University of Novi Sad, Faculty of Technology, *Laboratory for Materials in Cultural Heritage*

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ERE5.1
Constructional GeoMaterials: Resources, Properties, Uses, and Environmental Interactions

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

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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)

MSc Maja Franković

EXPERTISE

- 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

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

- 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

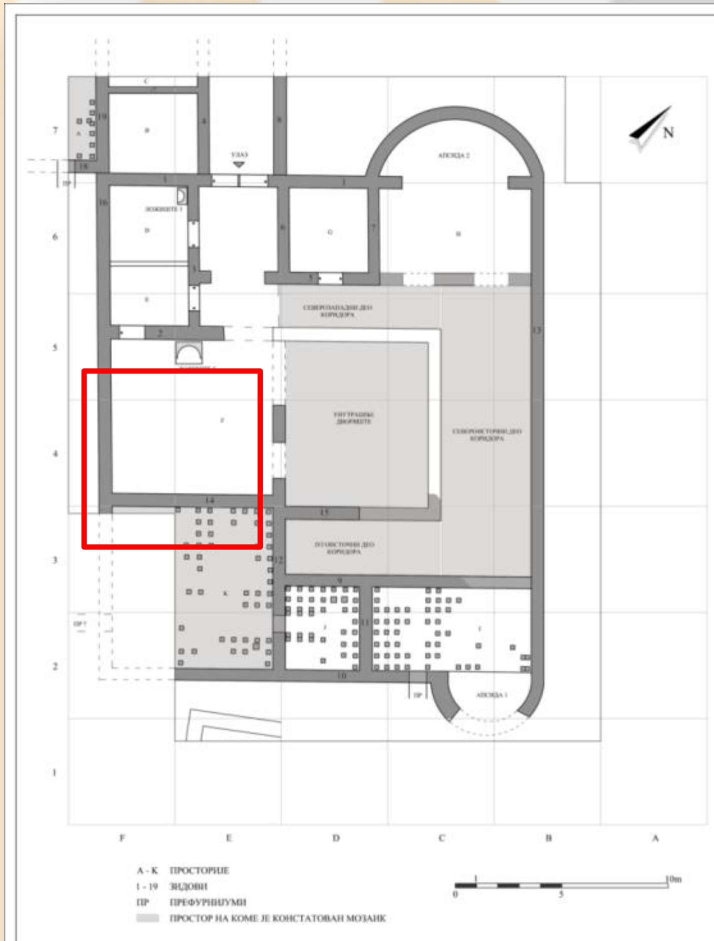
Design lightweight mortar respecting compatibility criteria:
chemical and mineralogical composition,
porosity and visual properties (colorimetric parameters),
mechanical properties and
formation of contact zone between original and restoration mortar

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*



ARCHAEOLOGICAL EXCAVATIONS IN 2014 room K. sq.E

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

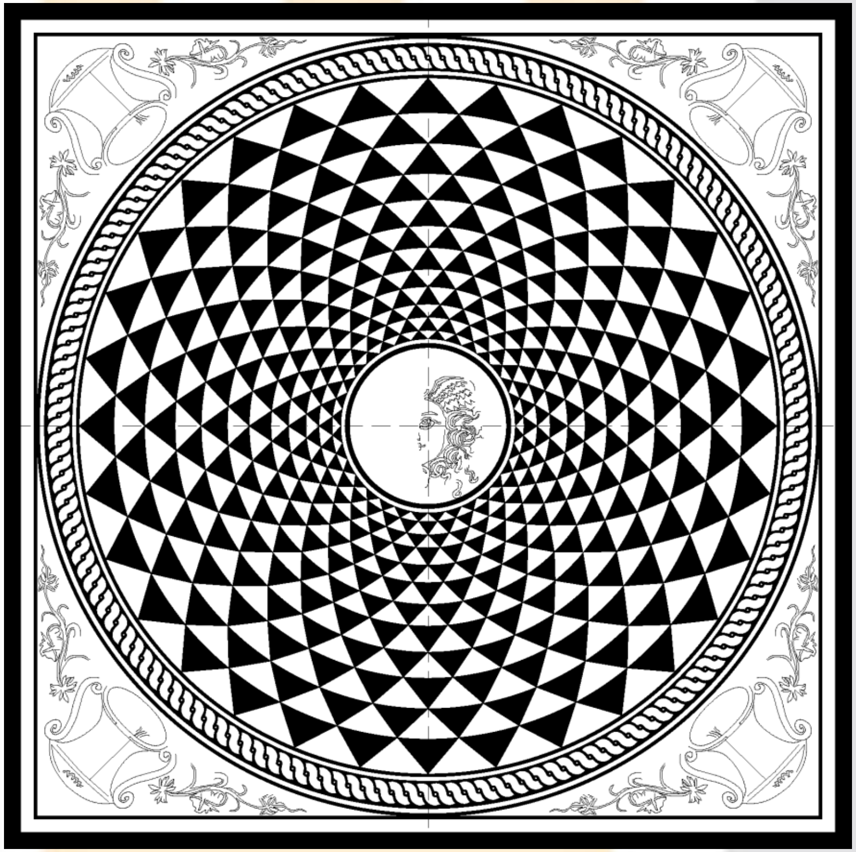
In the first phase mosaic conservation comprised of:

