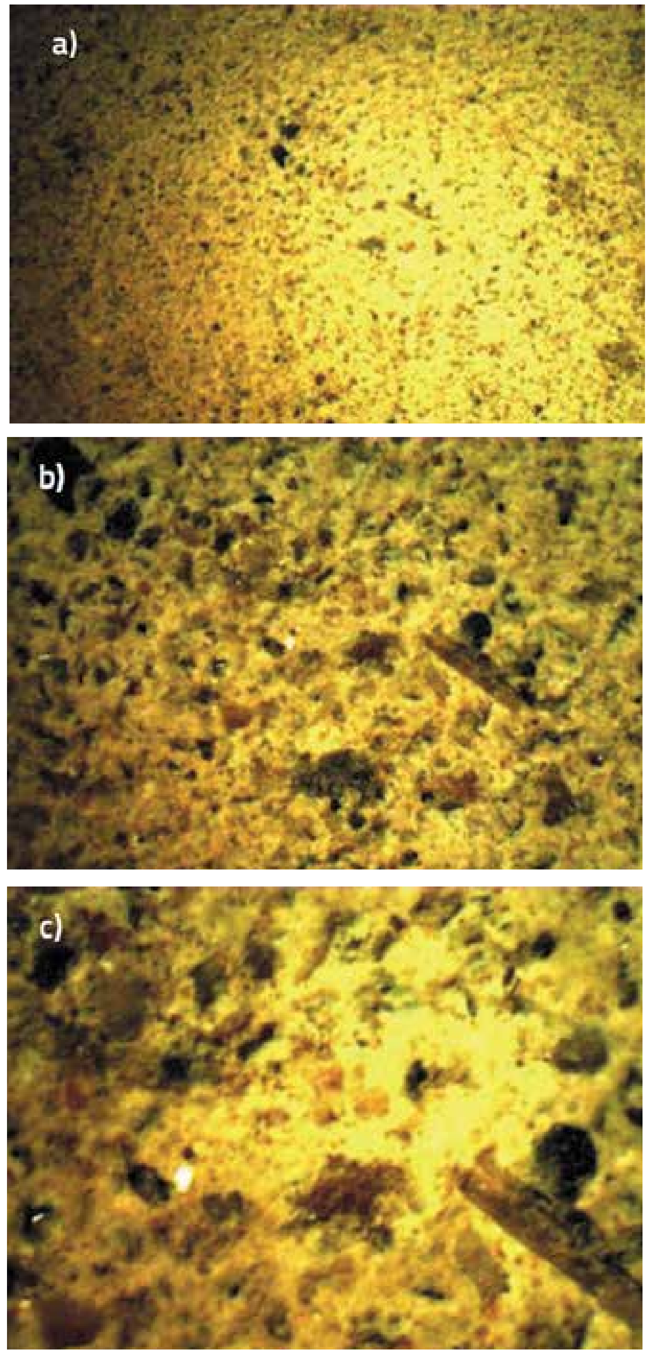

Figure 11. Stereo-optical micrographs of the applied mortar M4 after four years: a) magnification x 6.5; b) magnification x 25; a) magnification x 45

3. Conclusions

The multidisciplinary approach to the assessment of the Dungeon Tower values, as well as the design and application of the newly produced materials, confirmed the efficiency of the well-designed methodology, and the competence of the team work in the field of cultural heritage.

Two materials were tested as potentially pozzolanic materials: local clay and crushed old brick. The results of the performed pozzolanic activity test (volumetric method and electrical conductivity) show that the crushed old brick (Pozz3) can be classified as a material with the highest pozzolanic activity. The mortar DM3 based on the pozzolanic material Pozz3 satisfied the requirements such as good hardening time, adhesion properties, and compatibility with the authentic mortar preserved in small fragments, good aesthetic appearance (colour, texture, and surface finish), and good workability without any cracks during the hardening process. All floor surfaces in the Dungeon Tower were treated with the developed material M4 using the traditional placing technique. Four years after being placed into the floor, the mortar was screened on the site with a stereo-microscope. No cracks or micro cracks were detected. The newly developed mortar was proven to possess suitable mechanical resistance and was relatively stable in the existing microclimatic conditions of the Dungeon Tower.

The approach to solving the conservation problem and a dilemma in this case study belongs to the field of conservation science. The experience acquired through the work of a multidisciplinary team, and the advanced material production techniques used at this and other cultural monuments, have been integrated into broader research activities which have, in the meantime, developed into a notable international collaboration.

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