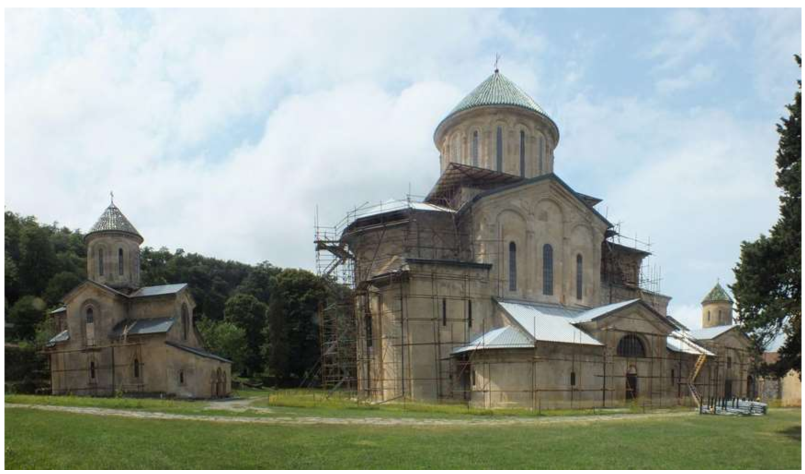
Safeguarding and conservation of Gelati Monastery World Heritage Property, Georgia

Agreement between LEPL "National Agency for Cultural Heritage Preservation of Georgia" and "ReStruere ltd, Florence University spin-off"



PHASE 1
Developing of knowledge for diagnostic purposes

Tests on material samples taken on-site
Scientific report explaining the results of the tests

Deliverable 1.1

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SUMMARY

I. Mineralogical-petrographic characterization of mortars, stones and tiles and chemical characterization of insulating materials from Gelati Monastery (Georgia)

II. Comment on the results of Laboratory analyses

MINERALOGICAL-PETROGRAPHIC CHARACTERIZATION OF MORTARS, STONES AND TILES AND CHEMICAL CHARACTERIZATION OF INSULATING MATERIALS FROM GELATI MONASTERY (GEORGIA)

TECHNICAL REPORT

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Rl e	Saggio Rl	Remaining part to simulate a stone (now disappeared or reduced)	
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R2 b	Saggio R2	Insulating, connection between roof and wall	
R3 a	Saggio R3	Mortar	
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T (red) 1 a	Provided old tile - Xll sec		
T (red) 1 b	Provided old tile - Xll sec		
T (red) 2	Interior old tile - XVI sec		
T (white)	Provided new white tile		
M.Xll-a	Provided, from Xll tile mortar Xll sec		
M.Xll-b	Provided, from Xll tile mortar, to check if Xll sec		
M.Xll-b	Provided, from Xll tile mortar, to check if Xll sec		glaze
M.Xll-c	Provided mortar, to check if Xll sec		
M.XVI	Provided mortar XVI sec		

Analytical methods

The following analysis has been performed:

- determination of the mineralogical composition through X ray diffraction (XRD) (X'Pert PRO diffractometer by PANalytical equipped with X'Celerator detector and HighScore software for acquisition and interpretation of data according to the following operative conditions: CuK α1= 1.545Å radiation, 40 KV, 30 mA, 2Θ = 3-70°);

- observations at the optical microscope in transmitted polarised light on thin section;

- samples R2b, R3b, R3c, R3d and S2 were subjected also to Fourier Transform Infrared Spectroscopy (FTIR) in order to investigate the nature of the organic compounds. The analyses were performed using an ALPHA Bruker spectrometer with SiC Globar source and DTGS detector, using the Platinum single reflection diamond module for attenuated total reflectance (ATR). Operating conditions: spectral range 4000-400 cm^{-1}, resolution 4 cm^{-1}, 24 scans. The spectra were processed with OPUS 7.2 software.

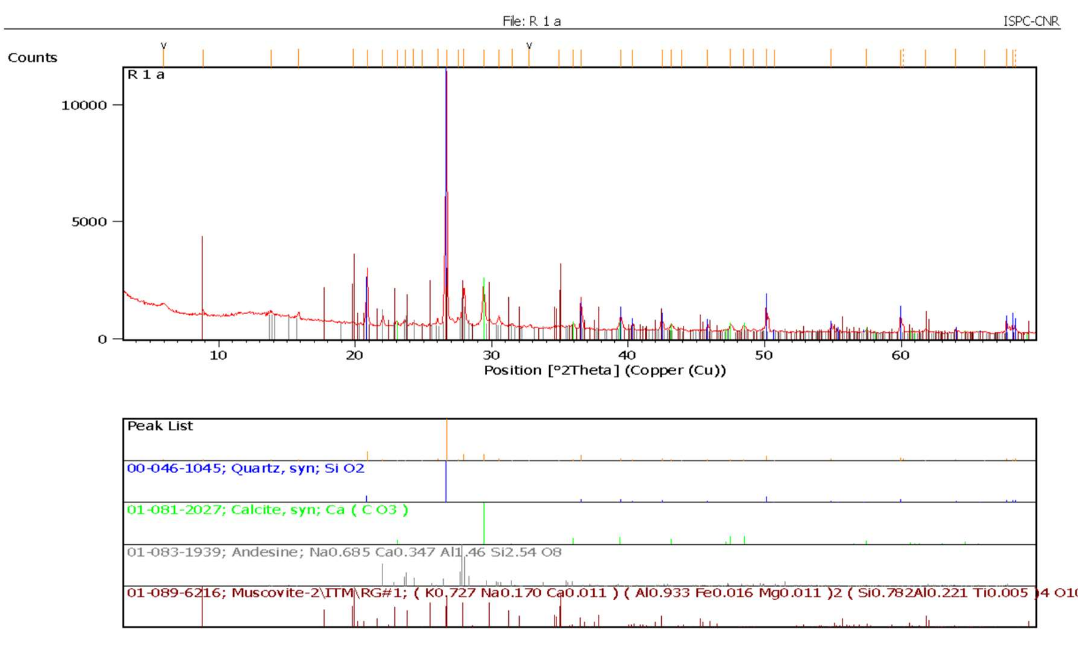
Results: mineralogical and petrographical analyses

R1 a   Saggio Rl   Tile filling mortar

R1 a - Cross section image

X ray diffraction analysis

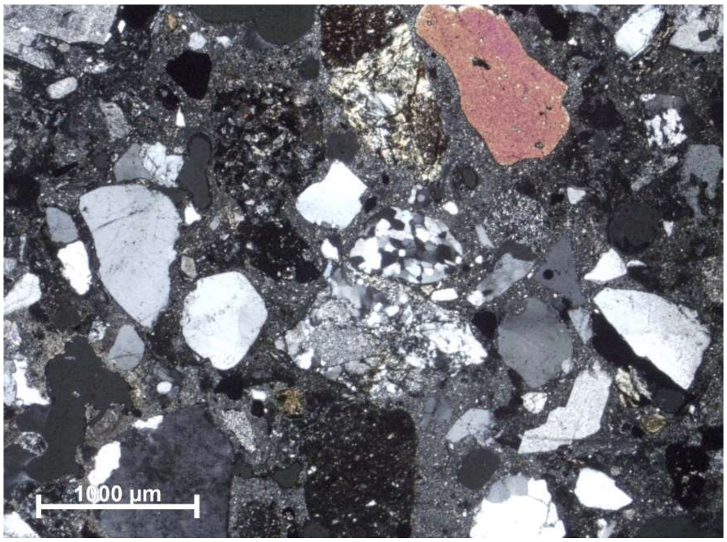
The mortar is made of calcite, quartz, micas (muscovite), feldspars (andesine).