- Cleaning mosaic fragments
- Triage and reconstruction of mosaic decorative sheme
- 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

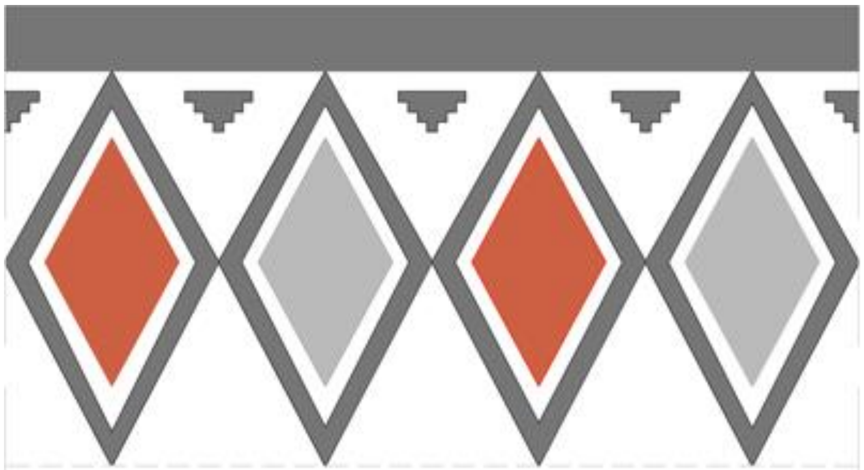
RECONSTRUCTION OF MOSAIC DECORATION

Central mosaic carpet



RECONSTRUCTION OF MOSAIC DECORATION

Borders



I

II

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**

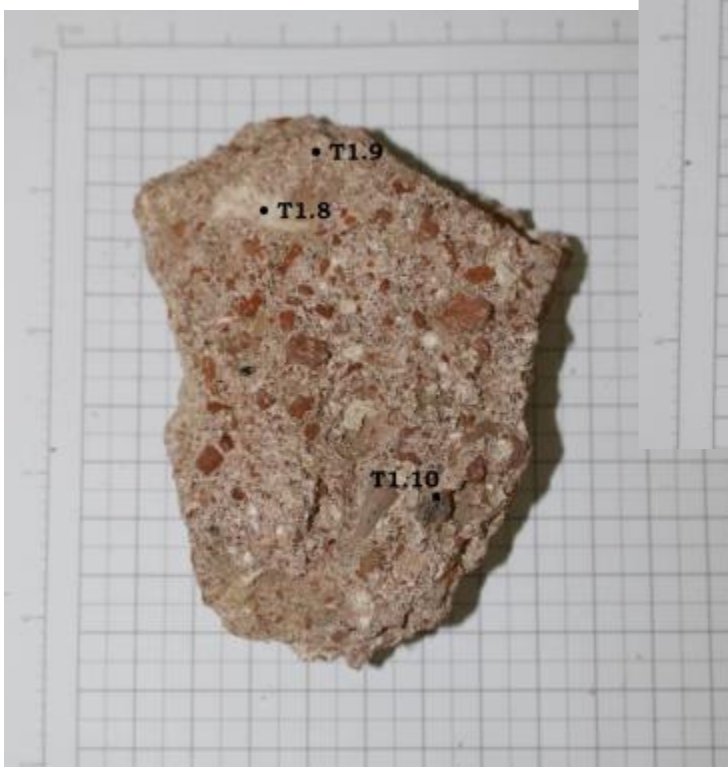
NON-DESTRUCTIVE METHODS

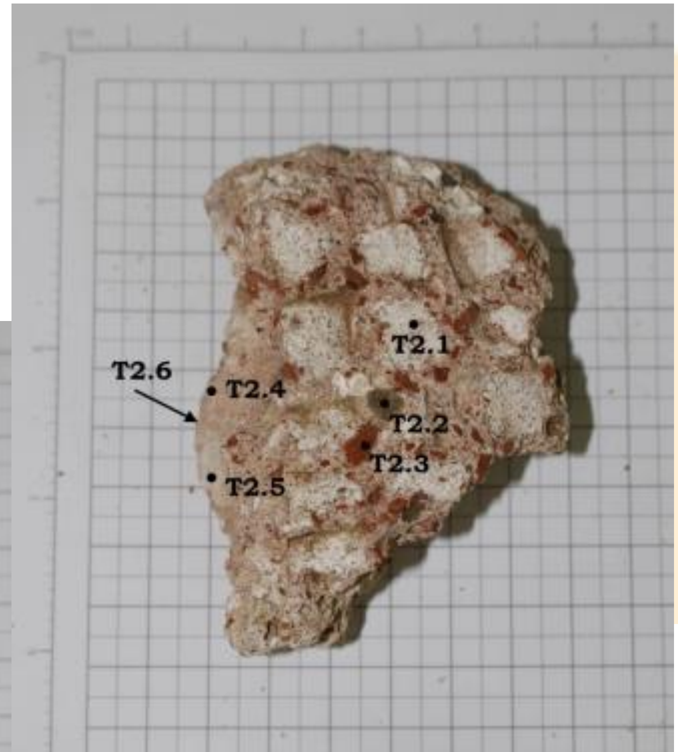
- Stereo optic microscopy
- X-ray Fluorescence Analysis (XRF)
- Fourier transform Infrared spectroscopy (FTIR)

