

## Research report of the STUDEC project

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### Material analysis of stucco decoration of the side chapel of

### Saint Nicholas church in Doboszowice, Poland



St. Nicholas church, Doboszowice (DSC\_1413)

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## Obsah

Samples .....	2
Aim of the analysis .....	2
Photodocumentation .....	3
Applied analytical methods.....	5
SDOB 1.....	6
SDOB 2.....	14
SDOB 3.....	28
Conclusion .....	38
List of analyses.....	39

STUDEC	St. Nicholas Church, Doboszowice	<b>Samples</b>

## Samples

Samples acquired by Jan Válek, Sylwia Svorová Pawełkowicz in May 2024.

SDOB 1	SDOB 1.1	Sample of plastic relief – ribbon. Left portal lining, side chapel, St. Nicholas Church, Doboszowice
	SDOB 1.2	
SDOB 2	SDOB 2A	Sample of plastic relief including mortar/plaster background. Left portal lining, side chapel, St. Nicholas Church, Doboszowice
	SDOB 2B	
	SDOB 2C	
	SDOB 2D	
SDOB 3	SDOB 3.1	Sample of green wall painting – decor. Left side of the presbytery, St. Nicholas Church, Doboszowice
	SDOB 3.2	

## Aim of the analysis

The material analysis is carried out as a part of Stucco Decoration across Europe Erasmus+ project (2022-1-CZ01-KA220-HED-000085652).

The research focuses on original stucco work from the late 17th century associated with the circle of artists around Baldassare Fontana (1661 Chiasso – 1733 *ibid.*). The aim is to describe the materials and stucco techniques used and thus create a basis for further research that can compare the findings with other contemporary works or build on them in an interdisciplinary manner with questions regarding authorship, preferences and knowledge of techniques and material processing, or even an assessment of the ability to use local resources, including the qualities they offer.

STUDEC	St. Nicholas Church, Doboszowice	Samples

## Photodocumentation



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## Applied analytical methods

### Optical Microscopy (PLM)

Mortar characteristics and composition were analysed in detail by means of polarised light microscopy and scanning electron microscopy. For these microscopic observations polished cross-sections were prepared. The thin-sections were studied in plane (PPL) and crossed polarised transmitted lights (XPL), polished sections were studied in reflected (RL) and fluorescent (UV) lights using an Olympus BX53M microscope with digital camera Olympus DP27.

### Scanning Electron Microscopy with Energy Dispersive Spectroscopy (SEM-EDS)

Scanning electron microscopy (SEM) was performed by using a Tescan MIRA II LMU instrument, with an energy dispersive analytical system (EDS) from Bruker AXS. The EDS measuring conditions were as follows: carbon coated polished surface, accelerating voltage of electrons 15 kV, WD of 15 mm, high vacuum. The images were taken with a back-scattered electron (BSE) detector. The sites where the elemental composition was determined were chosen with respect to the homogeneity of the measured areas.

### Thermal Analysis (TA)

The composition of the binder component of the mortars was characterised by a thermogravimetric analysis (TGA/DTG) and XRD. Firstly, coating layers were mechanically removed and the purged samples were gently crushed. The obtained material was passed through a sieve, the fraction below 63  $\mu\text{m}$  was used further for analyses.

The instrument SDT Q600 (TA Instruments) was used to measure thermal behaviour between 25°C and 1000°C for which a sample of approximately 10 mg was heated at the rate of 20°C/min in nitrogen atmosphere. Endothermic decomposition of calcite occurs between 600°C and 900°C, the exact temperature depends on the crystallinity, and the amount and type of calcium carbonate phase (**30**).

### X-ray powder diffraction (XRD)

A diffractometer D8Bruker Advance Pro (Cu K $\alpha$  radiation, 40 kV and 40 mA) with 0.01°C step size  $2\theta$  and counting time 0.4 s/step was used to acquire mineralogical data. The crystalline fraction was determined with combined Rietveld-RIR method.

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		<b>SDOB 1</b>

## SDOB 1



Figure 1. Plastic ribbon, sample of stucco. Extraction site of sample SDOB 1.

**Sample:** stucco used for low-relief including surface finishing layers

**Goal:** determination of surface finishing layers, composition of stucco binder

### Procedure

- Sample photographed and documented.
- Polished cross-sections prepared from two pieces of sample containing the surface finishing layers.
- Remaining part of the sample is archived.

### Methods

- Macroscopic description – photodocumentation.
- PLM, SEM + EDS

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 1</b>

## Results SDOB 1



Figure 2. Photography of sample SDOB 1.



Figure 3. Cross-section of two fragments of sample SDOB 1 – SDOB 1.1 (left) and SDOB 1.2 (right).

### SDOB 1.1

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 1</b>



Figure 4. Cross-section of sample SDOB 1.1.



Figure 5. Cross-section of sample SDOB 1.1. Polarised light.

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		<b>SDOB 1</b>

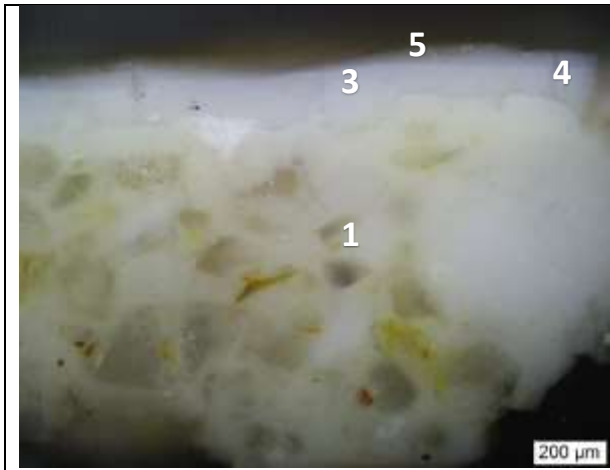


Figure 6. Cross-section of sample SDOB 1.1. Polarised light.

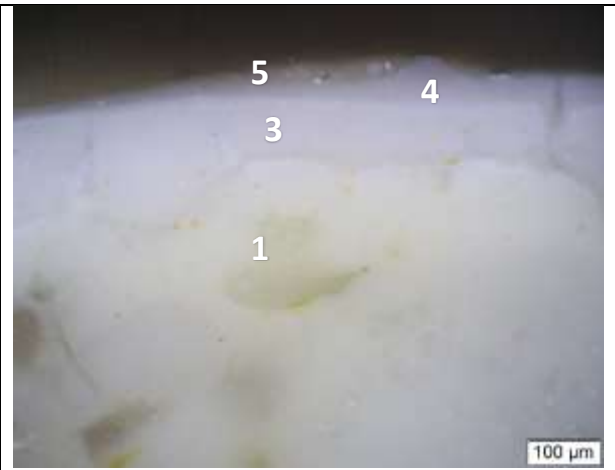


Figure 7. Cross-section of sample SDOB 1.1. Polarised light.

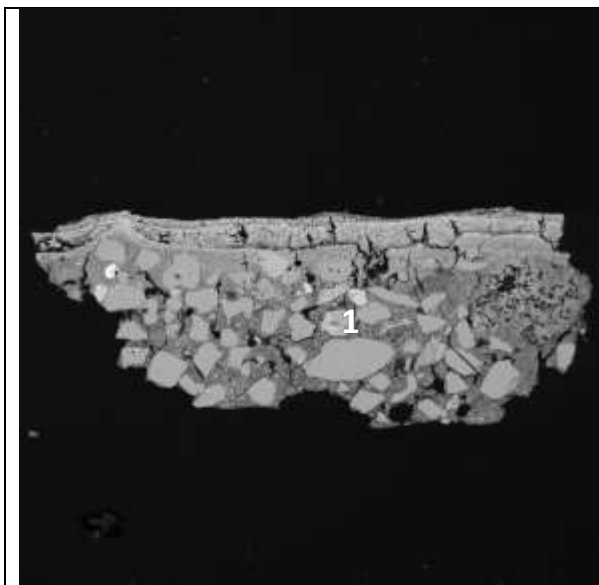


Figure 8. BSE image of sample SDOB 1.1.

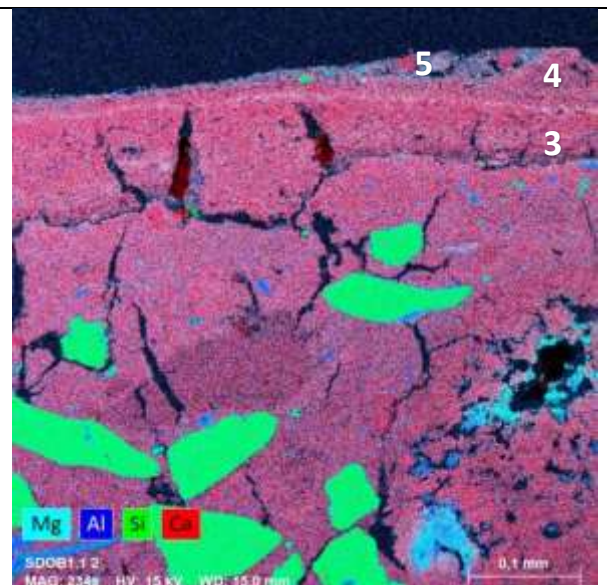


Figure 9. EDS map of elemental distribution in the cross-section.

Table 1. Description of the layers observed in the cross-section SDOB 1.1.

Layer No	Description
1	Plaster with less compact areas with BRP (binder related particles)
2	White lime wash – thin, discontinuous layer
3	Secondary white lime wash layer
4	White lime wash
5	Most probably layer of deposits

STUDEEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 1</b>

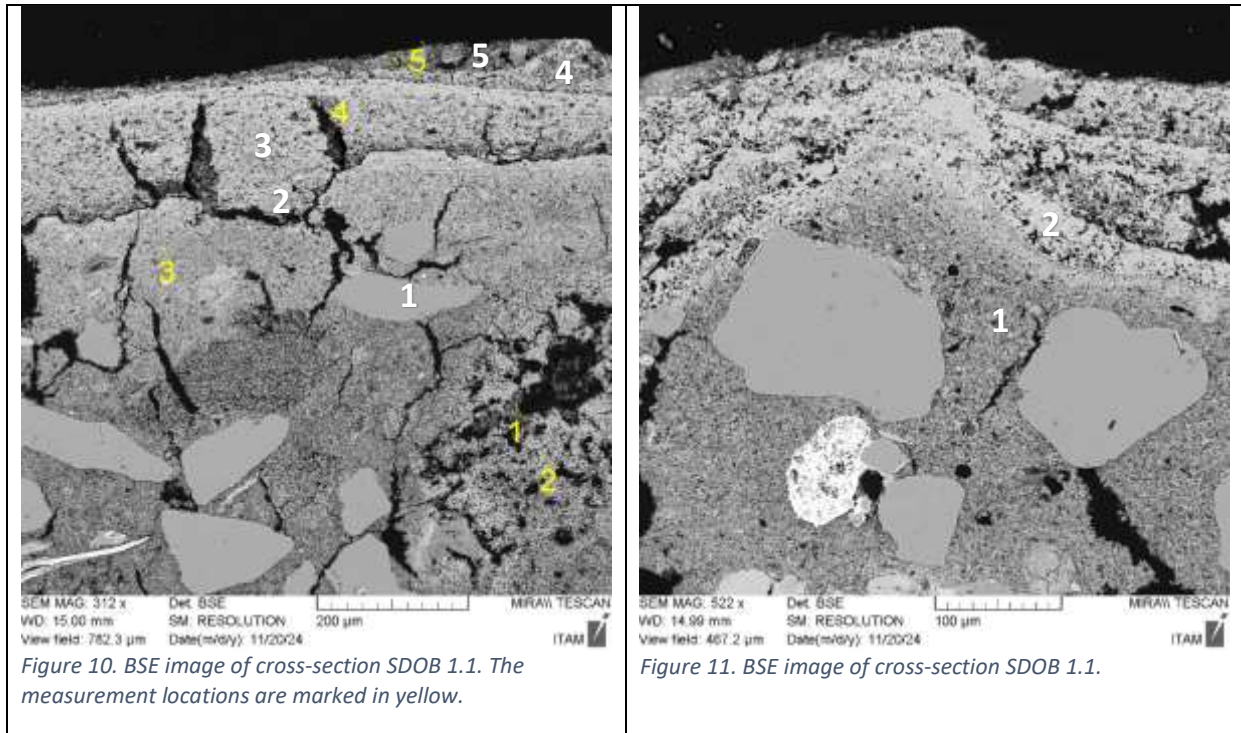


Table 2. Results of EDS measurements expressed in oxides and normalised to 100%. BRP – binder related particle.

Spectrum No	Cl	MgO	SO <sub>3</sub>	CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Interpretation
4		8.5	1.6	89.5	0.4		Layer 3. Secondary whitewash with dolomitic lime
3		5.4	1.1	85.6	6.1	1.8	Layer 1. Plaster – Mg -rich lime
2		4.4		90.6	5.0		Layer 1. BRP – Mg -rich lime
1	0.3	28.5	1.1	23.5	45.4	1.2	Layer 1. BRP/aggregate

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 1</b>

**SDOB 1.2**



*Figure 12. Photography of sample SDOB 1.2.*



*Figure 13. Photography of sample SDOB 1.2. Polarised light.*

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		<b>SDOB 1</b>

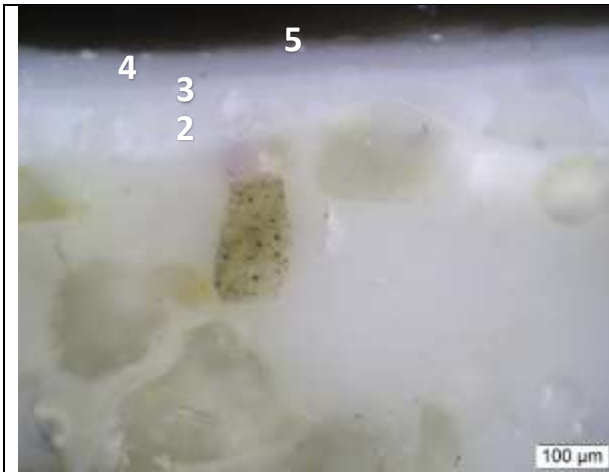


Figure 14. Cross-section of sample SDOB 1.2. Polarised light.



Figure 15. Cross-section of sample SDOB 1.2. UV light.

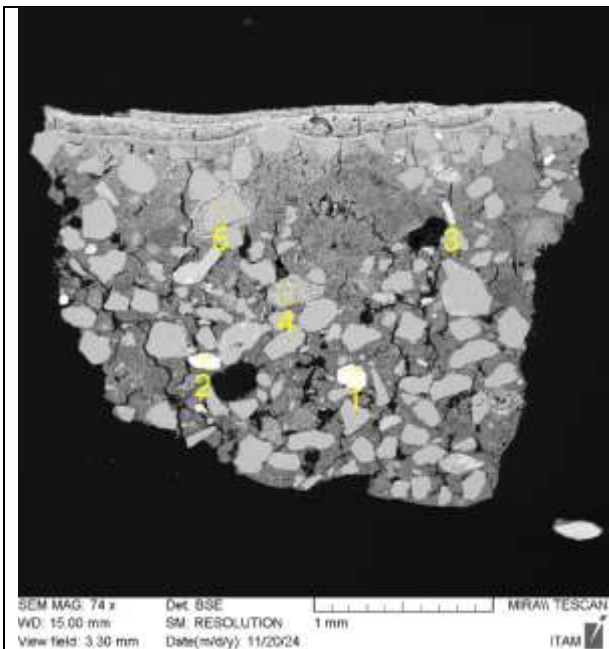


Figure 16. BSE image of sample SDOB 1.2 with measured areas.

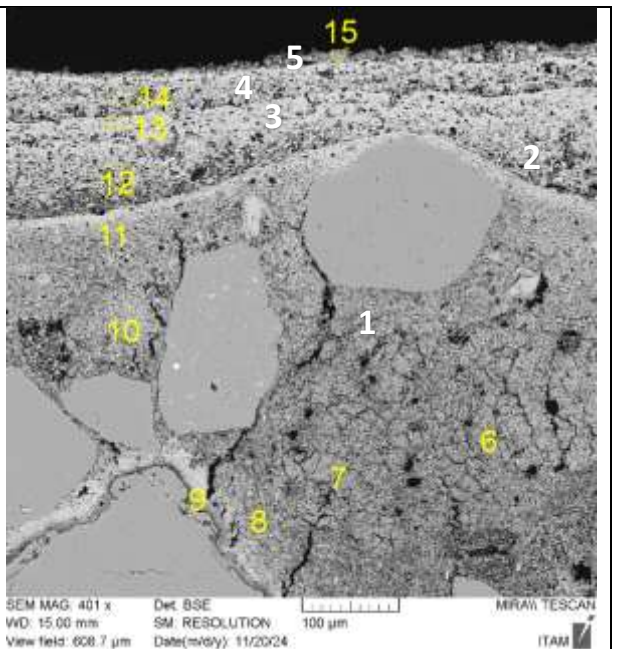


Figure 17. BSE image of sample SDOB 1.2 with measured areas.

Table 3. Results of EDS measurements expressed in oxides and normalised to 100%.

Spect. No	MgO	SO <sub>3</sub>	CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	MnO	FeO	P <sub>2</sub> O <sub>5</sub>	Interpretation
15	14.6	5.2	74.4	3.7	1.2	1.0						Layer 5. Deposits?
14	5.4	2.0	91.9	0.7								Layer 4. Limewash
13	4.8	1.9	92.0	0.8		0.5						Layer 3. Limewash
12	4.4	2.4	89.3	3.9								Layer 2. Limewash

STUDEC	St. Nicholas Church, Doboszowice					2024/2025					
						<b>SDOB 1</b>					

11	5.9	1.3	89.4	3.4								Layer 1. Dense layer on top
10	5.9	0.7	89.9	3.5								Layer 1. Matrix
9	4.8	0.8	85.1	8.4	1.0							Layer 1. Dense layer around Si-particle
8	3.3		89.3	6.5	0.9							Layer 1. Matrix - Mg -rich lime
7	4.2		89.6	6.2								Layer 1. BRP - Mg -rich lime
6	5.6	0.7	90.6	3.1								Layer 1. BRP - Mg -rich lime
5				100								Layer 1: quartz
4				93.6	3.6		2.8					Layer 1. feldspar
3				63.4	19.2	0.4	17.1					Layer 1. feldspar
2	1.1			14.5	6.3		0.6	54.7	1.8	21.0		Layer 1.
1	4.6	1.3	2.4	18.9	4.4		0.6		1.3	65.0	1.6	Layer 1.

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 2</b>

## SDOB 2



Figure 18. Transition between the plastic part and the flat background. Extraction site of sample SDOB 2.

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 2</b>



Figure 19. Extraction site of sample SDOB 2.

**Sample:** stucco used for low-relief including surface finishing layers. Sample divided into parts: SDOB 2A, transition (printed line) SDOB 2B, plane background SDOB 2C, core plaster SDOB 2D. SDOB 2C and SDOB 2D are similar or even identical samples

**Goal:** determination of surface finishing layers, composition of stucco binder and of the plane background.

## Procedure

- Sample photographed and documented.
- Cross-section executed from a piece of sample containing the surface finishing layers (SDOB 2A, SDOB 2B, SDOB 2C).
- Part of sample crushed and the fraction  $<0,063\text{mm}$  given for TA – SDOB 2D.
- Preservation of the rest of sample.

## Methods

- Macroscopic description – photodocumentation
- PLM, SEM + EDS, TA

## Results SDOB 2

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 2</b>

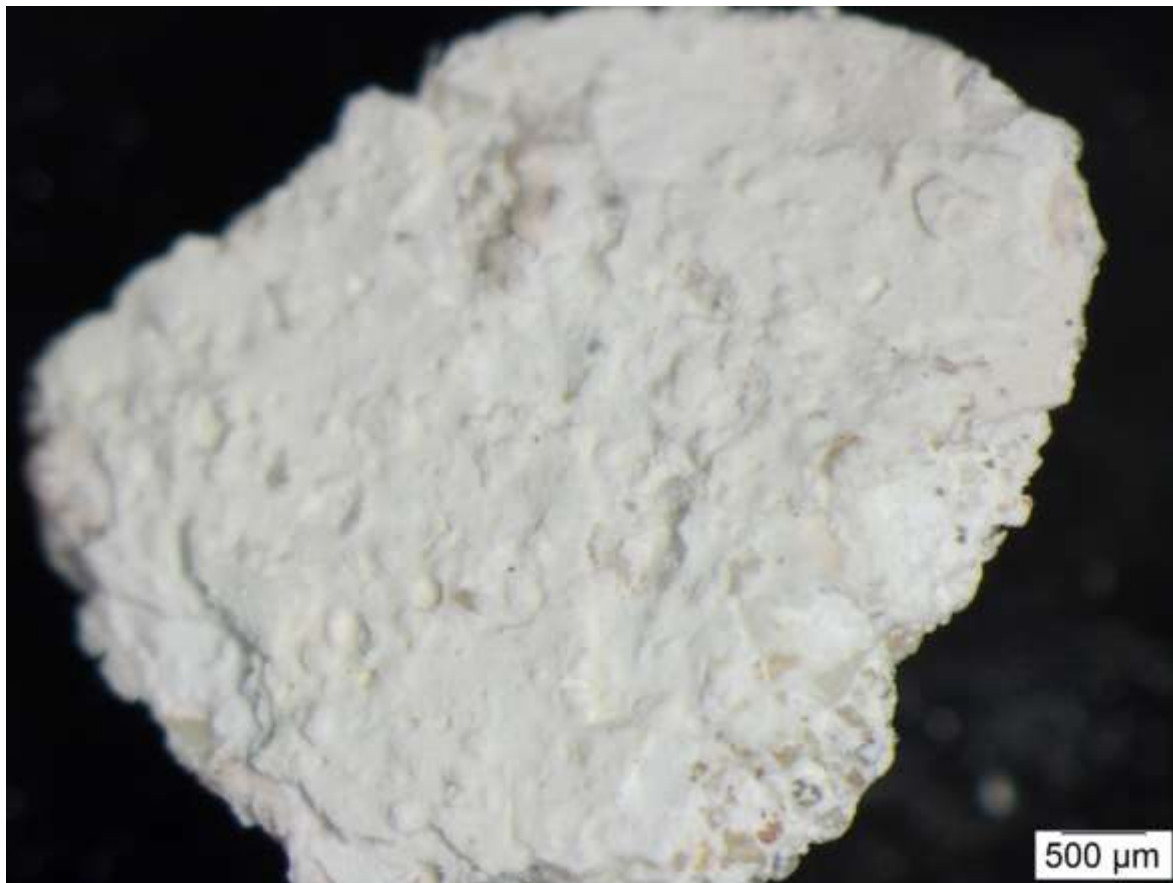


Figure 20. Photography of sample SDOB 2A.