R1 a - XRD diffraction pattern

Petrographic observation in thin section

The mortar shows a scarce binder (Binder/Aggregate ~ 1/3) with a micritic texture and a pure aspect of possible hydraulic nature. The aggregate is made of quartz, feldspars, micas, fragments of porphyric and granitic rocks. The grains are subrounded in shape with a bimodal grain-size distribution (400-500 µm and 1.5-2 mm). The macropores are subrounded in shape.



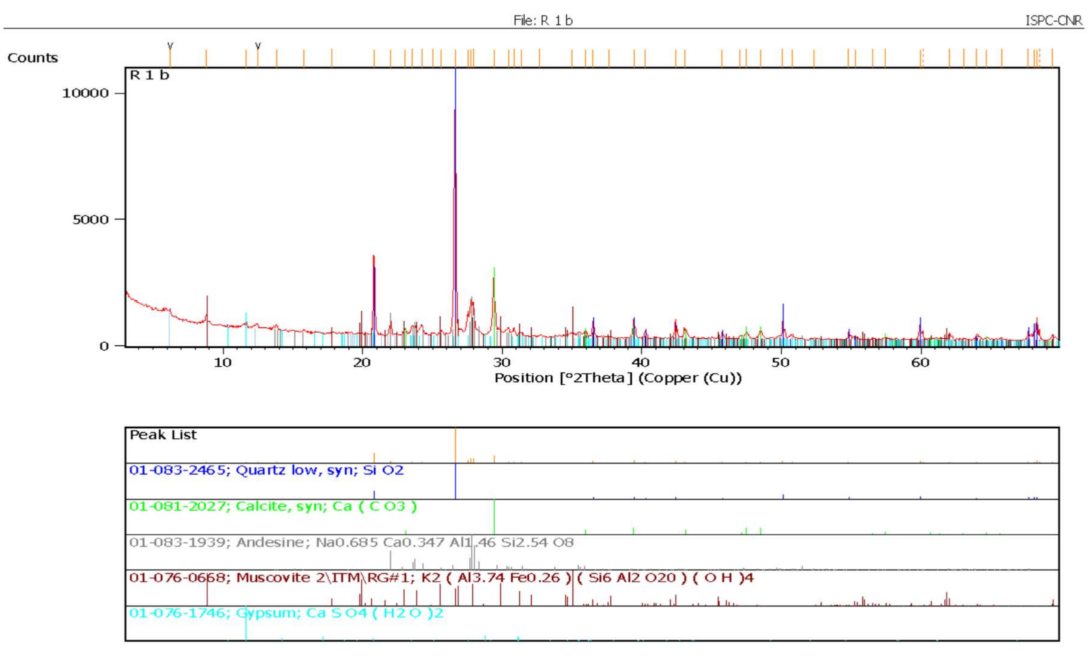
R1 a - Thin section image, transmitted light, crossed nicols

R1 b   Saggio R1   Mortar

R1 b - Cross section image

<u>*X ray diffraction analysis*</u>

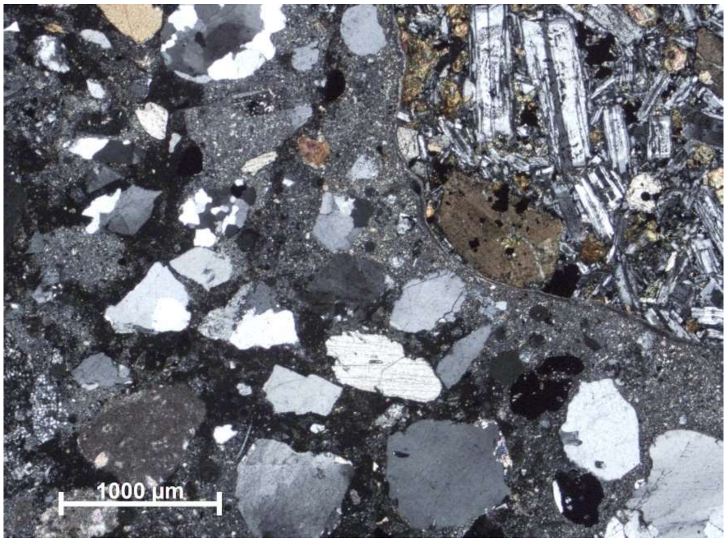
The mortar is made of calcite, quartz, micas (muscovite), feldspars (andesine) with traces of gypsum.



R1 b - XRD diffraction pattern

<u>Petrographic observation in thin section</u>

The mortar shows a scarce binder (Binder/Aggregate ~ 1/3) with a micritic texture and a pure aspect of possible hydraulic nature. The aggregate is made of quartz, feldspars, micas, fragments of porphyric and granitic rocks. The grains are subrounded in shape with a bimodal grain-size distribution (400-600 µm and 1.5–2.5 mm). The macropores are subrounded in shape.



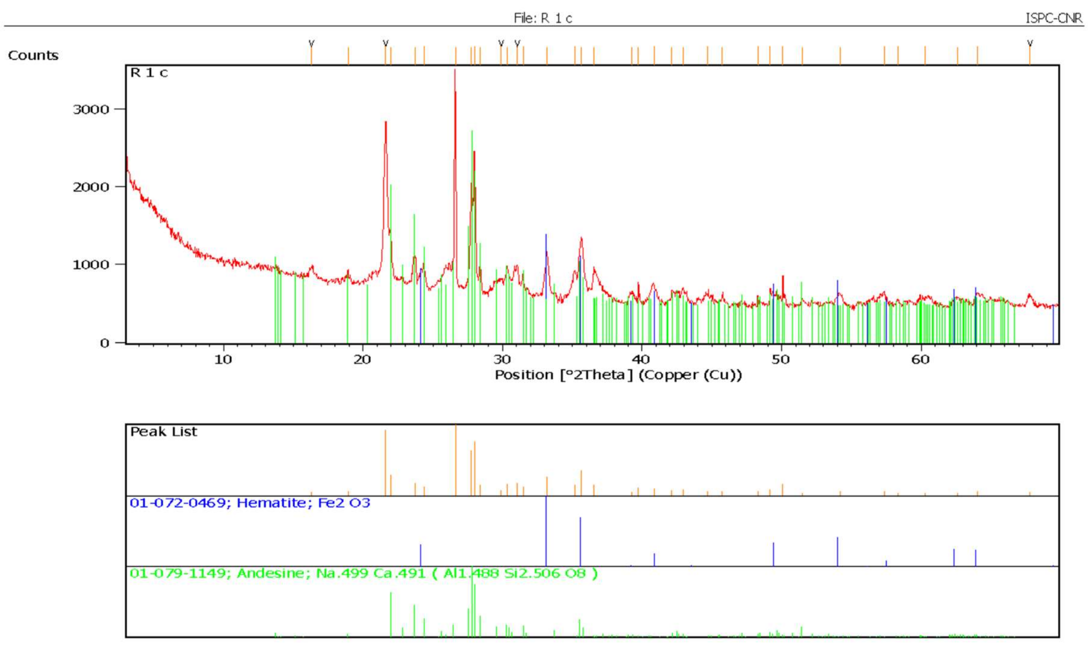
R1 b - Thin section image, transmitted light, crossed nicols

