CHARACTERIZATION OF HISTORICAL INORGANIC BINDERS AND DESIGN OF RESTORATIVE MATERIALS - CASE STUDY OF THE LATE ROMAN MOSAIC

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MULTIDISCIPLINARY RESEARCH GROUP

EXPERTISE

- **Examination** of building materials (historical and modern)
- **Design and processing** of new functional materials for cleaning and protection historical objects
- **Development of new methods** for materials characterization, functionality, compatibility, durability (*in situ* and laboratory)

- conservation of cultural heritage with emphasis on stone and mosaic conservation
- methodologies for the *in situ* conservation of built structures
- study of decay mechanisms of porous building materials, environmental monitoring, condition assessment, past treatments evaluation and planning of future conservation
Challenges: many archaeological sites suffer from inadequate intervention actions and practices, poor selection of materials and wrong policy decisions, or all of them together

Design of restorative mortars for historical buildings and artefacts is always a challenging task!

Restorative mortars need to comply well with chemical, mineralogical and mechanical compatibility criteria

One of success stories is the restoration of Late Roman mosaic discovered broken in 2014, with severely disturbed positioning of fragments.
AIM AND INTRODUCTION

The objective of the present project was to preserve remains of the original bedding layer and to connect and stabilize groups of individual fragments using compatible lightweight restoration mortar.

Characterization of historic mortar samples gave information about the composition of mosaic bedding layers and their preparation, as well as degradation mechanisms.

POSSIBLE SOLUTION

Design lightweight mortar respecting compatibility criteria: chemical and mineralogical composition, porosity and visual properties (colorimetric parameters), mechanical properties and formation of contact zone between original and restoration mortar.
LOCATION AND ARCHAEOLOGICAL CONTEXT

- Archaeological site “Zadružni dom” is located in village Skelani on the left bank of Drina river – on the border between Serbia and Republic of Srbska (BH).
- It was centre of municipium Malvesiatium - important administrative center of the Roman Empire II-IV c. AD.
- It was first discovered 120 years ago by Carl Patsch, Austro-Hungarian historian.
- First systematic archaeological excavation started in 2008 with extraordinary findings, including the late Roman building with hypocaust heating system and mosaic floors.
MEDUSA MOSAIC FROM SITE “ZADRUŽNI DOM”

- In one of the rooms of a late antic building, a fragmented mosaic was discovered in 2014.
- It was built over the *hypocaust*.
- Central mosaic representation was the head of *Medusa*.
Causes of mosaic deterioration:

- **building technique**
  (mosaic was built over brick pillars of the *hypocaust* system)

- **destruction of the building**

- **burial conditions**
  (flooding and soil movement due to proximity of the river)
STRATIGRAPHY OF MOSAIC ORIGINAL PREPARATION LAYERS

- **tessellatum** – mosaic tesserae placed in the fresh lime mortar
- **nucleus** – lime mortar with *cocciopesto* (crushed brick)
- **rudus** – coarser mortar with gravel and brick fragments
MOSAIC CONSERVATION

In the first phase mosaic conservation comprised of:

- Cleaning mosaic fragments
- Triage and reconstruction of mosaic decorative scheme
- Connecting fragments and positioning them in the sand basin

Final goal was to restore mosaic with the **compatible restoration mortar** and to **expose it on the site** under the shelter.
RECONSTRUCTION OF MOSAIC DECORATION

Central mosaic carpet
RECONSTRUCTION OF MOSAIC DECORATION

Borders
REQUESTS FOR THE RESTORATION MORTAR

- Keeping original nucleus on mosaic fragments
- Compatible with original *nucleus*
- Eco and health friendly
- Low cost
- To allow on site presentation of the mosaic (under the shelter)
RESEARCH APPROACH AND METHODS

RESEARCH APPROACH

1. Characterization of the original materials
2. Design of compatible laboratory models
3. Aging of laboratory models and original materials in weathering chamber
4. Characterization of aged samples
5. Decision about optimal mixture of compatible lightweight restoration mortar

NON-DESTRUCTIVE METHODS

- Stereo optic microscopy
- X-ray Fluorescence Analysis (XRF)
- Fourier transform Infrared spectroscopy (FTIR)

LABORATORY ANALYSIS

- Structural analysis (XRD)
- Chemical analysis
- Textural properties (Hg porosimetry)
- Scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS)
MICROSCOPY RESULTS
STRUCTURAL ANALYSIS - XRD

BRICK

STONE AGREGATE

BINDER
TEXTURAL PROPERTIES – Hg porosimetry

Pore size radius [μm]

Pore volume fraction (%)
SCANNING ELECTRON MICROSCOPY

<table>
<thead>
<tr>
<th>Element</th>
<th>C</th>
<th>O</th>
<th>Al</th>
<th>Si</th>
<th>Ca</th>
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<tr>
<td>Spektar 1</td>
<td>40.91</td>
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<tr>
<th>Element</th>
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<th>Si</th>
<th>Ca</th>
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<tr>
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## COMPATIBILITY CRITERIA