Figure 21. Photography of sample SDOB 2B.

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 2</b>



*Figure 22. Photography of sample SDOB 2C.*

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 2</b>



*Figure 23. Photography of sample SDOB 2D.*

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 2</b>

**SDOB 2A**



Figure 24. Cross-section of sample SDOB 2A.

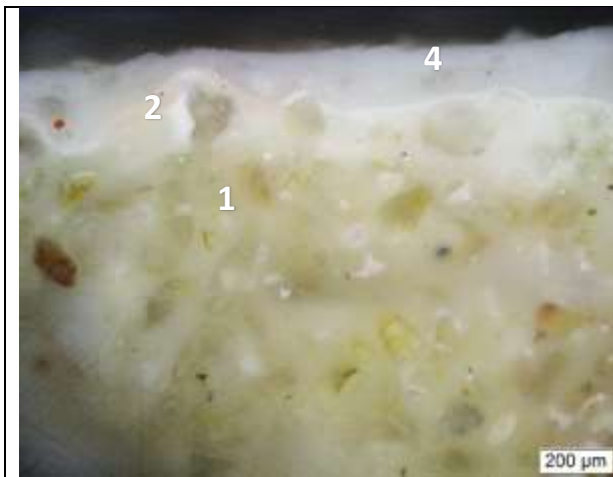


Figure 25. Cross-section of sample SDOB 2A. Polarised light.

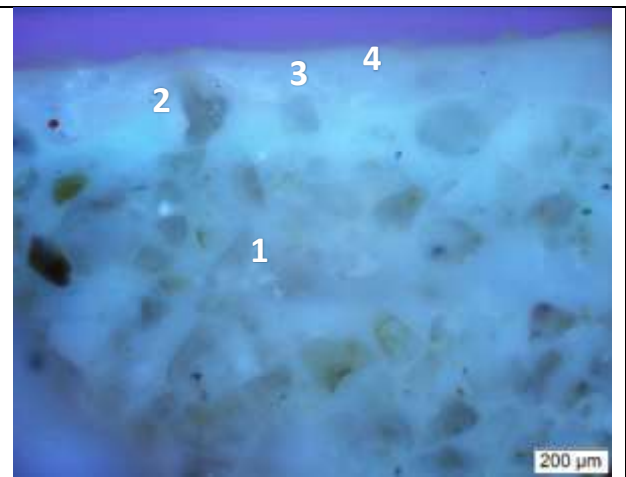


Figure 26. Cross-section of sample SDOB 2A. UV light.

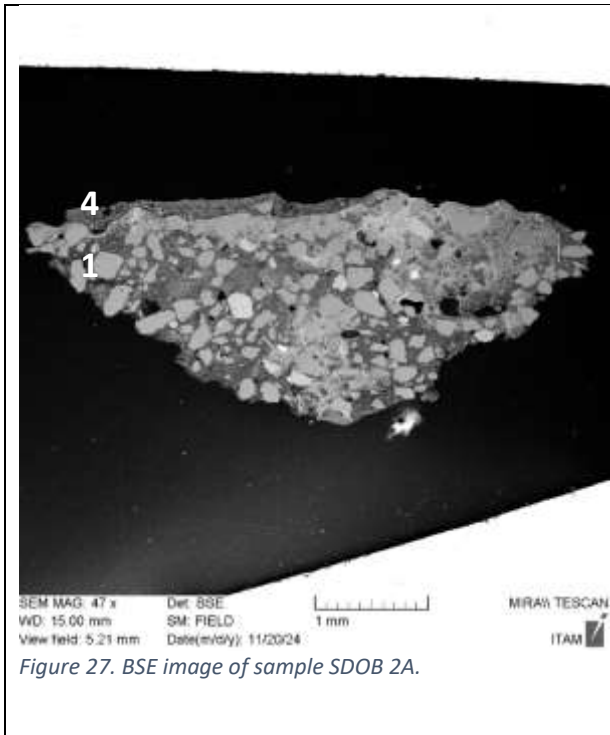


Figure 27. BSE image of sample SDOB 2A.

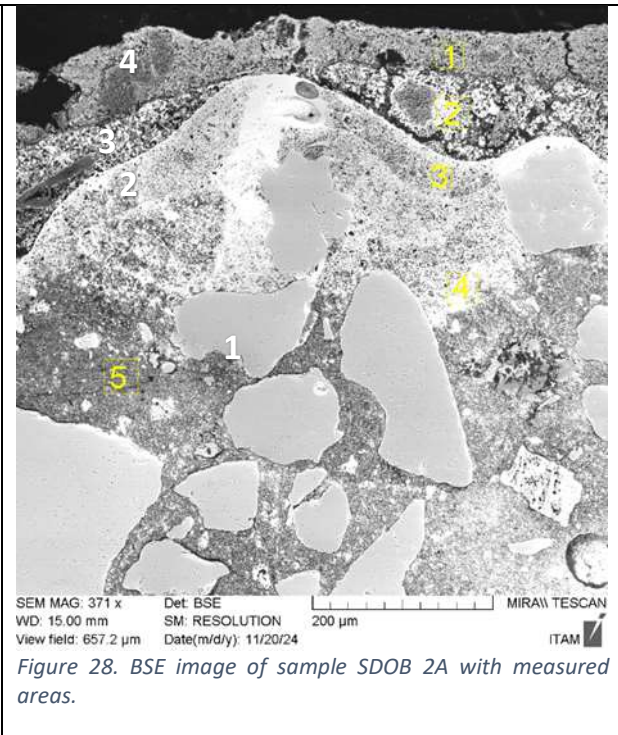


Figure 28. BSE image of sample SDOB 2A with measured areas.

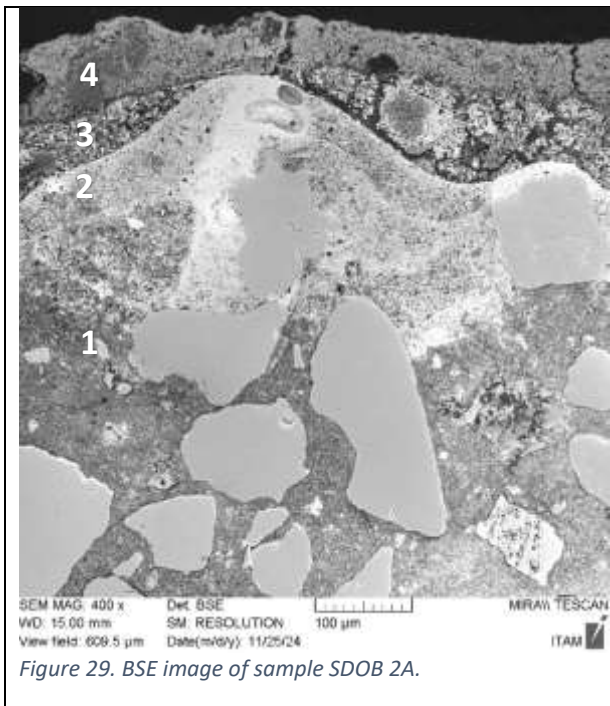


Figure 29. BSE image of sample SDOB 2A.

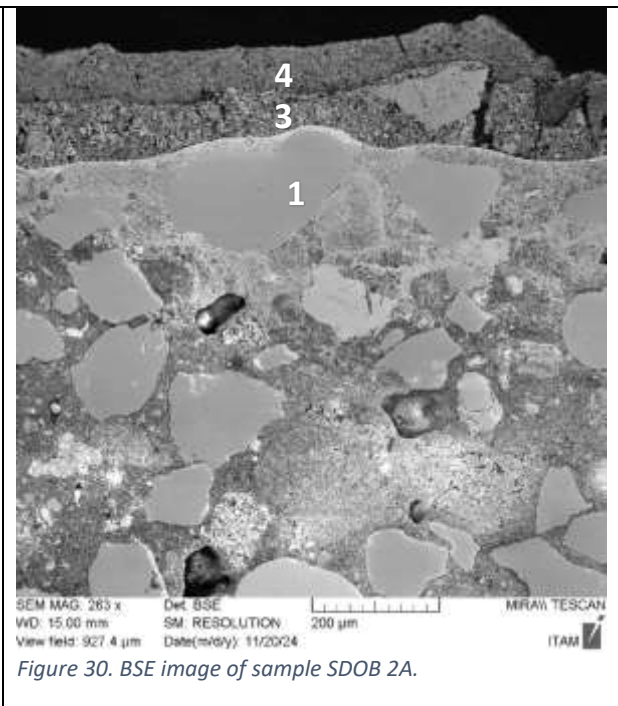


Figure 30. BSE image of sample SDOB 2A.

Table 4. Description of the layers observed in the cross-section SDOB2A.