Rl c   Saggio Rl   New red tile

R1 c - Cross section image

<u>*X ray diffraction analysis*</u>

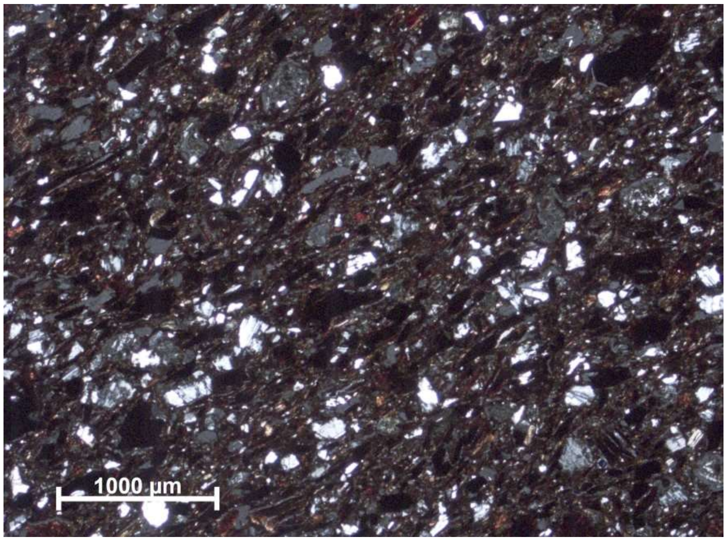
The crystalline phases present in the tile are andesine (feldspar), hematite and traces of quartz



R1 c - XRD diffraction pattern

Petrographic observation in thin section

The tile shows a slightly birefringent groundmass rich in bonherz (iron rich lumps). The framework is abundant, made of feldspars and scarce quartz with an unimodal grain size distribution (100 - 300 µm). The grains have an angular shape. The macropores show an elongated shape.



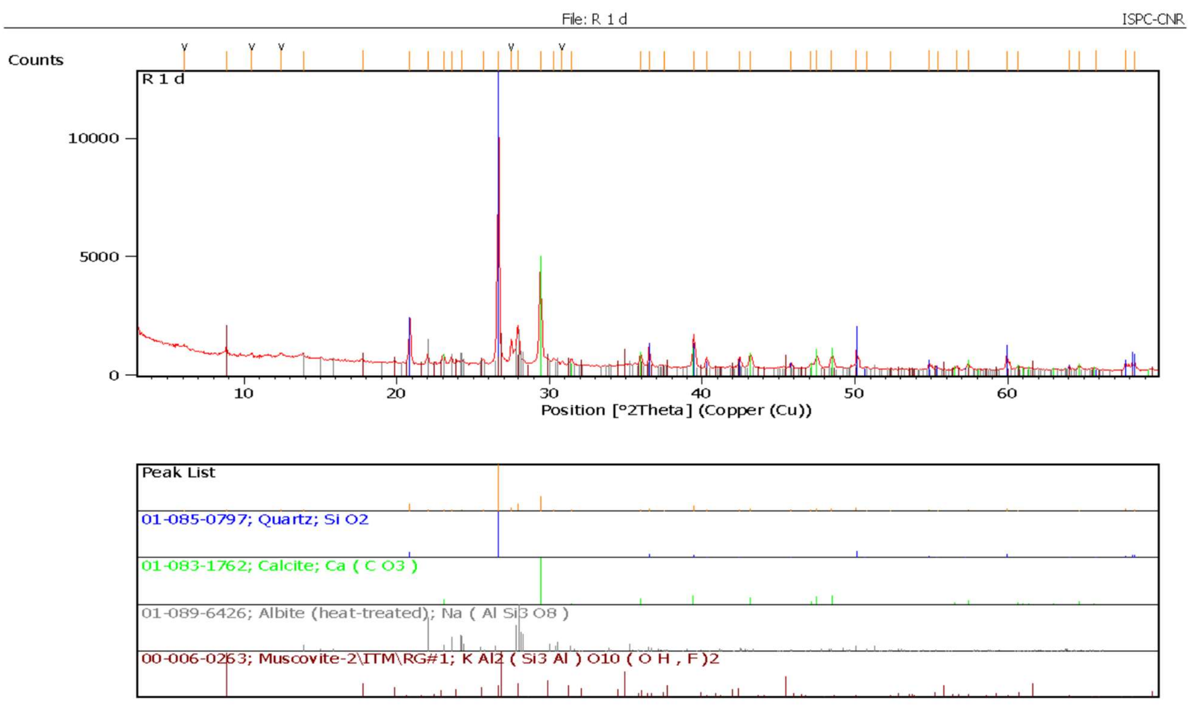
R1 c - Thin section image, transmitted light, crossed nicols

Rl d Saggio R1 Mortar, connection between roof tile and wall

R1 d - Cross section image

X ray diffraction analysis

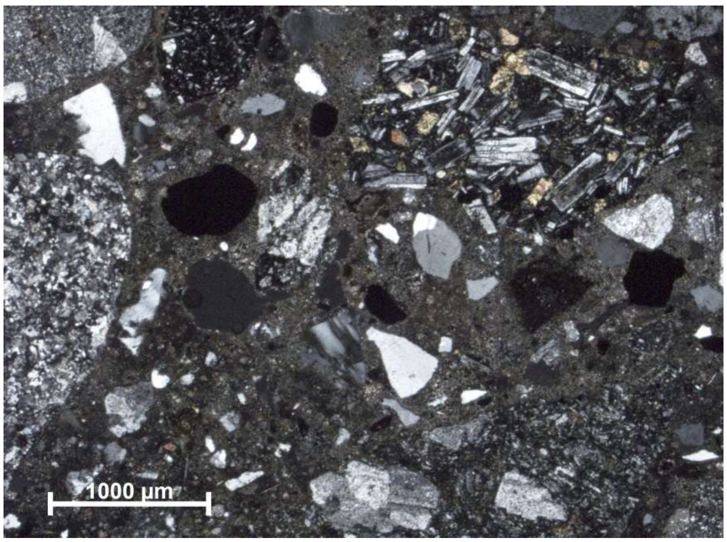
The mortar is made of calcite, quartz, micas (muscovite), feldspars (albite).



R1 d - XRD diffraction pattern

Petrographic observation in thin section

The mortar shows a scarce binder (Binder/Aggregate ~ 1/3) with a micritic texture and impure aspect of possible hydraulic nature. The aggregate is made of quartz, feldspars, micas, fragments of porphyric, granitic and basaltic rocks. The grains are subrounded in shape with a bimodal grain-size distribution (200-400 µm and 1–2 mm). The macropores are irregular in shape.