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Compatibility indicators</th>
<th>Incompatibility risks</th>
<th>Scale of incompatibility risk assessment</th>
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<tbody>
<tr>
<td><strong>AESTHETIC COMPATIBILITY</strong></td>
<td>Total colour change $\Delta E$</td>
<td>$\Delta E&lt;3$</td>
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<tr>
<td></td>
<td></td>
<td>$3&lt;\Delta E&lt;5$</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\Delta E&gt;5$</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>Appearance of cracks</td>
<td>No cracks</td>
<td>0</td>
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<tr>
<td></td>
<td></td>
<td>Visible cracks</td>
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<tr>
<td><strong>CONTACT ZONE</strong></td>
<td>Macroscopic and microscopic testing: before and after aging</td>
<td>Contact zone is formed</td>
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<tr>
<td></td>
<td></td>
<td>Contact zone is partially formed</td>
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<tr>
<td></td>
<td></td>
<td>Visible cracks in contact zone</td>
<td>10</td>
</tr>
<tr>
<td><strong>NATURE OF AGGREGATES AND BINDERS</strong></td>
<td>Aggregate nature</td>
<td>Similar</td>
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<tr>
<td></td>
<td></td>
<td>Different</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>Binder nature</td>
<td>Similar</td>
<td>0</td>
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<tr>
<td></td>
<td></td>
<td>Different</td>
<td>10</td>
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<td><strong>MECHANICAL CHARACTERISTICS</strong></td>
<td>Bending and compressive strength</td>
<td>Lower value of new mortar</td>
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<td></td>
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<td>Similar values</td>
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<td>Higher values of new mortar</td>
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<td>$\text{(depends on function)}$</td>
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LABORATORY SAMPLES DESIGN

New mortar with a small pieces of ancient mortar imbedded

Model mortars laboratory preparation
LABORATORY MODELS
WITH ANCIENT MORTAR SAMPLE

ANCIENT MORTAR
LABORATORY AGING - ARTIFICIAL WEATHERING

Aging conditions

• Room conditions
  \((T = 20^\circ\text{C} \text{ i } RH = 60\%); 7\text{ days}\)

• Extreme conditions:
  – \((T = 10^\circ\text{C} \text{ i } RH = 80\%); 3\text{ h}\)
  – \((T = 35^\circ\text{C} \text{ i } RH = 35\%); 3\text{ h}\)
  – \((T = 45^\circ\text{C} \text{ i } RH = 35\%); 2\text{ h}\)
    ▪ 5 cycles repeated

Laboratory aging chamber
Binder Climate chamber KBWF 240
## TOTAL COLOUR CHANGE

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<th>Sample</th>
<th>In point</th>
<th>Average</th>
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<tbody>
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<td>2</td>
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<td>2/2</td>
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<td>9.88</td>
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<tr>
<td>3</td>
<td>8.99</td>
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<td>4</td>
<td>8.34</td>
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<td>5</td>
<td>13.4</td>
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<tr>
<td>13/2</td>
<td>11.31</td>
<td>8.55</td>
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</table>
MINERALOGICAL COMPATIBILITY AND MECHANICAL CHARACTERISTICS

Almost identical phase composition

<table>
<thead>
<tr>
<th>Sample</th>
<th>Compressive strength (kPa)</th>
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</thead>
<tbody>
<tr>
<td>Original mortar</td>
<td>1215</td>
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<tr>
<td>Newly-designed 9</td>
<td>274</td>
</tr>
<tr>
<td>Newly-designed 11</td>
<td>486</td>
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</table>
CONTACT ZONE EXAMINATION - MICROSCOPY

Good results of contact zone formation.

Crack inside of new mortar & in the contact zone.
CONCLUSIONS (I)

- Results of the characterization of original materials confirmed composition typical for late Roman period based on lime, broken bricks, clay fractions, sand and marble.

- Lime matrix preserved excellent contact zone between brick and binder phase, indicating that the binder had also pozzolanic properties.

  Morphology of the brick grain is not rounded, revealing that it was prepared by breaking (up to several mm in size) rather than grinding (SEM/EDS results also confirmed that the brick was not added in the form of brick flour).

  Clay fraction also played a role as a pozzolanic material.

  Regarding the ratio of binders and aggregates it was revealed that ratio in volume fractions ranged from 1:3 to 1:4.7

- Based on the characterization results of mortar samples from ancient mosaic, the compatible lightweight restoration mortar recipes were prepared.
CONCLUSIONS (II)

• **Sixteen systems were designed and aged** under laboratory conditions in artificial weathering chamber. The most promising systems were selected based on the compatibility criteria, respecting their future functionality and reversibility.

• **Two systems stood out and fully complied with the set compatibility criteria.** These were designed based on refraction of brick, lime, vermiculite, sand, ground marble and yellow (carbonate) brick clay. Both **met the criteria of aesthetic compatibility and contact zone formation** during the initial carbonation phase and after aging. In addition, both had the **same mineralogical nature of aggregates and binders compared to the ancient mosaic mortar**, with the presence of the same mineral forms.

• Finally, the systems were designed **with targeted weaker mechanical characteristics, which is very important feature in order to satisfy reversibility principles and their future functionality.**
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