LABORATORY ANALYSIS

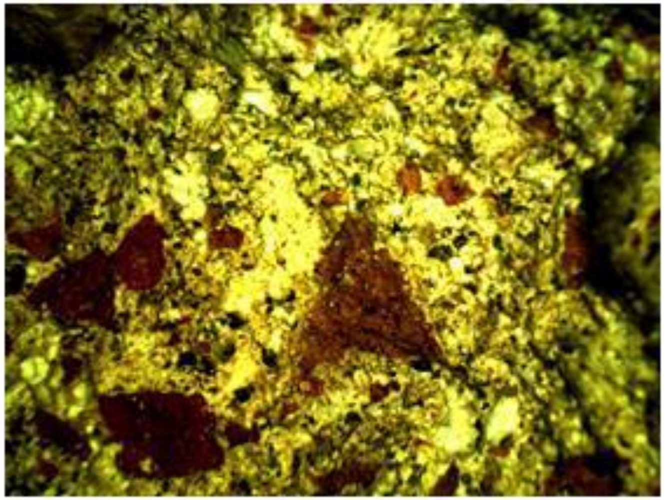
- Structural analysis (XRD)
- Chemical analysis
- Textural properties (Hg porosimetry)
- Scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS)

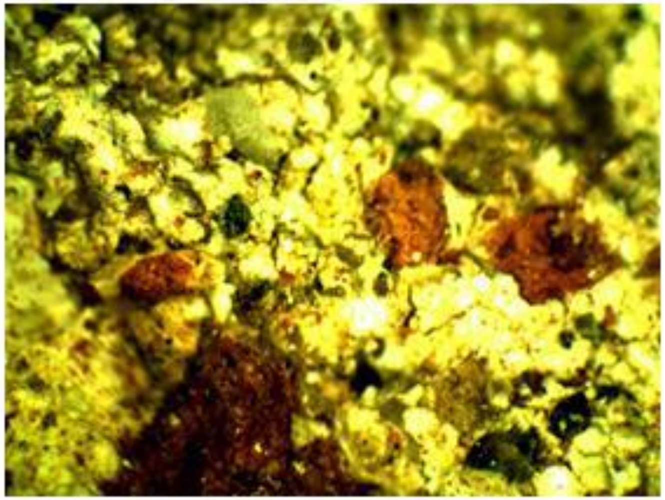
ANCIENT MOSAIC SAMPLES

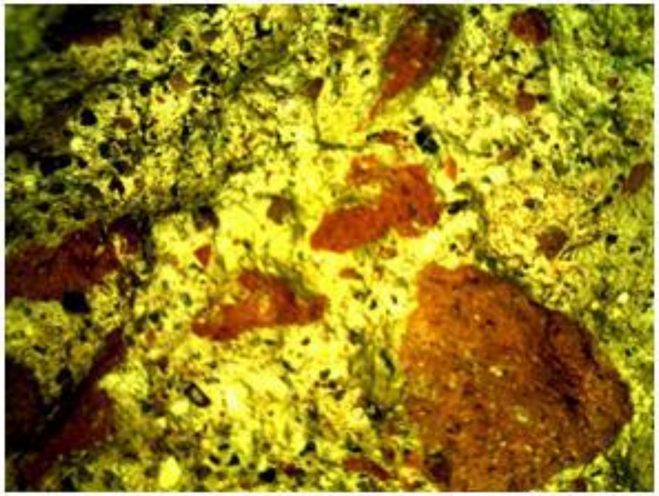


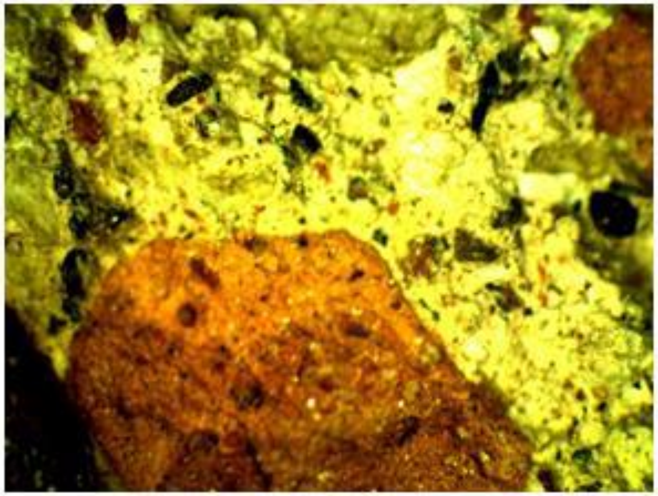


MICROSCOPY RESULTS



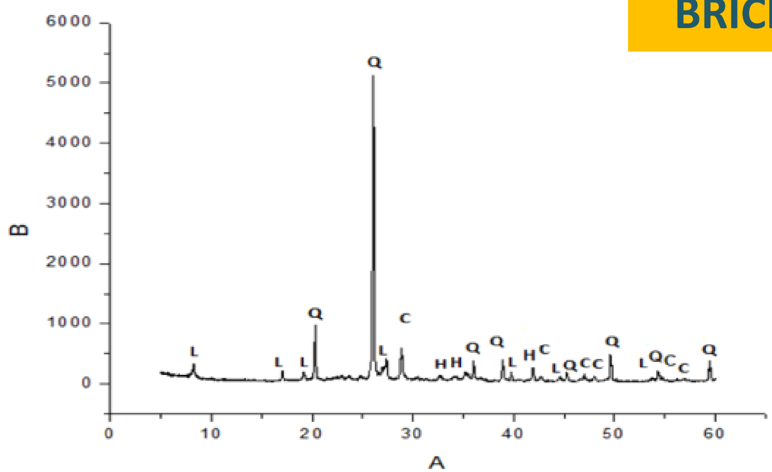




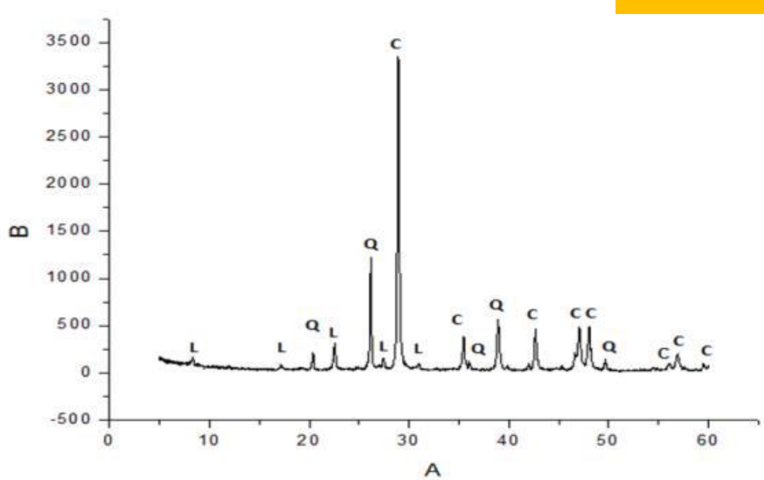


STRUCTURAL ANALYSIS - XRD

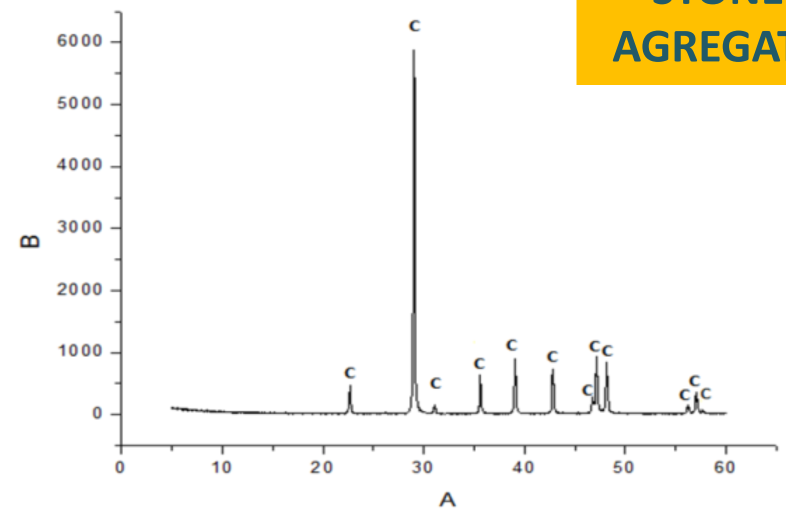
BRICK



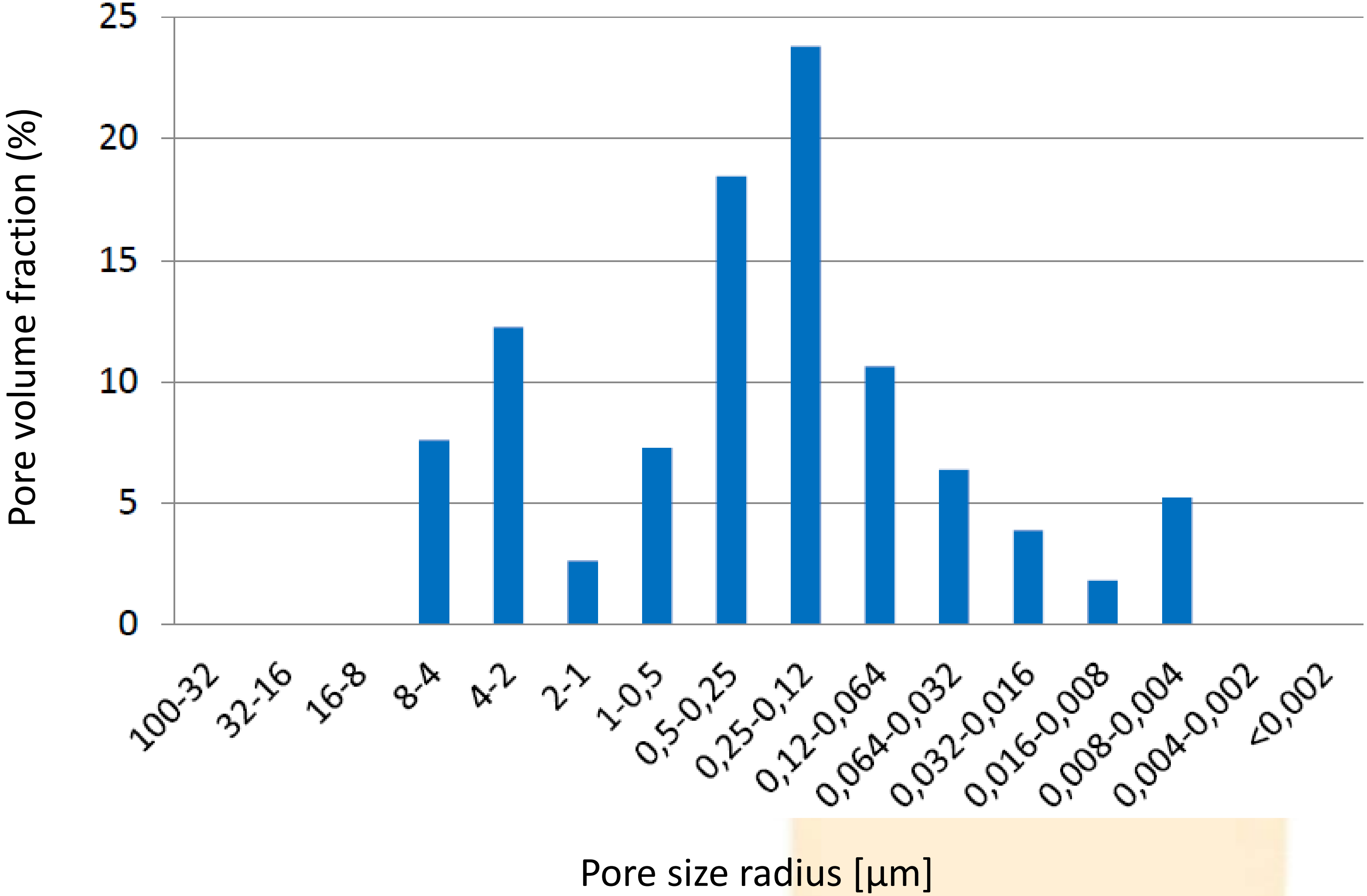
BINDER



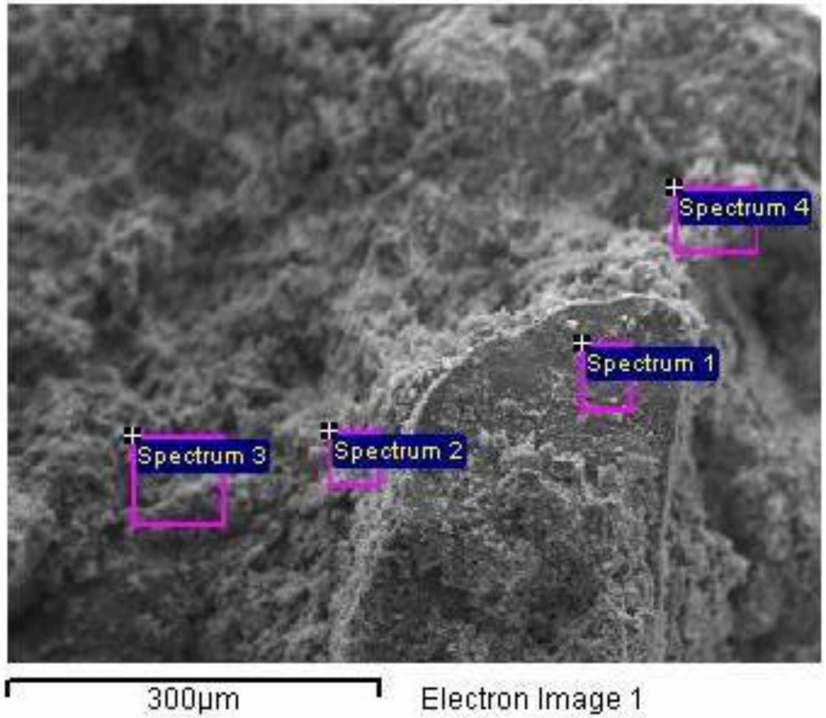