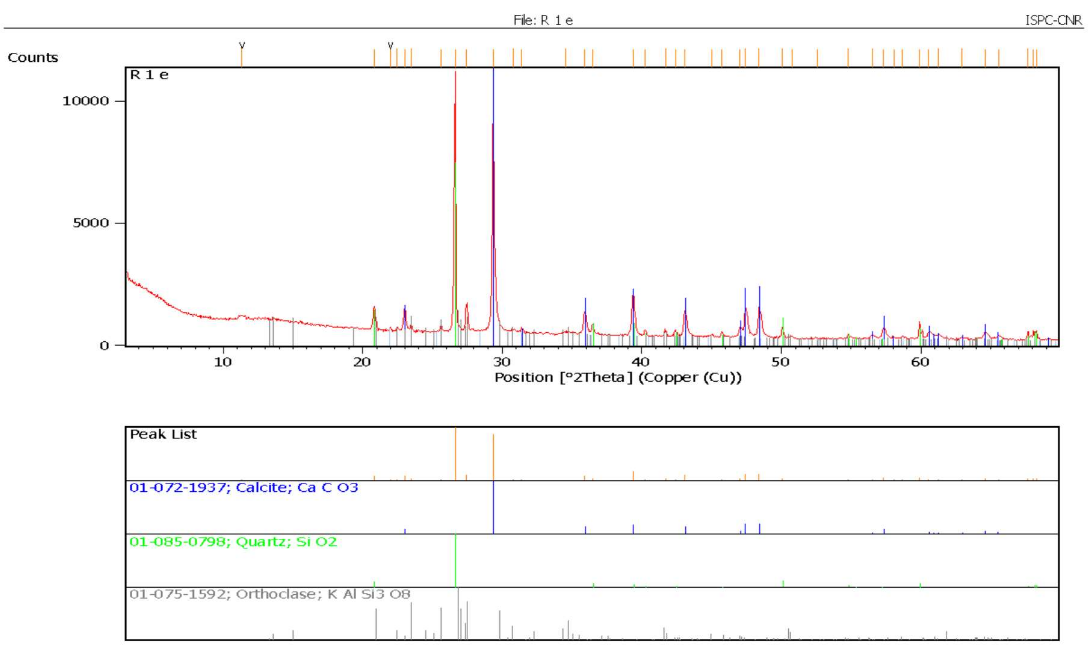
R1 d - Thin section image, transmitted light, crossed nicols

Rl e   Saggio Rl   Campione parte restante per simulare pietra

R1 e - Cross section image

X ray diffraction analysis

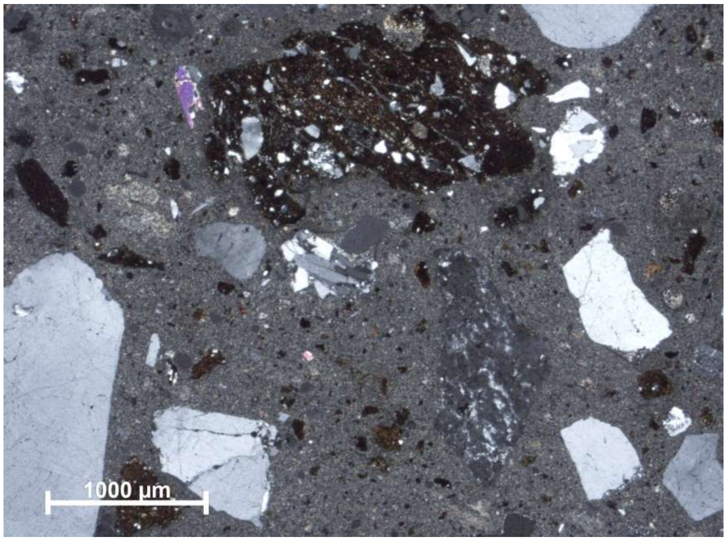
The material is constituted by calcite followed by quartz and feldspars (orthoclase)



R1 e - XRD diffraction pattern

Petrographic observation in thin section

The mortar shows an abundant binder (Binder/Aggregate 1/1-1/2) made of air hardening lime with a micritic texture. The aggregate is made of brick fragments, quartz, feldspars. The grains are angular in shape with a bimodal grain-size distribution (100-300 µm and 800 µm –1.5 mm). Rare lime lumps are present. The macropores are sub-spherical in shape.



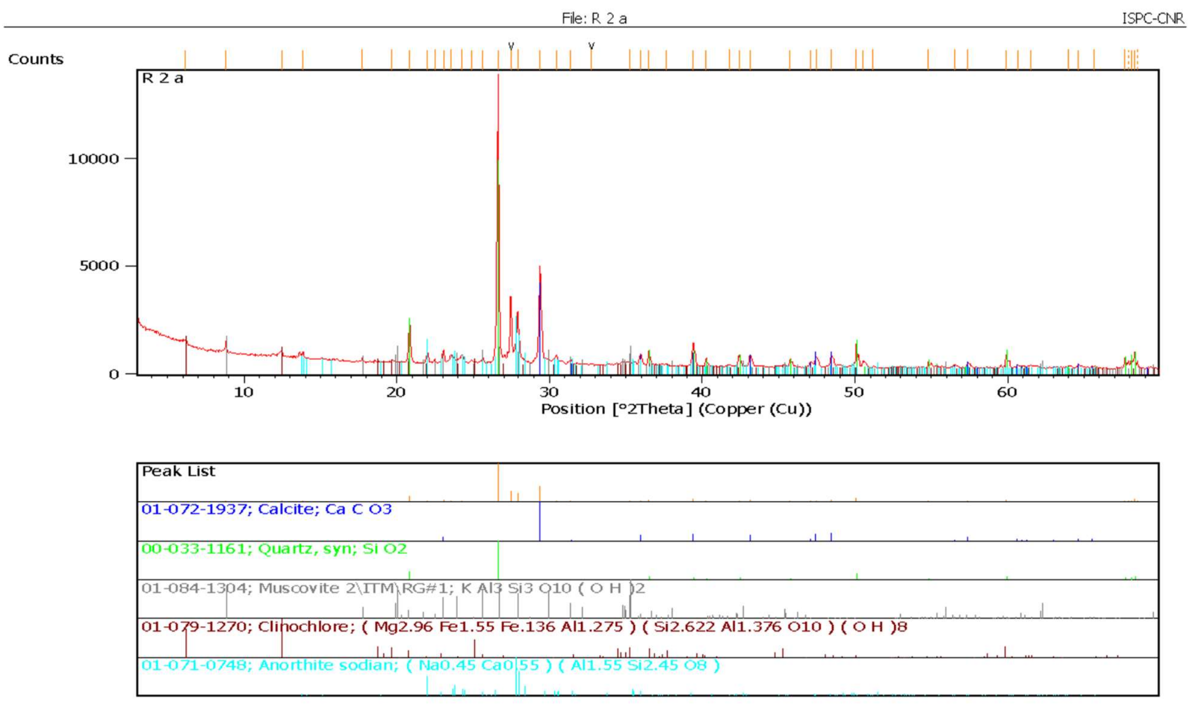
R1 e - Thin section image, transmitted light, crossed nicols