Layer No	Description
1	Plaster with less compact structure and with BRP (binder related particles)
2	Pink paint layer
3	Secondary white lime wash layer
4	White lime wash layer

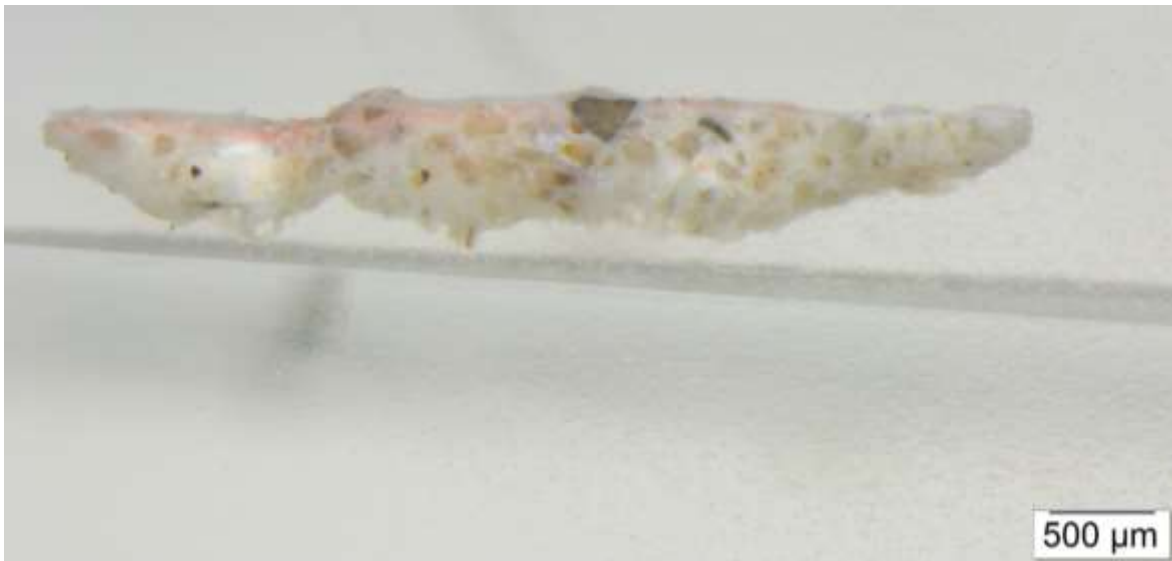
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		<b>SDOB 2</b>

Table 5. Results of EDS measurements expressed in oxides and normalised to 100%.

Spectrum No	MgO	SO <sub>3</sub>	CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	Interpretation
5	4.6	3.6	70.6	16.7	2.6	2.0	Layer 1. matrix
4	4.5	1.4	88.3	3.8		2.1	Layer 1. matrix
3	3.1		88.7	6.4		1.7	Layer 2. Paintlayer; the pigment is dispersed in limewash
2	8.5		87.8	2.6		1.0	Layer 3. Limewash. Dolomitic lime
1	40.9	1.3	49.1	4.6	2.5	1.7	Layer 4. Limewash. Highly dolomitic lime

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 2</b>

**SDOB 2B**



*Figure 31. Cross-section of sample SDOB 2B.*



*Figure 32. Cross-section of sample SDOB 2B. Polarised light.*

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 2</b>

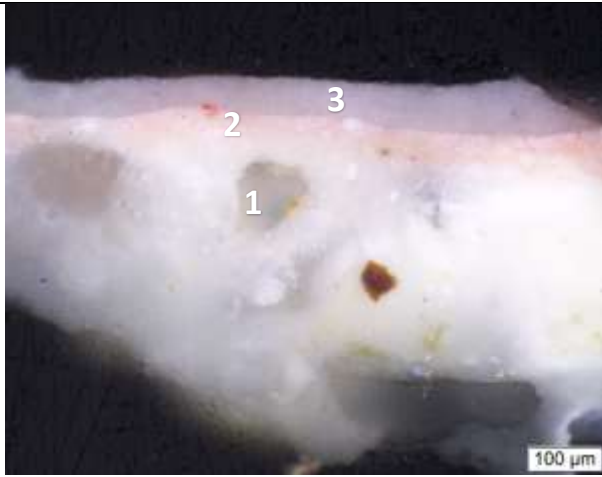


Figure 33. Cross-section of sample SDOB 2B. Polarised light.

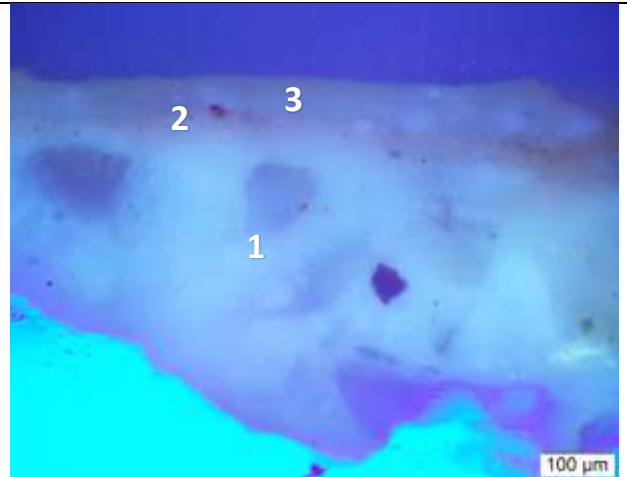


Figure 34. Cross-section of sample SDOB 2B. UV light.

4

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		<b>SDOB 2</b>



Figure 35. Cross-section of sample SDOB 2B. Area mapped with EDS detector.

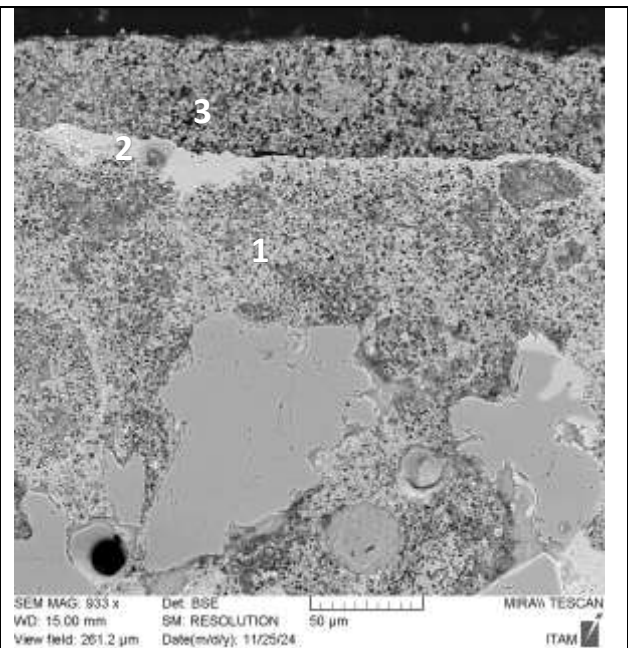


Figure 36. BSE image of area mapped with EDS detector.

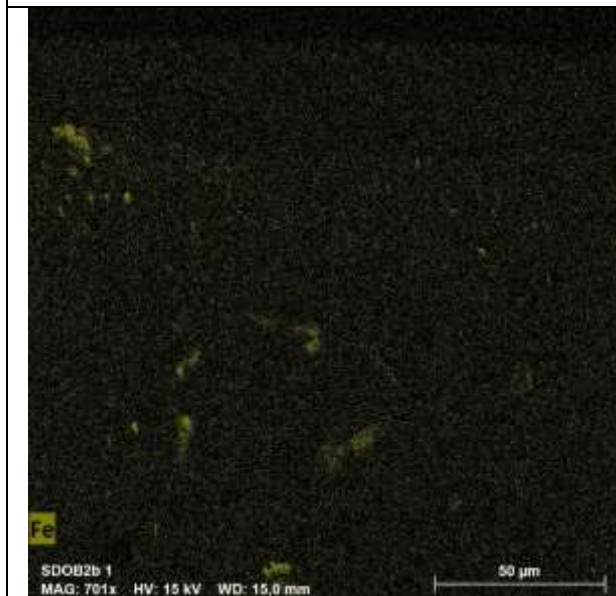


Figure 37. EDS map of distribution of iron in the cross-section.

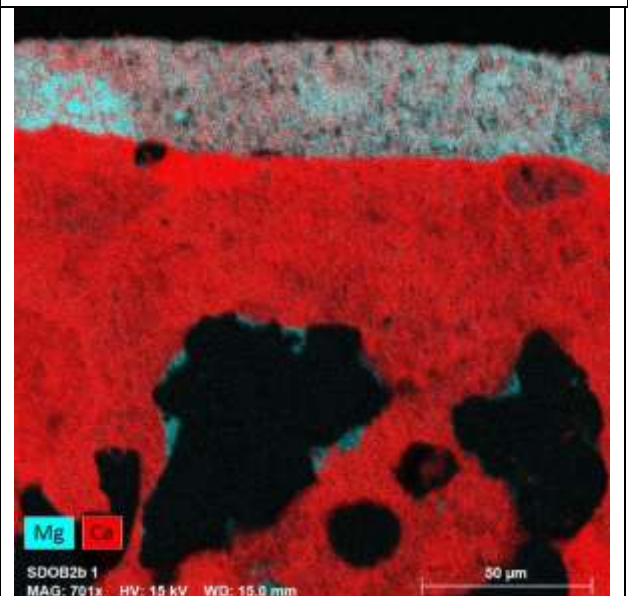


Figure 38. EDS map of distribution of magnesium and calcium in the cross-section.

Table 6. Description of the layers observed in the cross-section SDOB 2A

Layer No	Description
1	Plaster
2	Pink paint layer; the interface between layers 1 and 2 is not clearly defined, suggesting a fresco technique
3	Secondary, highly dolomitic white lime wash layer
4	White highly dolomitic lime wash layer

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		<b>SDOB 2</b>

**SDOB 2C**



Figure 39. Cross-section of sample SDOB 2C.

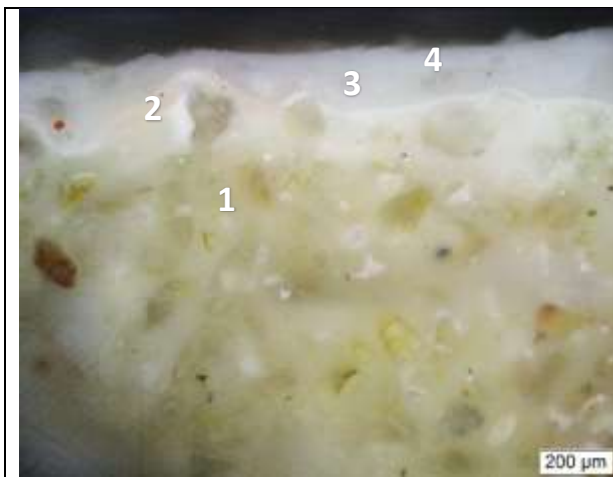


Figure 40. Cross-section of sample SDOB 2C.

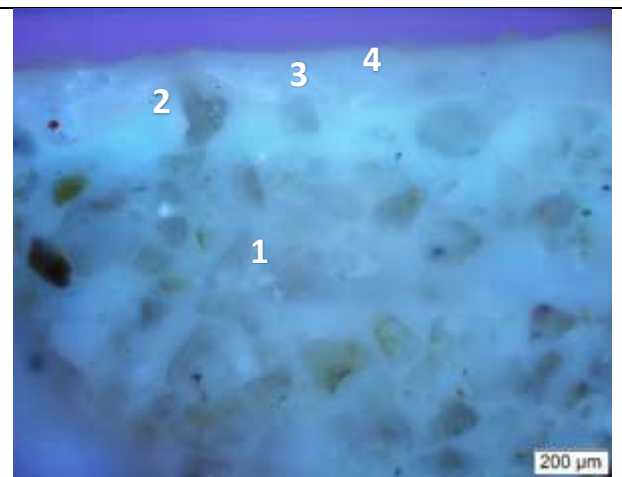


Figure 41. Cross-section of sample SDOB 2C. UV light.