STONE AGREGATE

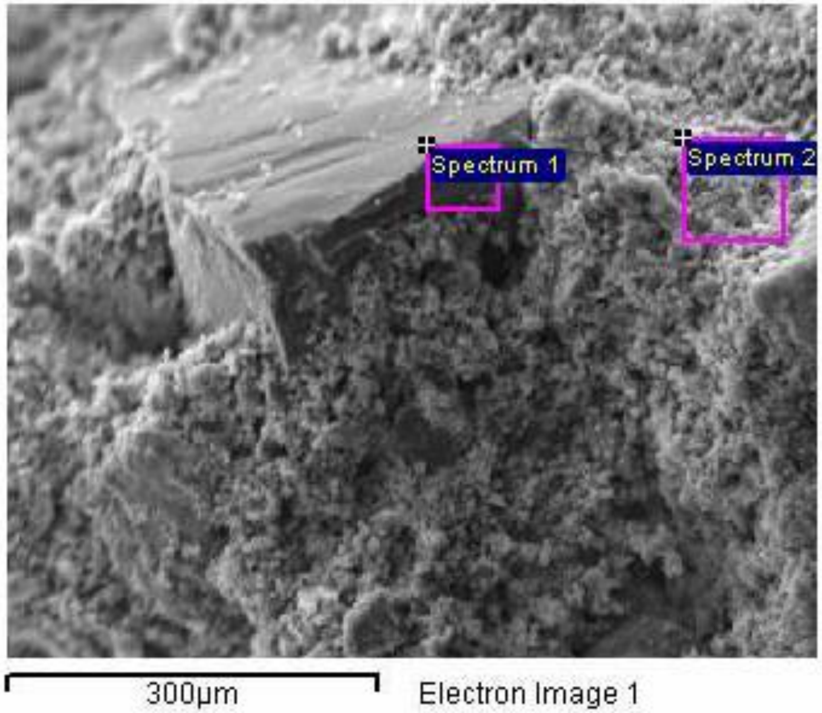


TEXTURAL PROPERTIES – Hg porosimetry



SCANNING ELECTRON MICROSCOPY





Element	**C**	**O**	**Al**	**Si**	**Ca**
Spektar 1	40,91	36,03	0,10	20,13	2,74
Spektar 2	15,94	55,43	0,77	1,96	25,90
Spektar 3	14,59	47,76	1,01	4,23	32,42
Spektar 4	13,85	51,86	1,08	2,90	30,32

Element	**C**	**O**	**Si**	**Ca**
Spektar 1	24,06	42,97	31,61	1,36
Spektar 2	13,36	50,71	0,89	35,04

COMPATIBILITY CRITERIA

Criteria	Compatibility indicators	Incompatibility risks	Scale of incompatibility risk assessment
AESTHETIC COMPATIBILITY	Total colour change ΔE	ΔE<3 3<ΔE<5 ΔE>5	0 5 10
	Appearance of cracks	No cracks Visible cracks	0 10
CONTACT ZONE	Macroscopic and microscopic testing: before and after aging	Contact zone is formed Contact zone is partially formed Visible cracks in contact zone	0 7 10
NATURE OF AGGREGATES AND BINDERS	Aggregate nature	Similar Different	0 5
	Binder nature	Similar Different	0 10
MECHANICAL CHARACTERISTICS	Bending and compressive strength	Lower value of new mortar Similar values Higher values of new mortar	0 0-3 *(depends on function)* 5-10 *(depends on function)*

J. Delgado Rodrigues, A. Grossi, Indicators and ratings for compatibility assessment of conservation actions. Journal of Cultural Heritage, 8(2007), pp. 32-43