R2 a   Saggio R2   Mortar, connection between roof tile and wall

R2 a - Cross section image

X ray diffraction analysis

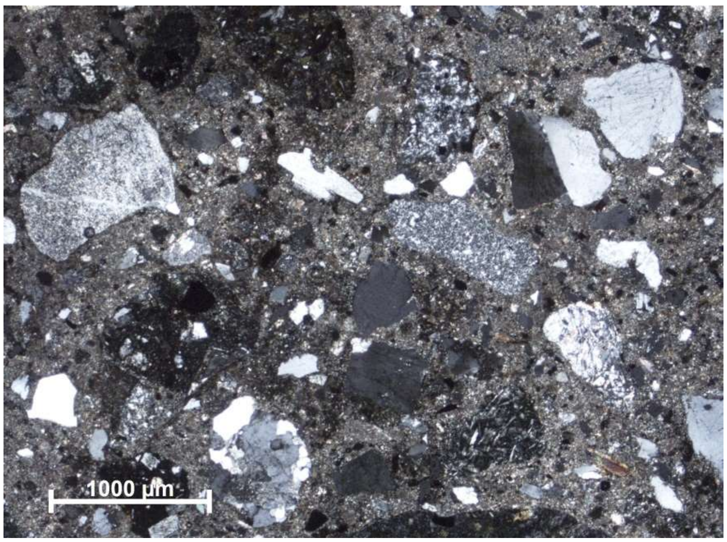
The mortar is made of calcite, quartz, micas (muscovite), feldspars (anorthite), clinochlore



R2 a - XRD diffraction pattern

Petrographic observation in thin section

The mortar shows a quite abundant binder (Binder/Aggregate 1/2- 1/3) with a microsparitic texture and impure aspect of possible hydraulic nature. The aggregate is made of quartz, feldspars, micas, fragments of sandstones and porphyric, granitic and basaltic rocks. The grains are angular in shape with a bimodal grain-size distribution (200-300 µm and 1–1.5 mm). The macropores are sub-spherical in shape.



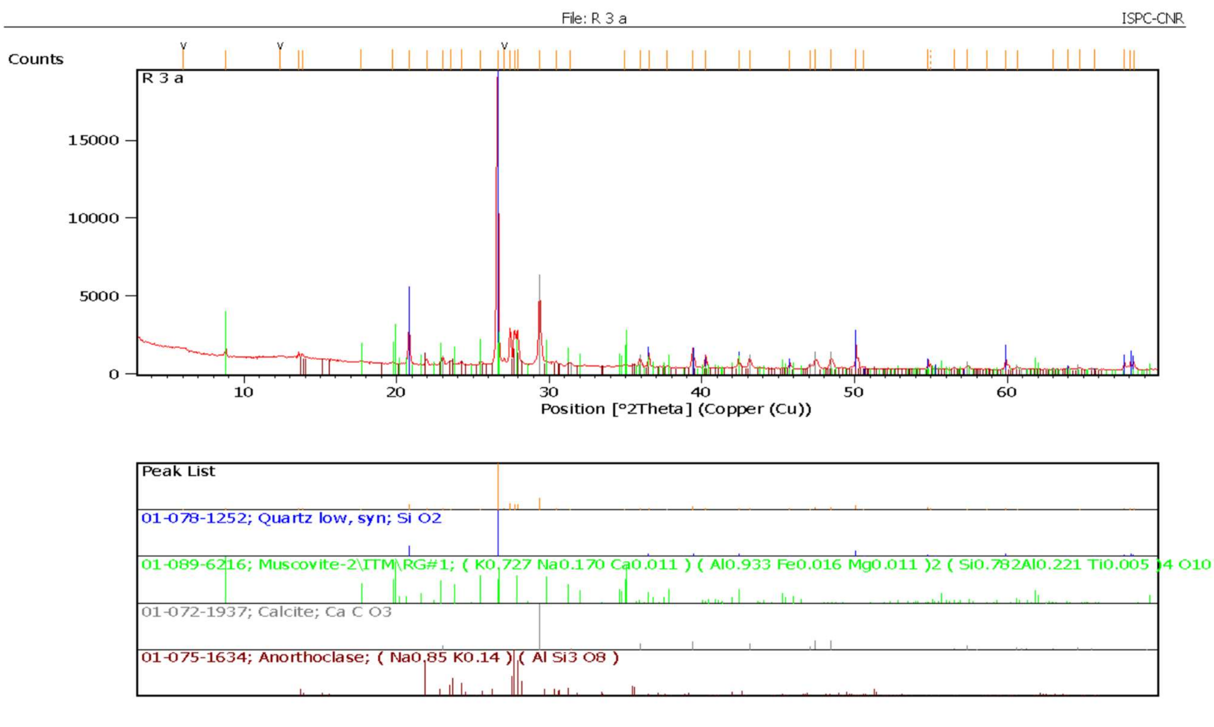
R2 a - Thin section image, transmitted light, crossed nicols

R3 a   Saggio R3   Mortar

R3 a - Cross section image

<u>*X ray diffraction analysis*</u>

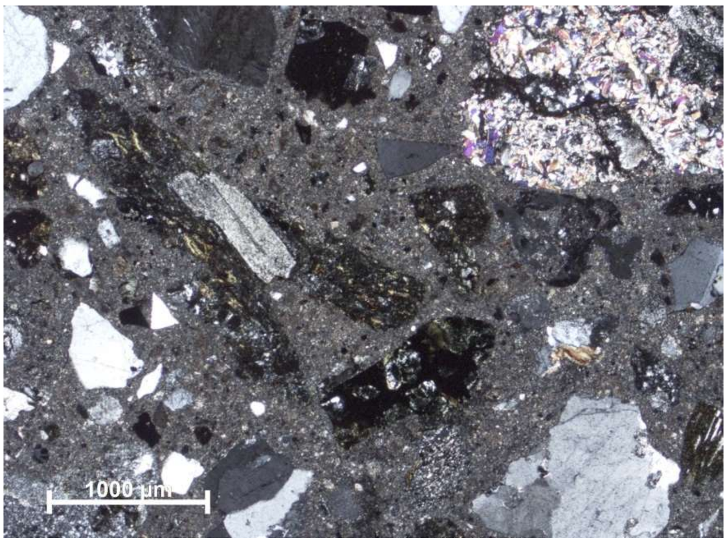
The mortar is made of calcite, quartz, micas (muscovite), feldspars (anorthoclase)



R3 a - XRD diffraction pattern

Petrographic observation in thin section

The mortar shows a quite abundant binder (Binder/Aggregate 1/2- 1/3) with a microsparitic texture and impure aspect of possible hydraulic nature.. The aggregate is made of quartz, feldspars, micas, fragments of sandstones and porphyric, granitic and basaltic rocks. The grains are angular in shape with a bimodal grain-size distribution (300-400 µm and 1–1.5 mm). The macropores are sub-spherical in shape.