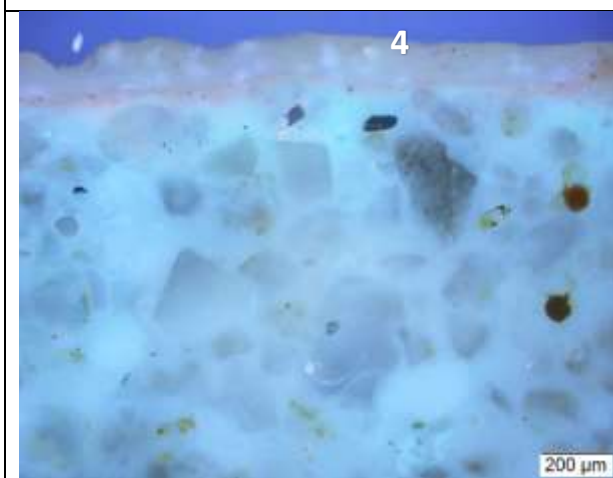


Figure 42. Cross-section of sample SDOB 2C. UV light.



Figure 43. Cross-section of sample SDOB 2C. Polarised light.

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 2</b>

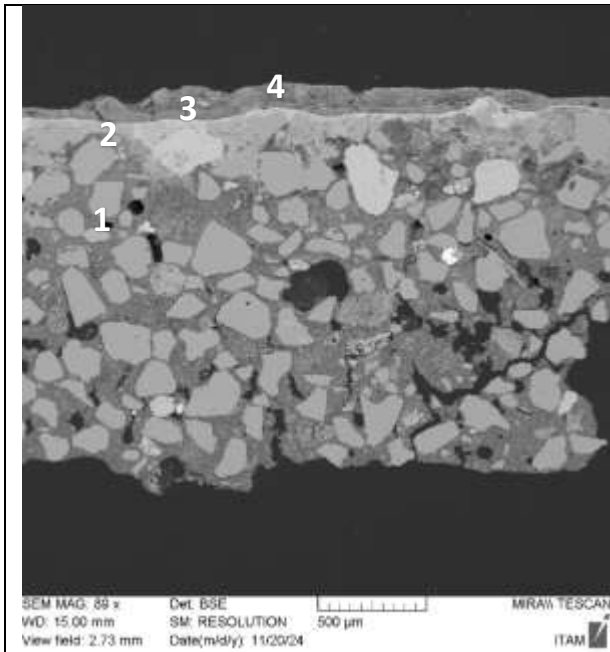


Figure 44. BSE image of sample SDOB 2C.

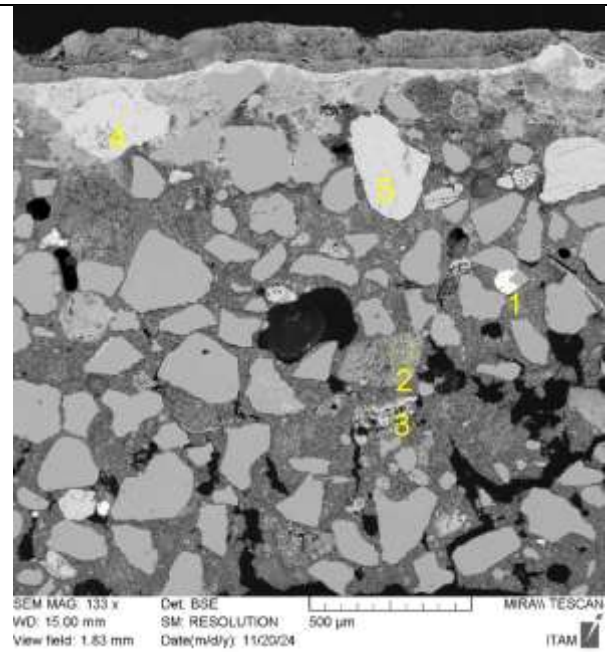


Figure 45. BSE image of sample SDOB 2C with measured areas.

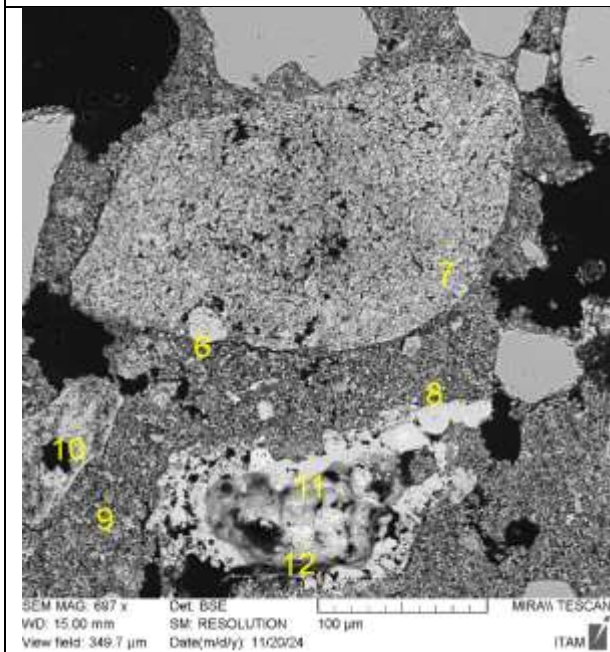


Figure 46. BSE image of sample SDOB 2C with measured areas.

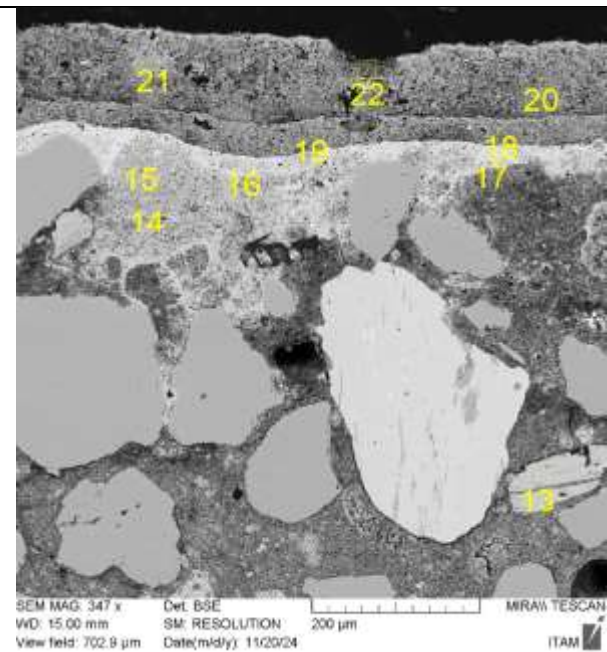


Figure 47. BSE image of sample SDOB 2C with measured areas.

Table 7. Description of the layers observed in the cross-section SDOB 2C.

Layer No	Description
1	Plaster
2	Pink paint layer; the interface between layers 1 and 2 is not clearly defined, suggesting a fresco technique
3	Secondary, highly dolomitic white lime wash layer
4	White highly dolomitic lime wash layer
5	Deposit or secondary pink layer

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 2</b>

Table 8. Results of EDS measurements expressed in oxides and normalised to 100%.

Spec. No	MgO	SO <sub>3</sub>	CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	MnO	FeO	P <sub>2</sub> O <sub>5</sub>	Cl	Interpretation
24	0.7		39.5	1.2		0.7				58.0			Layer 2. Iron oxide as red pigment
23	1.0		24.3	1.2						73.5			Layer 2. Pigment
22	10.4	2.3	65.8	15.3	3.3	1.5				1.3			Layer 5. Deposit?
21	26.1	1.2	69.3	2.0		1.4							Layer 4. Matrix
20	35.1	2.0	60.8	2.1									Layer 4. Matrix
19	34.1	1.4	63.3	1.2									Layer 3. Matrix
18	26.2	1.7	66.5	2.9	1.8	1.0							Layer 3. Matrix – highly dolomitic lime
17	1.2	1.4	96.2	1.2									Layer 2. matrix
16	1.5		50.4	3.8						44.2			Layer 2.
15	2.4	0.9	94.8	1.9									Layer 1. Deposits?
14	2.8		94.0	2.3		0.9							Layer 1. Limewash
13	1.0			47.5	37.6	0.5	11.3	0.6		1.5			Layer 1. feldspar
12	4.8	1.1	76.7	13.1	4.3								Layer 1. Calcareous particle
11	0.8	1.3	97.1	0.8									Layer 1. Calcareous particle
10	1.6	1.6	94.9	2.0									Layer 1. BRP
9	2.3	2.3	89.9	5.5									Layer 1. matrix- dolomitic lime
8	2.0	1.9	86.2	8.4	1.2							0.3	Layer 1. Matrix - dolomitic lime
7	2.0	1.1	94.5	2.5									Layer 1. BRP – dolomitic lime
6	1.3	1.2	95.8	1.7									Layer 1. BRP – dolomitic lime
5				63.7	19.4	0.8	16.1						Layer 1: feldspar
4				63.6	19.2	0.6	16.6						Layer 1. feldspar
3		1.4	97.9	0.8									Layer 1. Calcareous particle
2	1.0	0.8	95.7	2.2								0.3	Layer 1. BRP
1	3.6	1.4	2.3	15.7	3.1				0.8	70.6	2.7		Layer 1. Brownish grain

STUDEEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 3</b>

## SDOB 3



Figure 48. Extraction site of sample SDOB 3 from the presbytery.

**Sample:** wall painting - sample of green including the plaster

**Goal:** determination of pigment and comparison of the plaster layer with the layer under the stucco in the chapel

### Procedure

- Sample photographed and documented.
- Cross-section executed from a piece of sample containing green pigment.
- Part of sample crushed and the fraction  $<0,063\text{mm}$  given for TA.
- Preservation of the rest of sample.

### Methods

- Macroscopic description – photodocumentation.
- cross-section optical microscopy of cross-section
- SEM + EDS
- TA

### Results SDOB 3

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 3</b>



Figure 49. Photography of sample SDOB 3.

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		<b>SDOB 3</b>

4

**SDOB 3.1**

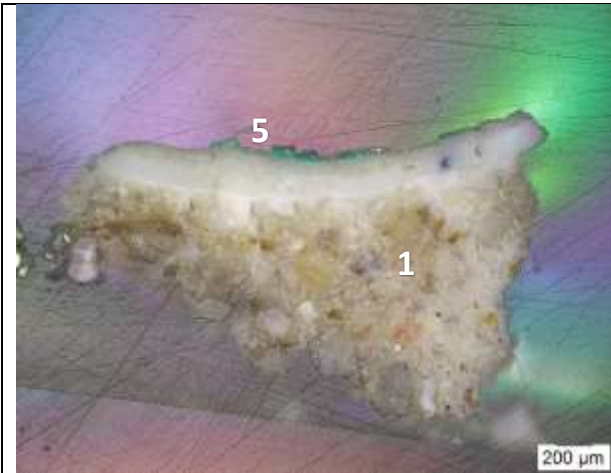


Figure 50. Cross-section of sample SDOB 3.1.