LABORATORY SAMPLES DESIGN

Model mortars laboratory preparation

New mortar
with a small pieces
of ancient mortar
imbedded

LABORATORY MODELS
WITH ANCIENT MORTAR SAMPLE

LABORATORY AGING - ARTIFICIAL WEATHERING

Laboratory aging chamber
Binder Climate chamber KBWF 240

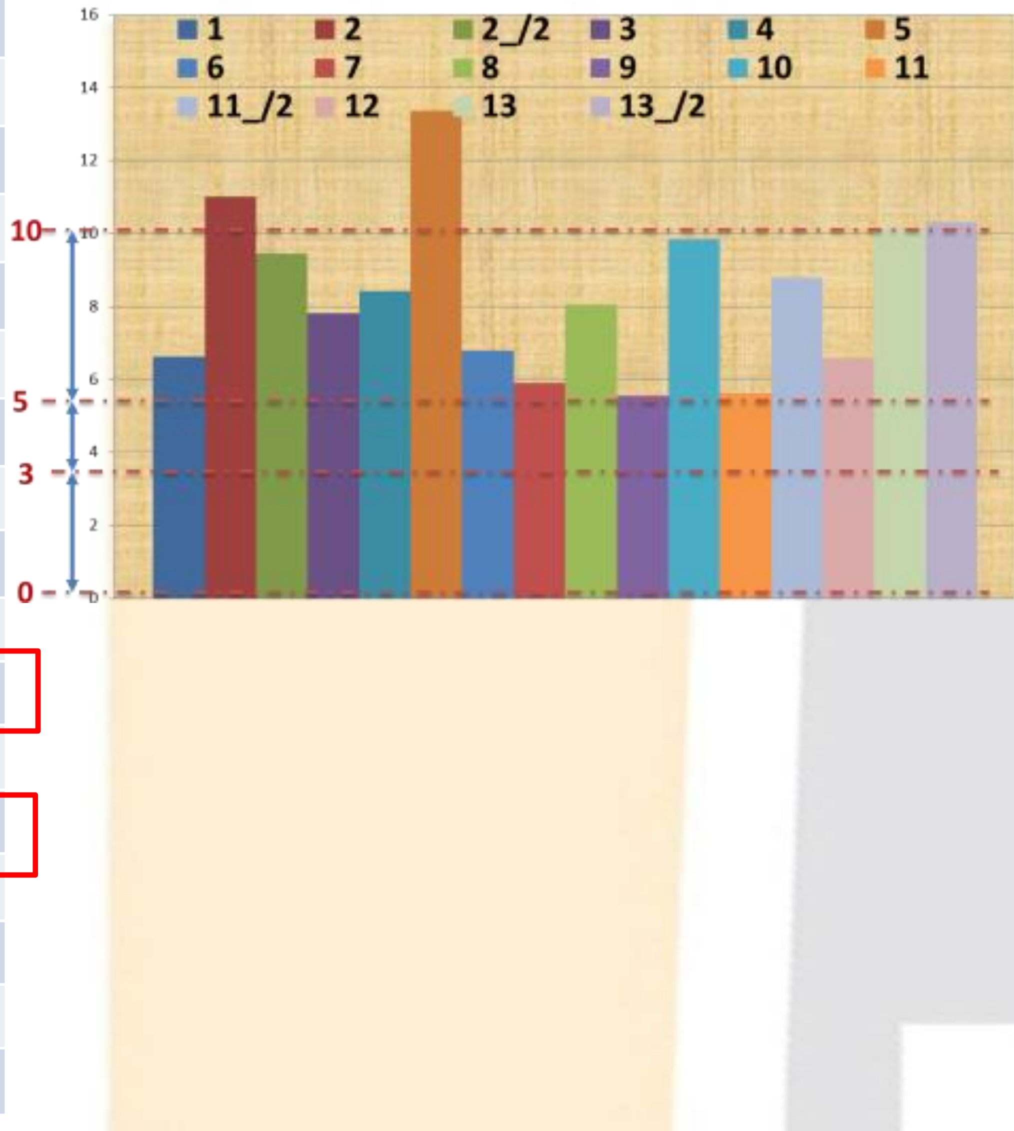
Aging conditions

- Room conditions
  (T = 20˚C i RH = 60%); 7 days

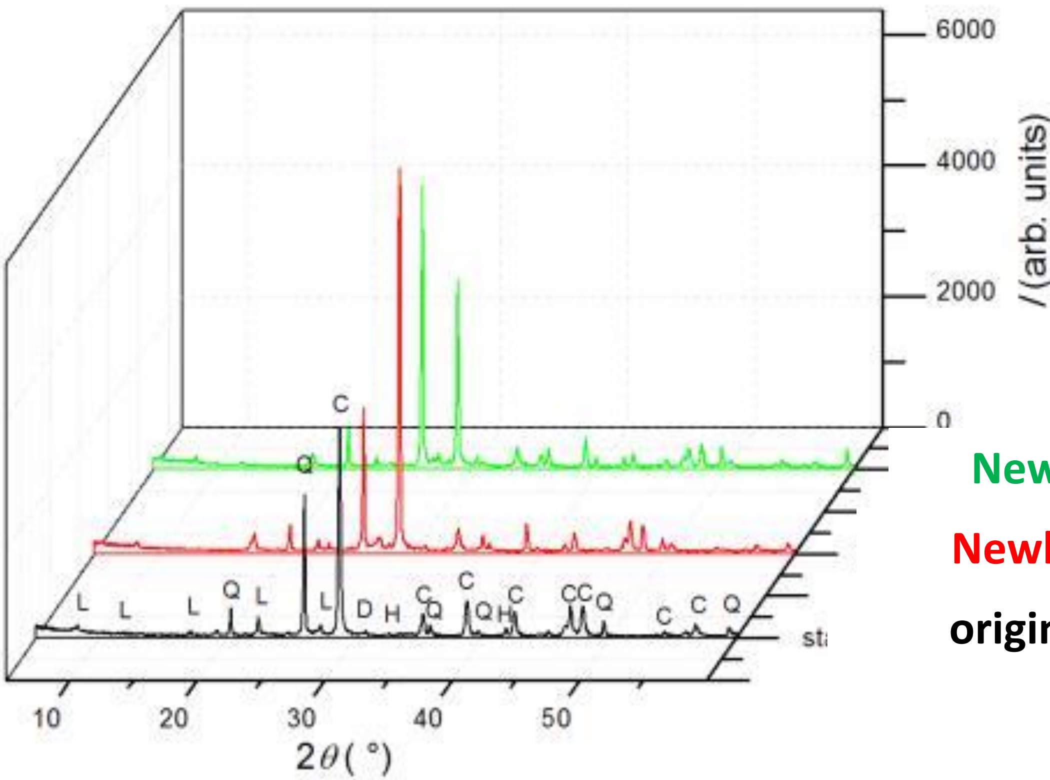
- Extreme conditions:
  - (T = 10˚C i RH = 80%); 3 h
  - (T = 35˚C i RH = 35%); 3 h
  - (T = 45˚C i RH = 35%); 2 h
  - 5 cycles repeated

TOTAL COLOUR CHANGE

Sample	(ΔE)			
	In point			Average
1	6,1	6,97	6,86	6,64
2	11,86	9,76	11,43	11,02
2/2	8,95	9,53	9,88	9,45
3	8,99	6,44	8,01	7,81
4	8,34	9,16	7,79	8,43
5	13,4	13,33	13,35	13,36
6	9,24	5,49	5,63	6,79
7	4,85	7,77	5,08	5,90
8	8,11	7,62	8,4	8,04
9	4,95	5,79	5,92	5,55
10	10,61	10,2	8,73	9,85
11	6,11	5,79	5,01	5,63
11/2	9,58	9,36	7,44	8,79
12	6,19	7,4	6,15	6,58
13	9,57	10,87	10,05	10,16
13/2	11,31	11,06	8,55	10,31