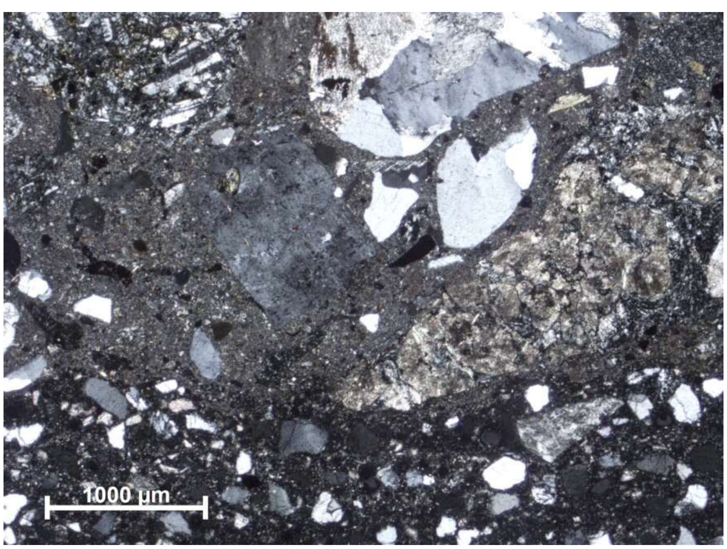
R3 a - Thin section image, transmitted light, crossed nicols

R3 b Saggio R3 Insulating, connection between roof and wall

R3 b - Cross section image

Petrographic observation in thin section

The sample shows a darker portion constituted by a mortar with a quite abundant binder (Binder/Aggregate 1/2- 1/3) with a microsparitic texture and impure aspect of possible hydraulic nature. The aggregate is made of quartz, feldspars, micas, fragments of porphyric, granitic and basaltic rocks. The grains are angular in shape with a bimodal grain-size distribution (300-400 µm and 1–1.5 mm). The macropores are sub-spherical in shape.
The whitish portion shows a scarce organic binder (Binder/Aggregate ~ 1/3) and an aggregate made of quartz and feldspars. The grains are angular in shape with a unimodal grain size (150-200 µm).



R3 b - Thin section image, transmitted light, crossed nicols:

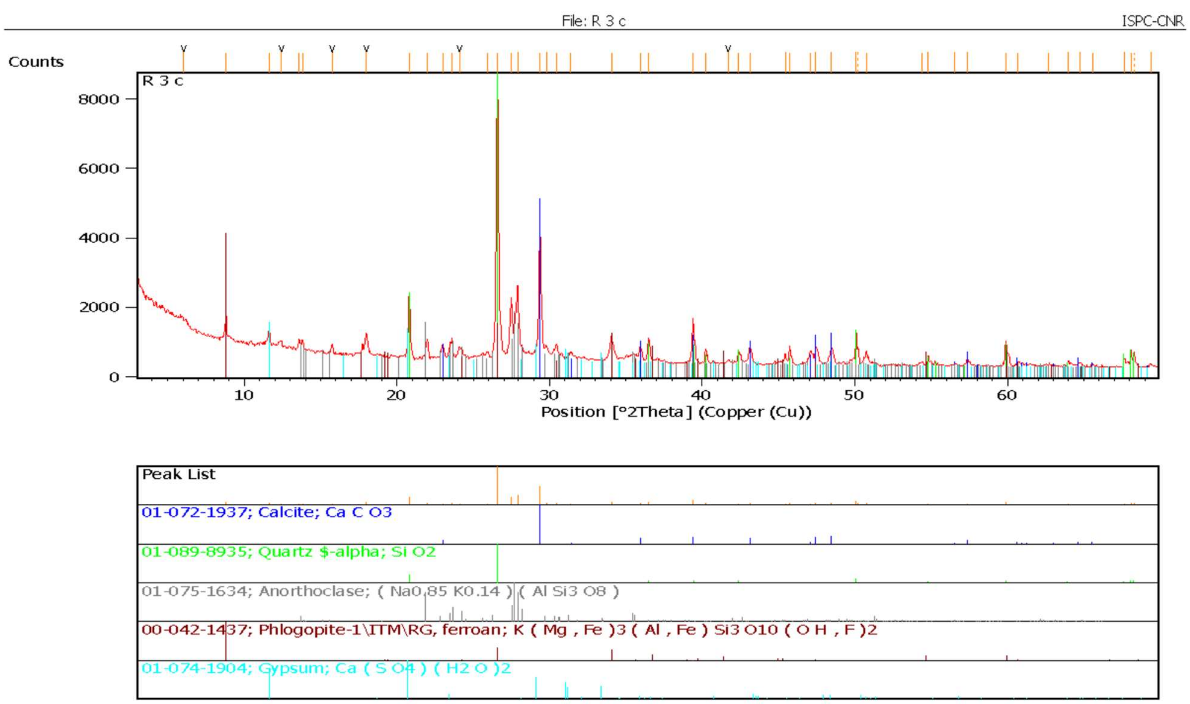
in the bottom is the whitish portion with the organic binder

R3 c   Saggio R3   Insulating, liquid   (malta + parte organica)

R3 c - Cross section image

X ray diffraction analysis

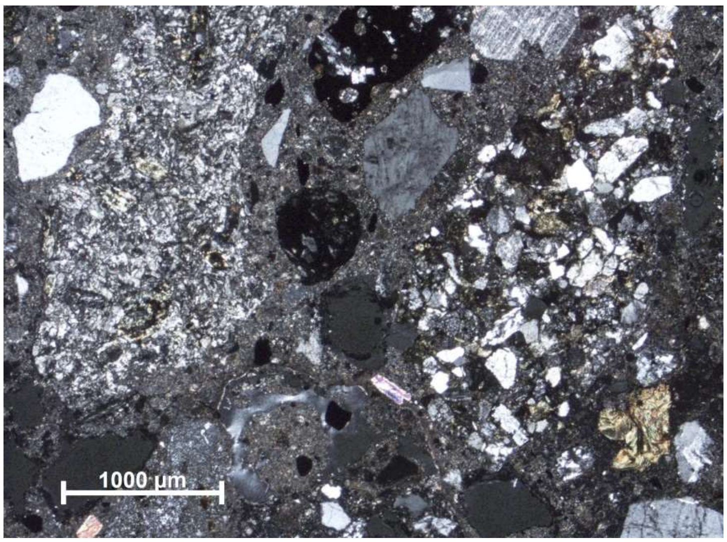
The mortar is made of calcite, quartz, micas (muscovite, phlogopite), feldspars (anorthoclase) with traces of gypsum



R3 c - XRD diffraction pattern

Petrographic observation in thin section

The mortar shows a quite abundant binder (Binder/Aggregate 1/2- 1/3) with a microsparitic texture and impure aspect of possible hydraulic nature. The aggregate is made of quartz, feldspars, micas, fragments of basaltic rocks, siltites and limestones. The grains are sub-rounded in shape with a bimodal grain-size distribution (300-400 µm and 1–1.5 mm). The macropores are sub-spherical in shape.