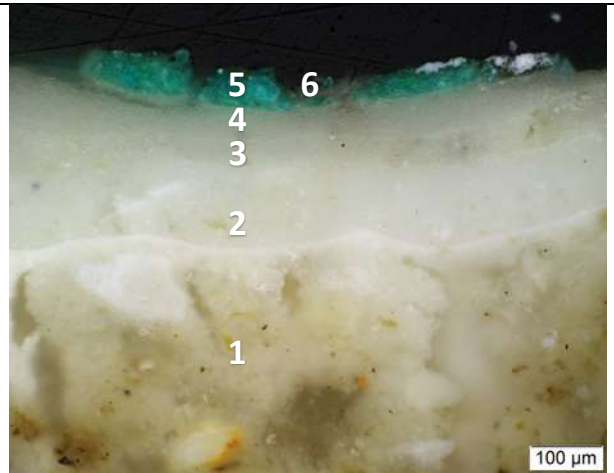


Figure 51. Cross-section of sample SDOB 3.1. UV light.



Figure 52. Cross-section of sample SDOB 3.1. UV light.

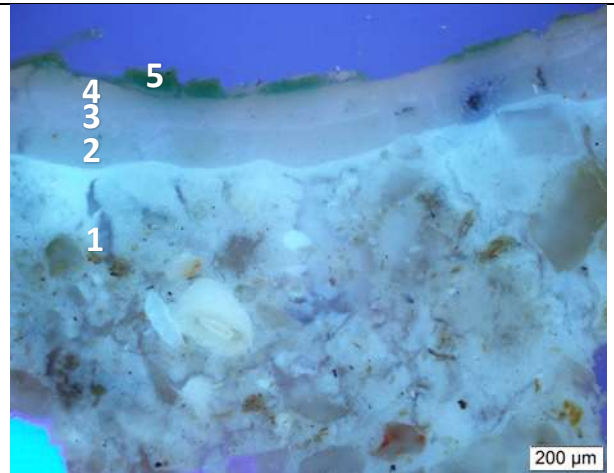


Figure 53. Cross-section of sample SDOB 3.1. Polarised light.

STUDEEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 3</b>

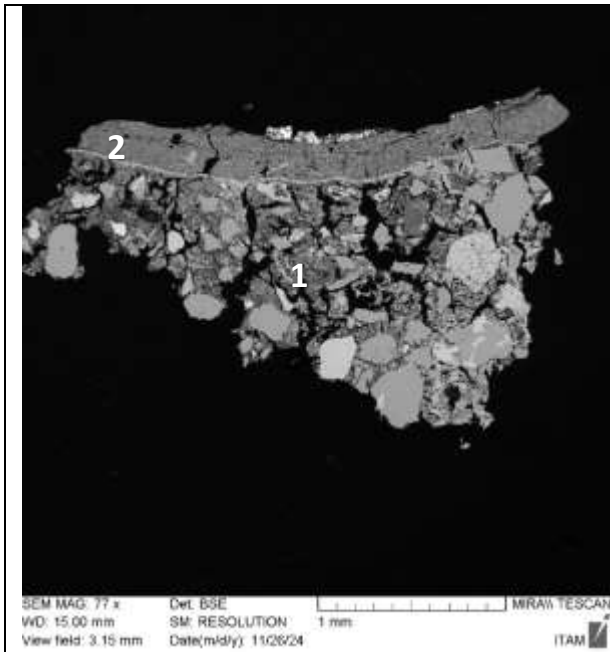


Figure 54. BSE image of sample SDOB 3.1.

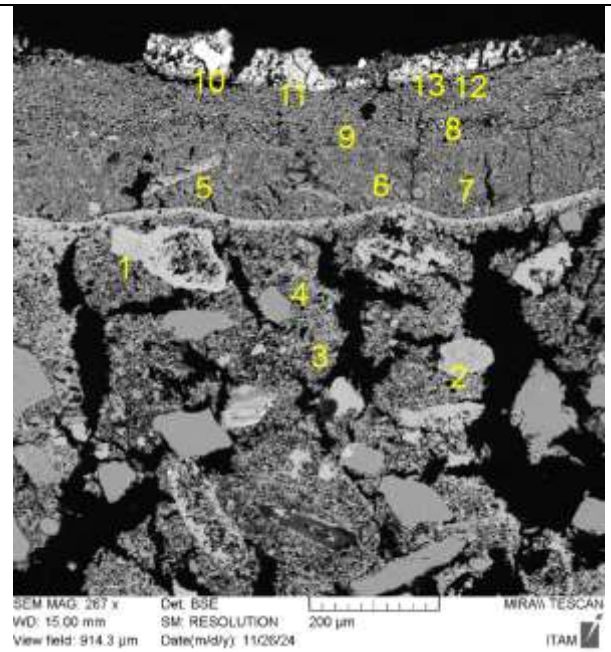


Figure 55. BSE image of sample SDOB 3.1 with measured areas.

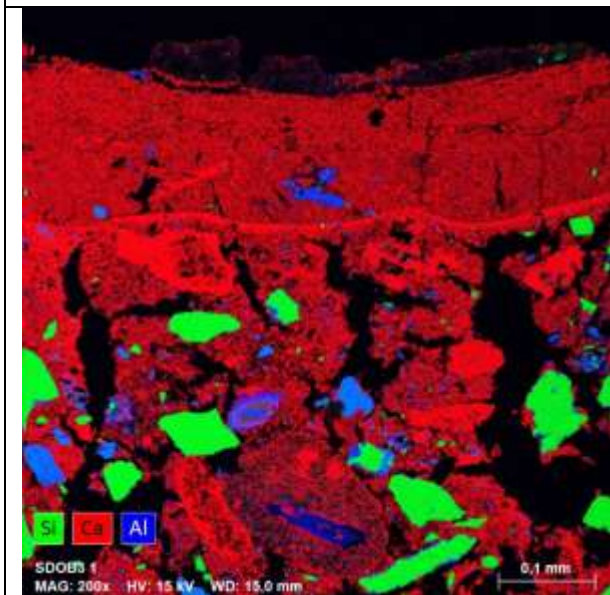


Figure 56. EDS map of distribution of silica, calcium and alumina in the cross-section.

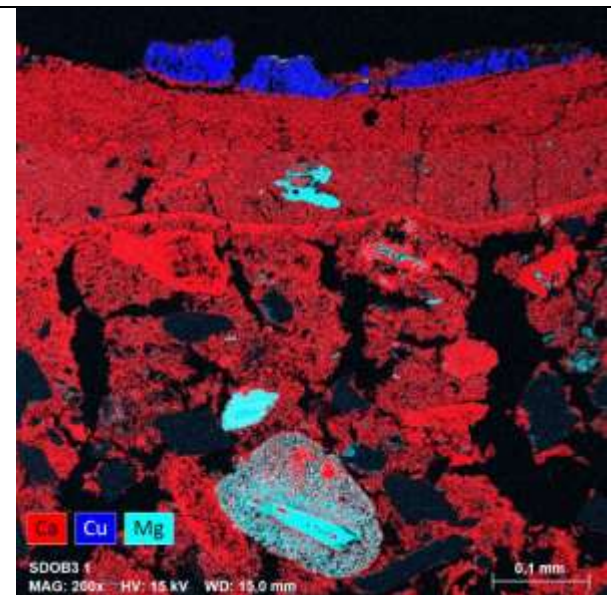
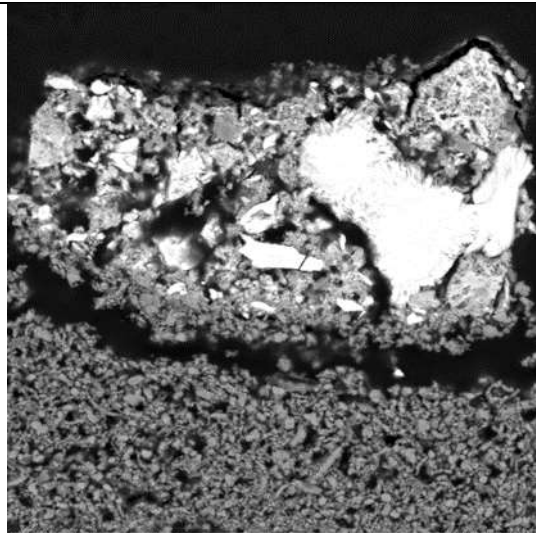


Figure 57. EDS map of distribution of calcium, copper and magnesium in the cross-section.

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 3</b>



Figure 58. Photo of a mapped area.



SEM MAG: 1.75 kx Det: BSE  
 WD: 15.00 mm SM: RESOLUTION 20 µm  
 View field: 139.6 µm Date(m/d/y): 11/26/24  
 MIRAM TESCAN  
 ITAM

Figure 59. BSE image of a mapped area..

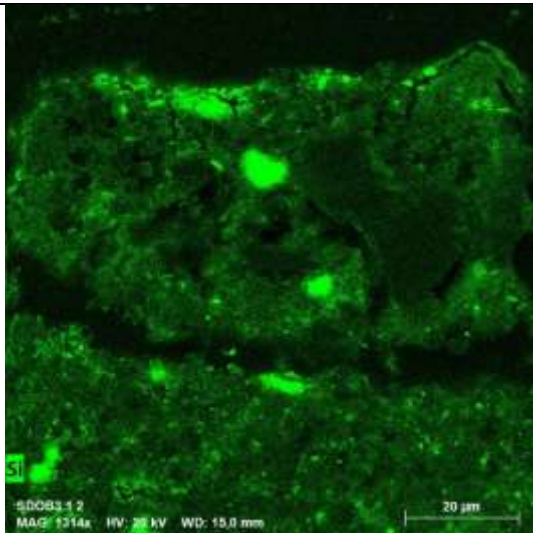


Figure 60. EDS map of distribution of calcium in the cross-section.

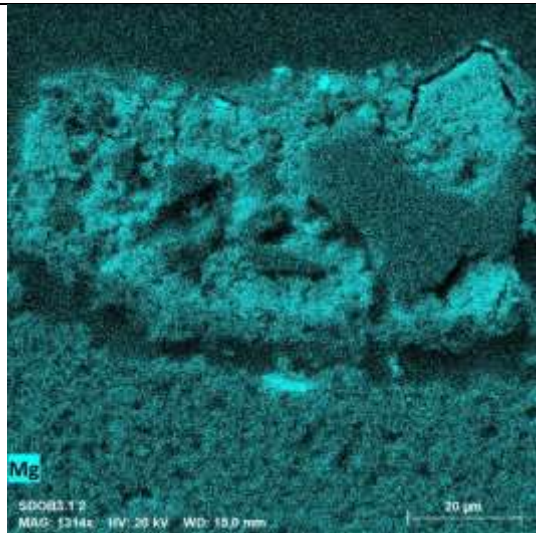


Figure 61. EDS map of distribution of magnesium in the cross-section.