MINERALOGICAL COMPATIBILITY AND MECHANICAL CHARACTERISTICS



Almost identical phase composition

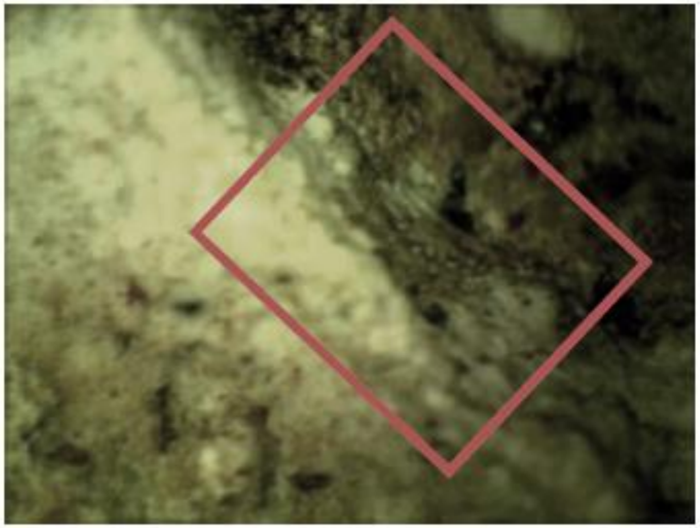
Newly-designed 11

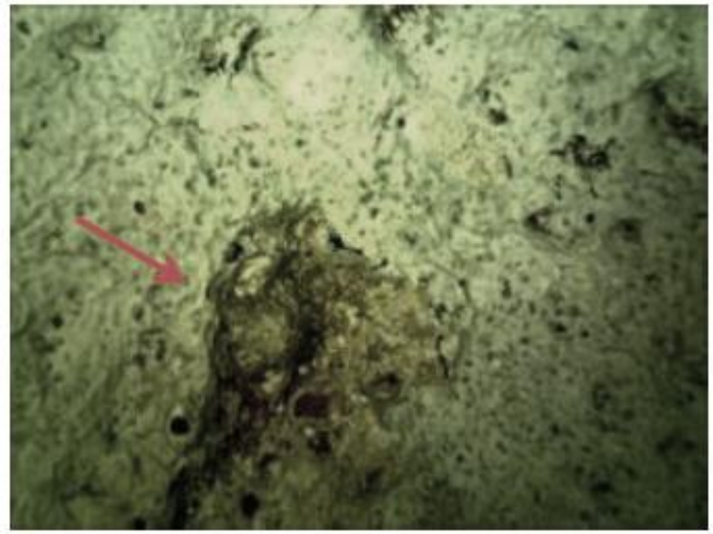
Newly-designed 9

original mortar

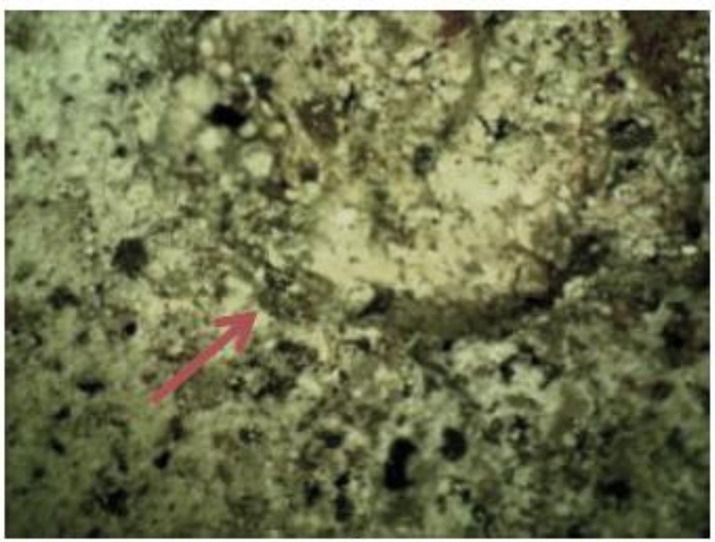
Sample	Compressive streinght (kPa)
Original mortar	1215
Newly-designed 9	274
Newly-designed 11	486

CONTACT ZONE EXAMINATION - MICROSCOPY

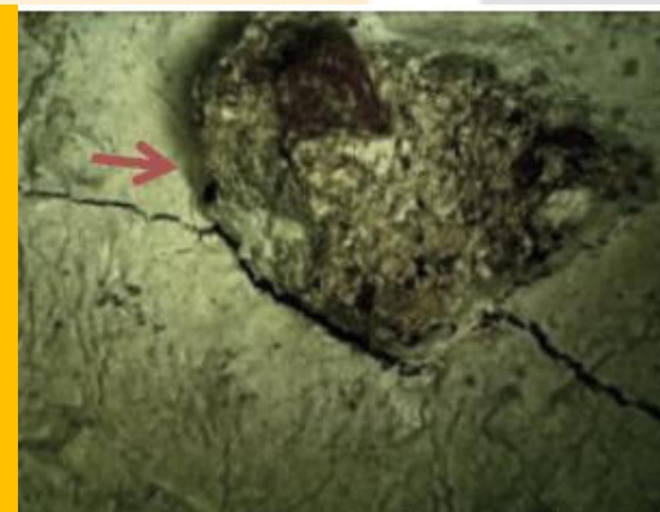




good results of contact zone formation



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.**

Thank you for attention

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