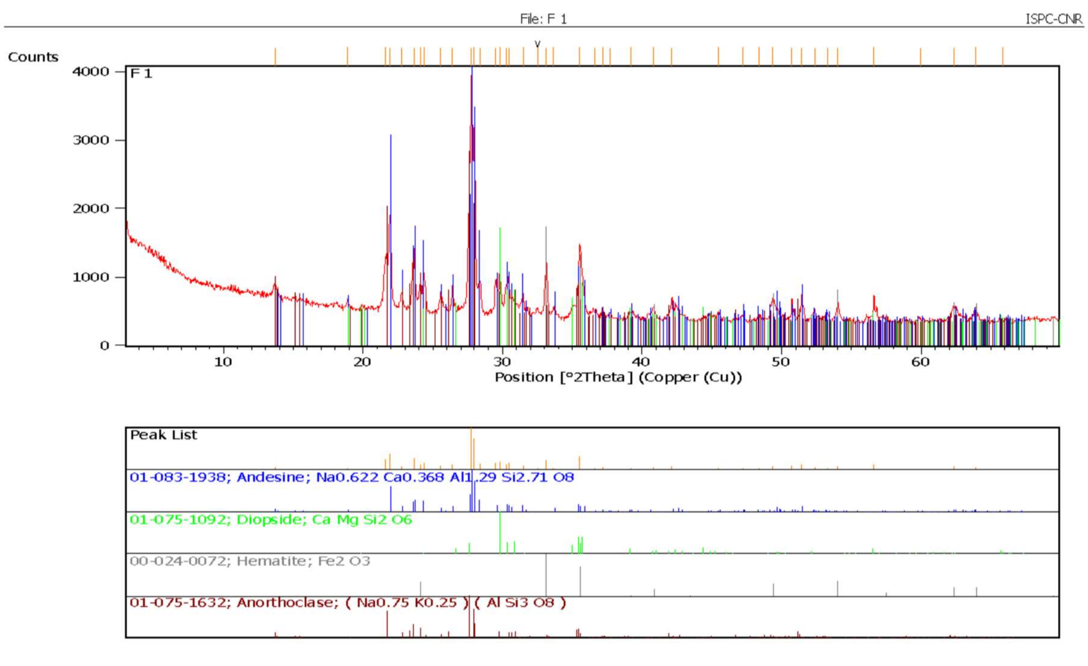
R3 c - Thin section image, transmitted light, crossed nicols

F1 ***Sottotetto Falda 22*** *Pumice, filling of the Roof #22*

F 1 - Cross section image

<u>*X ray diffraction analysis*</u>

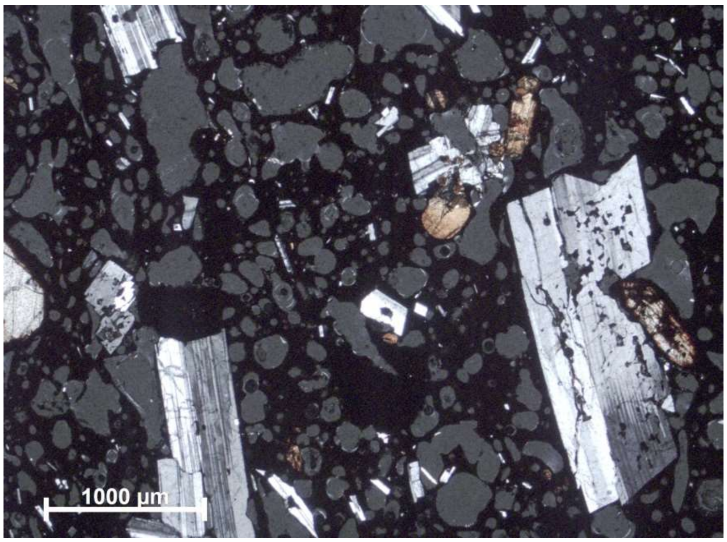
The crystalline phases of the rock are constituted by feldspars (andesine, anorthoclase), pyroxenes (diopside) and hematite



F 1 - XRD diffraction pattern

Petrographic observation in thin section

The rock is constituted by a completely vitreous groundmass and porphyroblasts with dimensions of 400-800 µm, made of prevailing feldspars (andesine, anorthoclase) and secondarily by pyroxenes. Suspheric pores are abundant with dimension of 400-500 µm.



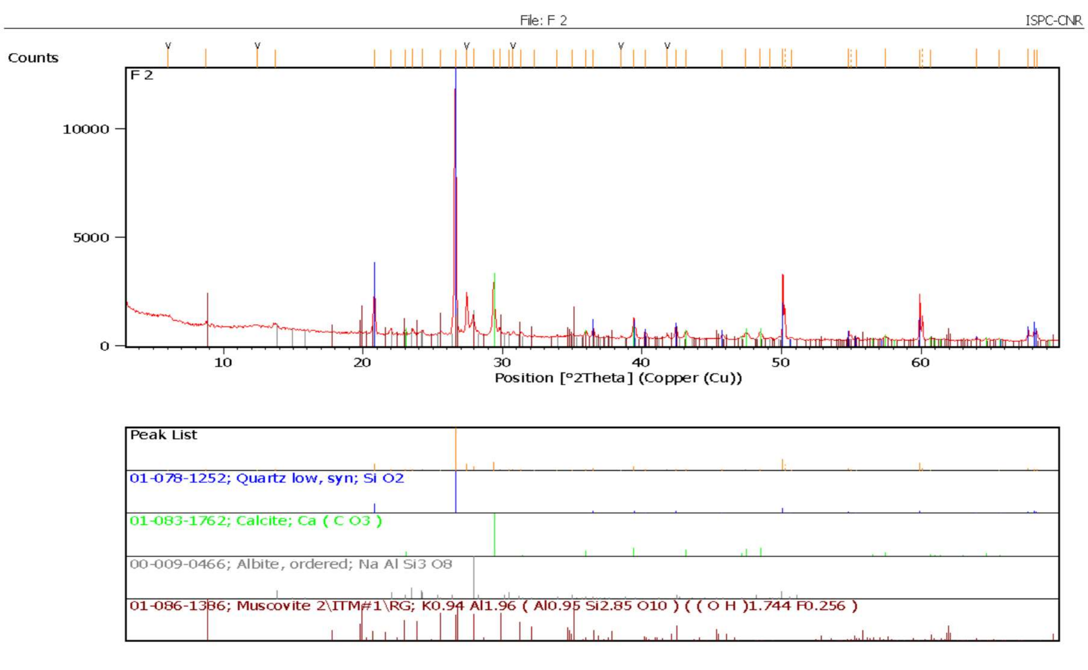
F 1 - Thin section image, transmitted light, crossed nicols

F2 Sottotetto Falda 22 Mortar (cement), filling of the Roof #22

F 2 - Cross section image

<u>*X ray diffraction analysis*</u>

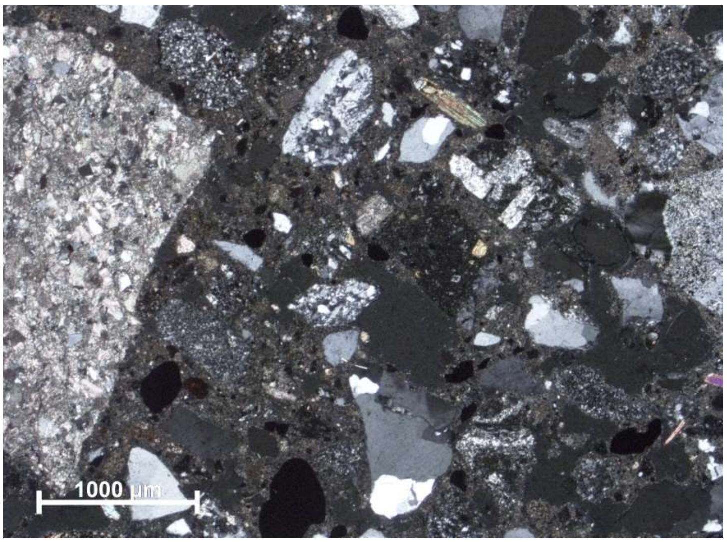
The mortar is made of calcite, quartz, micas (muscovite), feldspars (albite)