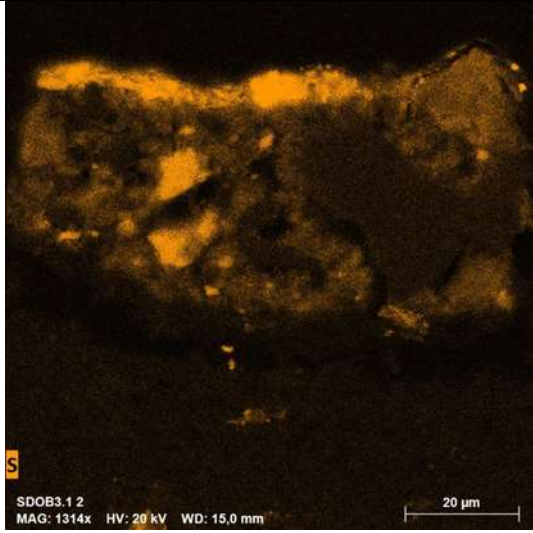


Figure 62. EDS map of distribution of sulphur in the cross-section.

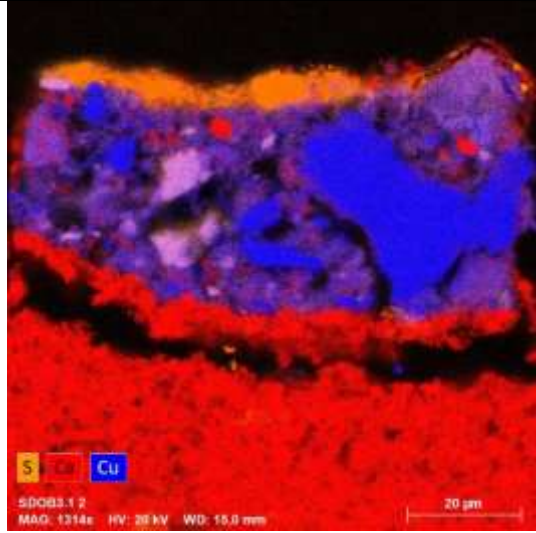


Figure 63. EDS map of distribution of sulphur, calcium and copper in the cross-section.

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 3</b>

Table 9. Description of the layers observed in the cross-section SDOB 3.1.

Layer No	Description
1	Ochre plaster with low magnesium content (below 2%)
2	White limewash with magnesium content of ca. 2%
3	White limewash
4	White limewash with low magnesium content (below 1%)
5	Green paint layer – probably copper carbonate mixed (malachite?) with small amount of copper sulphates (brochantite?); the binder of the paint layer must be organic as almost no lime has been detected in the layer
6	Deposit and degradation thin layer visible in EDS map more than in optical microscope; with high silica and sulphur content

Table 10. Results of EDS measurements expressed in oxides and normalised to 100%.

Spect. No	MgO	SO <sub>3</sub>	CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CuO	P <sub>2</sub> O <sub>5</sub>	Cl	Interpretation
14		21.4	0.6	0.5		77.4			Layer 5. copper sulphate (brochantite?)
13			0.7			99.3			Layer 5. Copper carbonate (malachite ?)
12			1.2			98.8			Layer 5. Malachite?
11	3.3	9.7	6.5	2.5		76.1	1.0	0.8	Layer 5. Cu pigment
10			1.0	1.1		97.9			Layer 5. Malachite?
9	0.8	1.2	96.3	1.8					Layer 4. matrix
8			99.4	0.6					Layer 4. Matrix
7	2.5		93.4	4.1					Layer 2. matrix – Mg lime
6	2.0		95.2	2.8					Layer 2. matrix – Mg lime
5	0.8	1.2	96.9	1.2		0.8			Layer 2: Calcareous particle
4	1.8		96.4	1.8		0.6			Layer 1. Mg lime
3	1.5		94.3	3.6	0.6				Layer 1. Mg lime
2	1.5		96.9	1.6					Layer 1. BRP
1	1.0	1.2	95.0	2.8					Layer 1. Calcareous particle

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 3</b>

**SDOB 3.2** 4

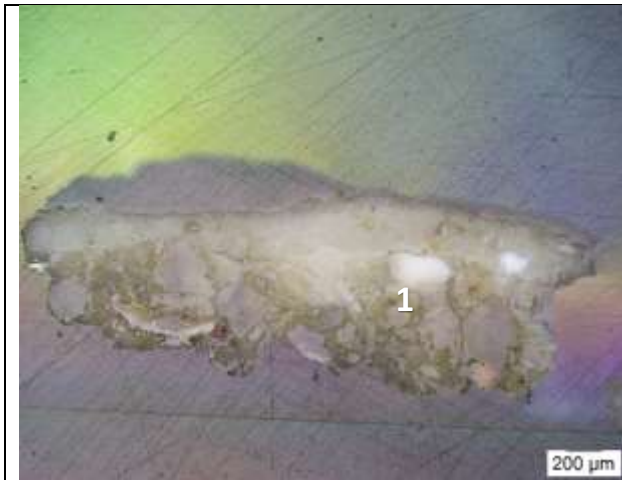


Figure 64. Cross-section of sample SDOB 3.2.

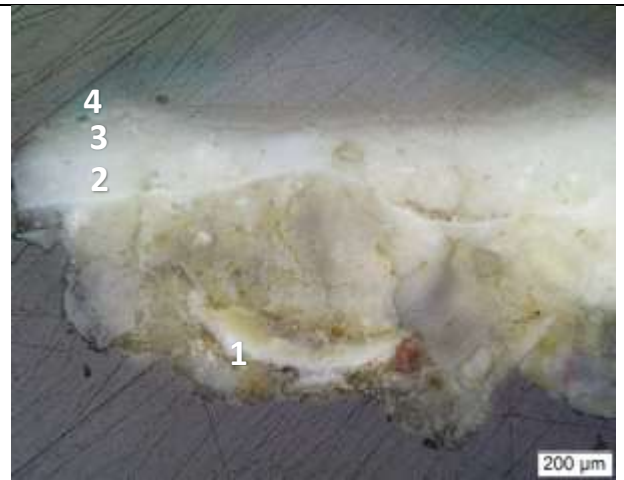


Figure 65. Cross-section of sample SDOB 3.2.



Figure 66. Cross-section of sample SDOB 3.2.

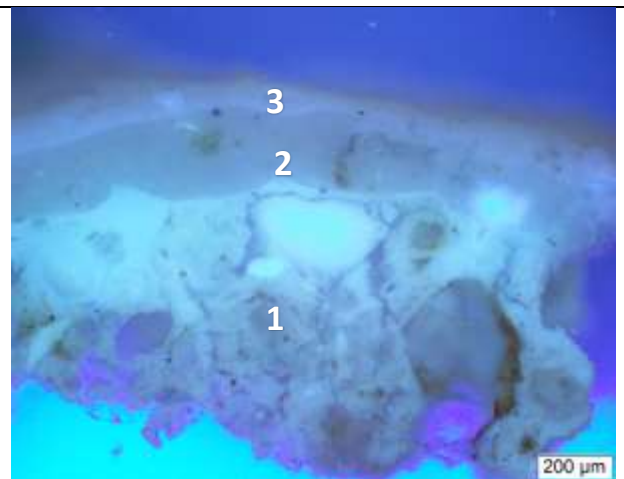


Figure 67. Cross-section of sample SDOB 3.2. UV light.

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 3</b>

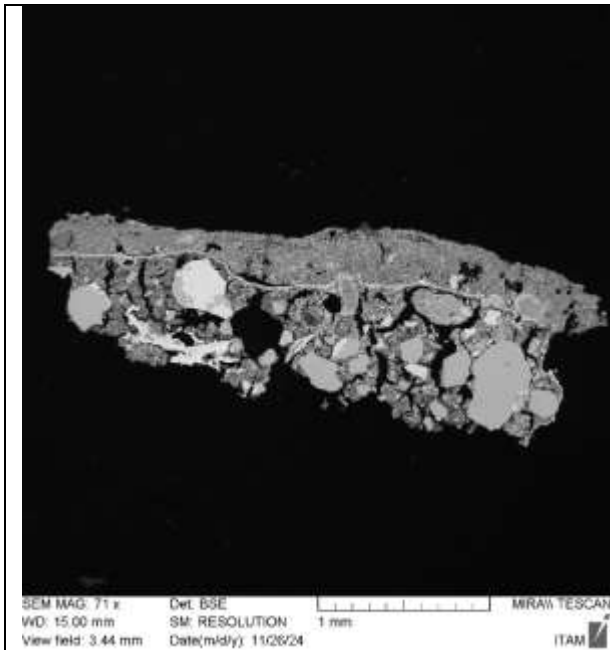


Figure 68. BSE image of sample SDOB 3.2.

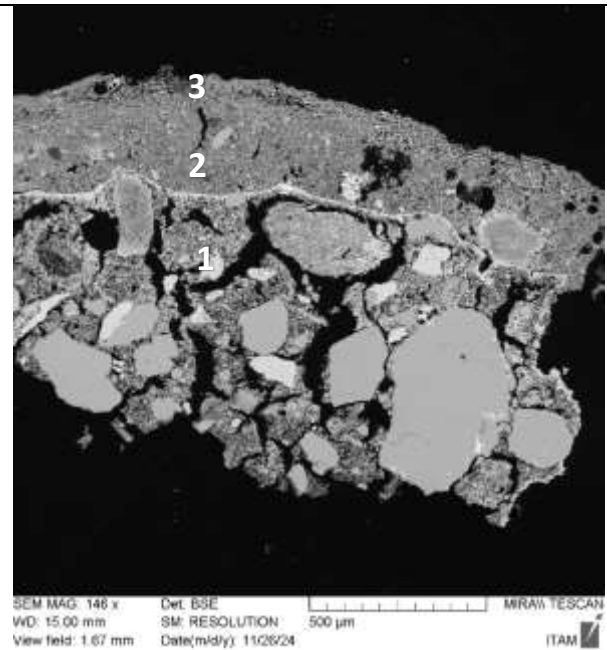


Figure 69. BSE image of sample SDOB 3.2 with measured areas.

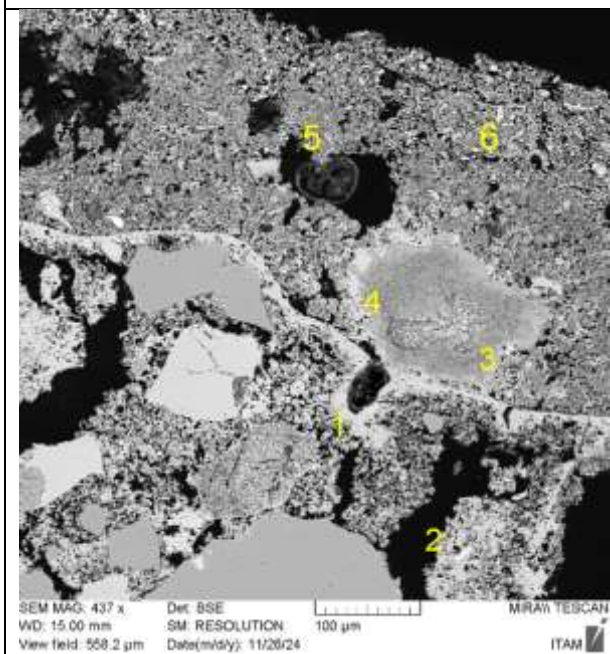


Figure 70. BSE image of sample SDOB 3.2 with measured areas.

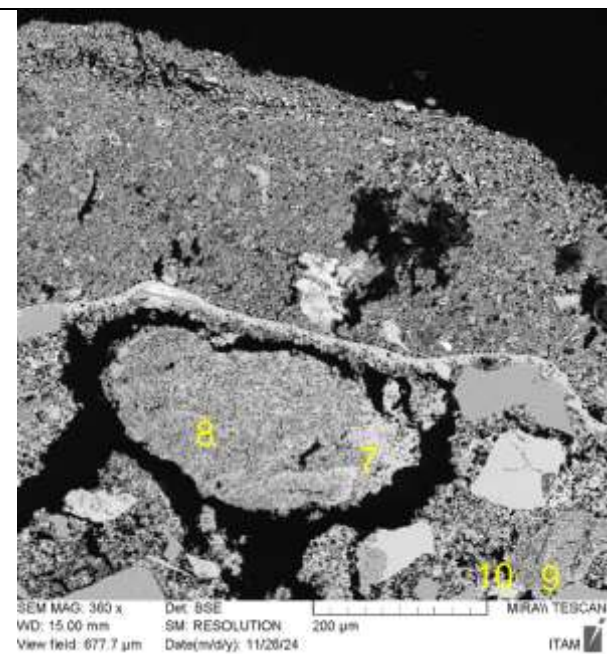


Figure 71. BSE image of sample SDOB 3.2 with measured areas.

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 3</b>

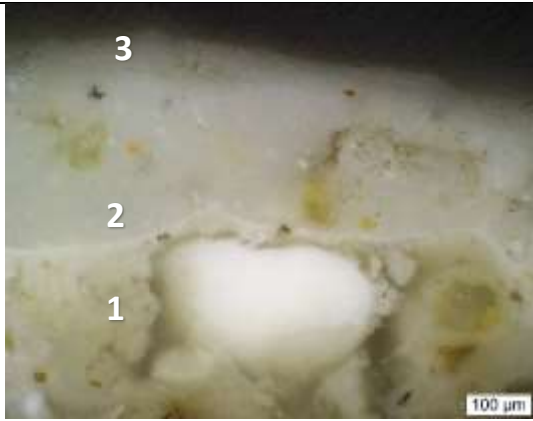


Figure 72. Photo of a mapped area.

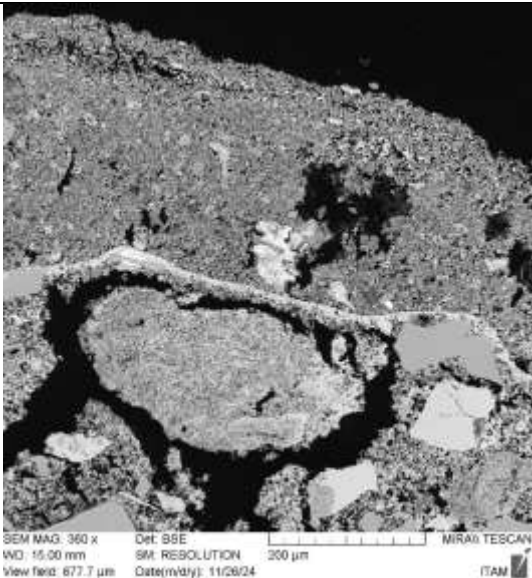


Figure 73. BSE image of a mapped area..

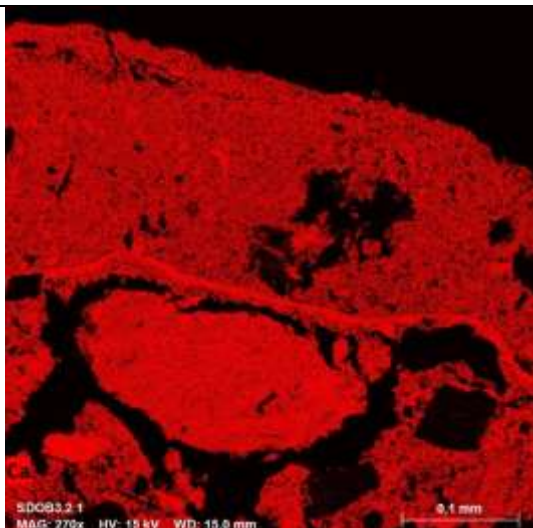


Figure 74. EDS map of distribution of calcium in the cross-section.

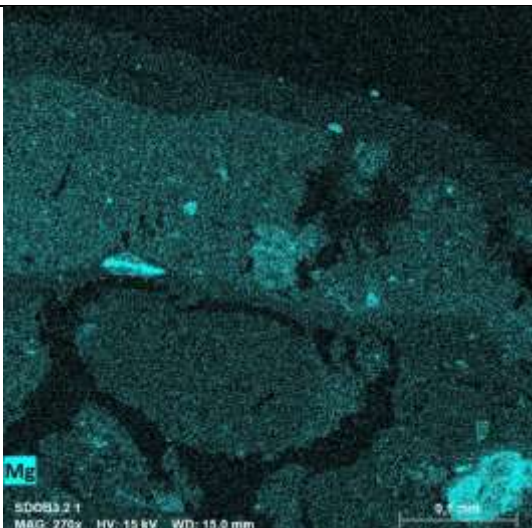


Figure 75. EDS map of distribution of magnesium in the cross-section.

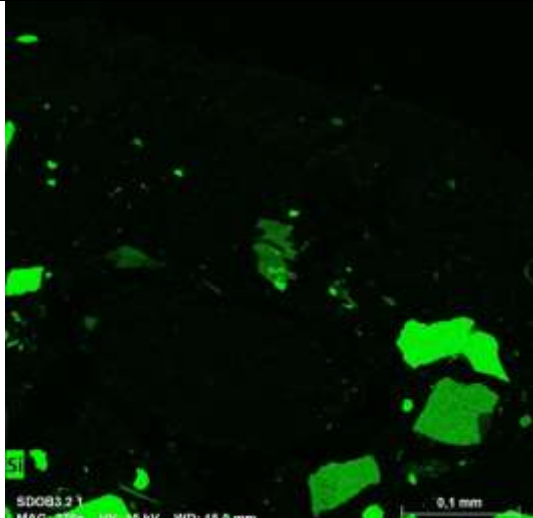


Figure 76. EDS map of distribution of silica in the cross-section.

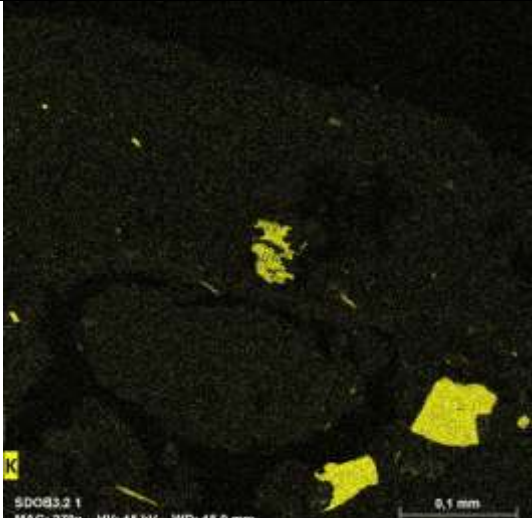


Figure 77. EDS map of distribution of potassium in the cross-section.

STUDEC	St. Nicholas Church, Doboszowice	2024/2025
		<b>SDOB 3</b>

Table 11. Description of the layers observed in the cross-section SDOB 3.1.

Layer No	Description
1	Ochre plaster with low magnesium content (below 2%)
2	White limewash with dolomitic magnesium content of ca. 2%
3	White limewash

Table 12. Results of EDS measurements expressed in oxides and normalised to 100%.

Spectrum No	MgO	SO <sub>3</sub>	CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Interpretation
10	14.9		83.5	1.6		Layer 1. BRP. Particle of dolomitic lime
9	5.6		93.1	1.3		Layer 1. BRP. Particle of dolomitic lime
8	1.6		95.8	1.9	0.8	Layer 1. BRP. Lime with low Mg content
7	1.5		96.8	1.8		Layer 1. BRP. Lime with low Mg content
6			100.0			Layer 3. Matrix. Pure calcitic lime
5	3.9		88.8	5.6	1.7	Layer 2: Matrix. Mg-rich lime
4	35.3	0.9	62.0	1.8		Layer 2. Particle of dolomitic lime
3	47.3		47.0	5.7		Layer 2. Particle of dolomitic lime
2	1.4		97.4	1.3		Layer 1. Matrix. Lime with low Mg content
1	1.1		97.9	1.0		Layer 1. Matrix. Lime with low Mg content

STUDEC	St. Nicholas Church, Doboszowice	
		<b>Conclusions</b>

## Conclusion

There are clear differences in the composition of the plaster from the chapel and from the presbytery.

Stucco finishing plaster (SDOB1) is built of angular and poorly rounded sand grains with some iron-rich inclusions, big white binder raletad particles, and matrix with Mg-rich lime. The plaster has been coated with a lime wash.

The background of the stucco decoration (SDOB2) is made of similar plaster but with bigger part of rounded sand grains. On top of it, there is a pink lime-based paint layer (with iron oxide as colourant). Lack of interfaces between the plaster and paint layer shows a fresco or semi-fresco technique was used.

The plaster in the presbytery (SDBO3) has an ochre matrix colour with large calcareous particles, some BRP, and large angular sand grains. It is heavily cracked. The green paint layer lays on 3 limewashes and contains almost no calcium, which suggests the use of an organic binder. The elemental composition of the green paint layer indicates copper carbonate (malachite most probably) with minor quantity of copper sulphate (such as brochantite, for instance) was used. In case of a further research the Raman spectroscopy can be used for identification of mineral phases of the Cu pigment.

STUDEC	St. Nicholas Church, Doboszowice	
		<b>List of analyses</b>

## List of analyses

	makrofoto	mikrofoto	Cross-section	Thin section	TA	XRD	Rozpuštění HCl	Organika	Mikrosonda nátěru	Další
SDOB 1.1		✓	✓	-	-	-	-	-	✓	-
SDOB 1.2		✓	✓	-	-	-	-	-	✓	-
SDOB 2A		✓	✓	-	-	-	-	-	✓	-
SDOB 2B		✓	✓	-	-	-	-	-	✓	-
SDOB 2C		✓	✓	-	-	-	-	-	✓	-
SDOB 2D		✓	-	-	-	-	-	-	-	-
SDOB 3.1		✓	✓	-	-	-	-	-	✓	-
SDOB 3.2		✓	✓	-	-	-	-	-	✓	-