F 2 - XRD diffraction pattern

Petrographic observation in thin section

The mortar shows a quite abundant binder (Binder/Aggregate 1/2- 1/3) with a microsparitic texture and impure aspect of possible hydraulic nature. The aggregate is made of quartz, feldspars, micas, fragments of porphyric rocks, siltites and marble. The grains are sub-rounded in shape with a bimodal grain-size distribution (400-500 μm and 1–1.5 mm). The macropores are sub-spherical in shape



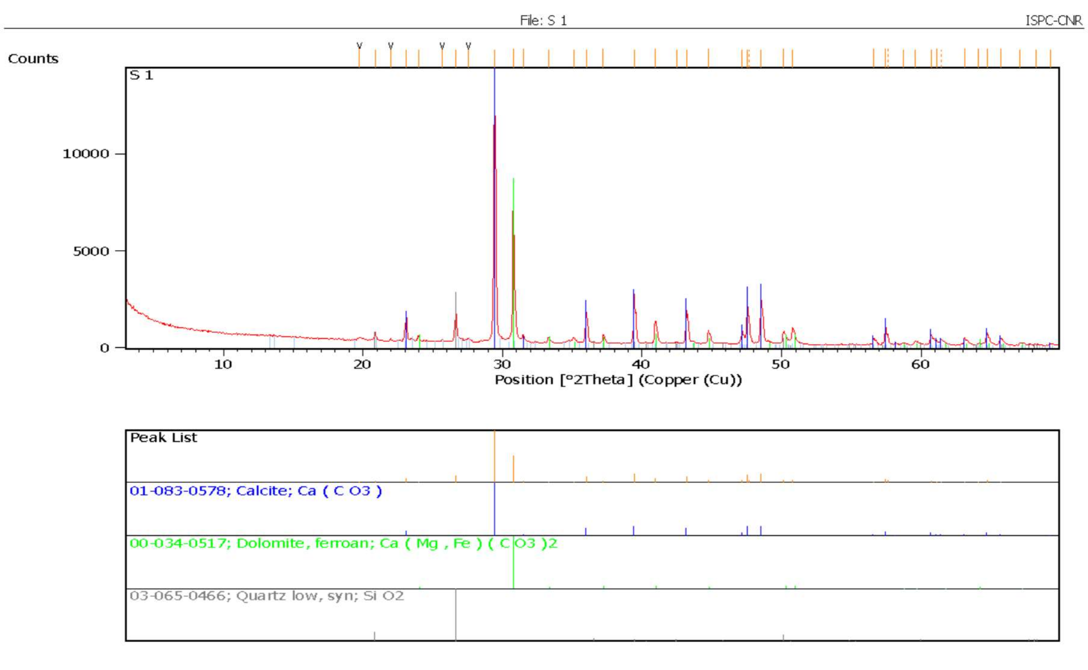
F 2 - Thin section image, transmitted light, crossed nicols

S1 Collected North side Wall stone

S 1 - Cross section image

X ray diffraction analysis

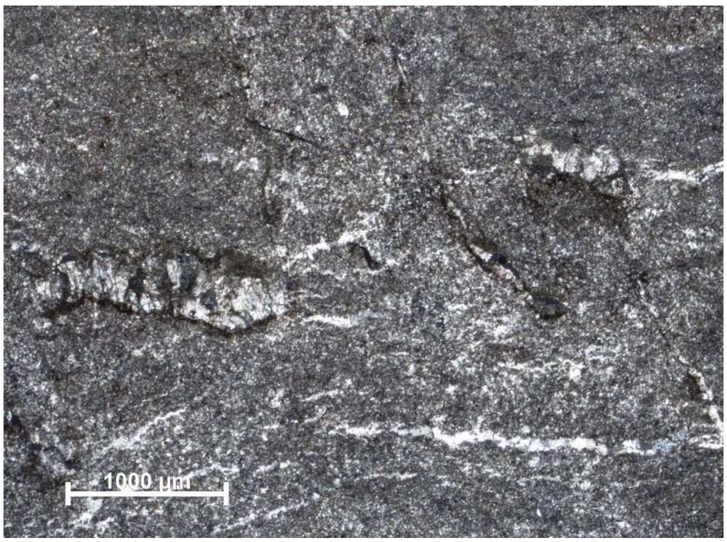
The stone is made of calcite, dolomite with a little amount of quartz. According to this composition it can be classified as dolomitic limestone



S 1 - XRD diffraction pattern

Petrographic observation in thin section

The stone shows a microsparitic texture with rare intraclasts and quartz grains. Rare calcite veins are present. The macropores are rare, with an elongated shape.
According to the petrographic aspect the rock can be classified as "intraclasts bearing micrite" (Dunham 1962) and mudstone (Folk 1959).
According to the mineralogical composition it can be classified as a dolomitic limestone



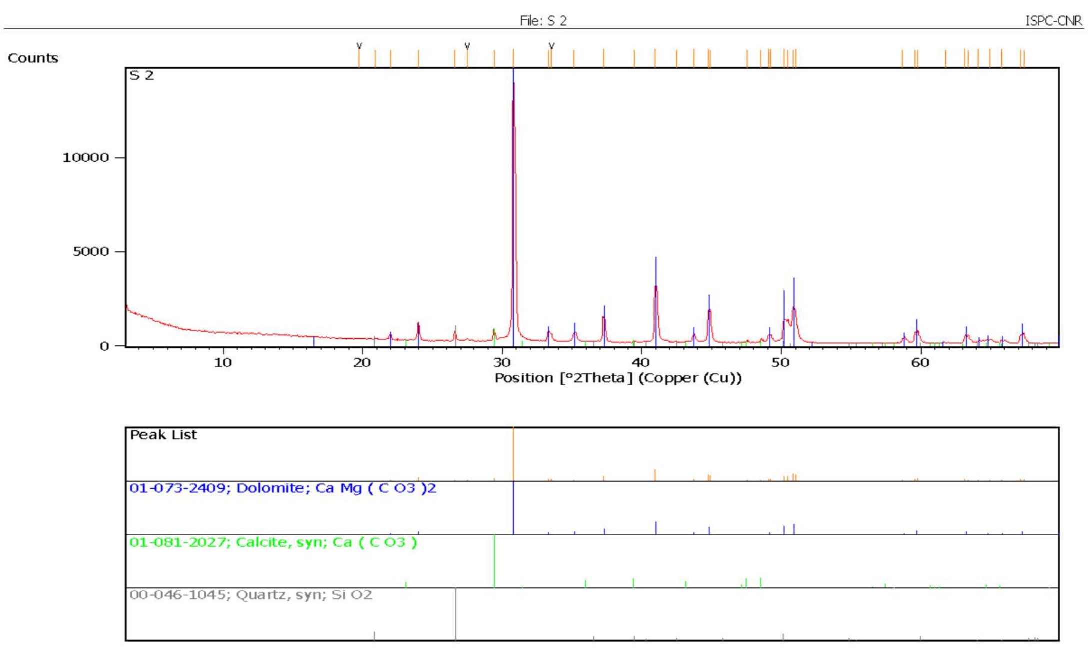
S 1 - Thin section image, transmitted light, crossed nicols

S2 Drum near R3 Wall stone


S 2 - Cross section image

X ray diffraction analysis

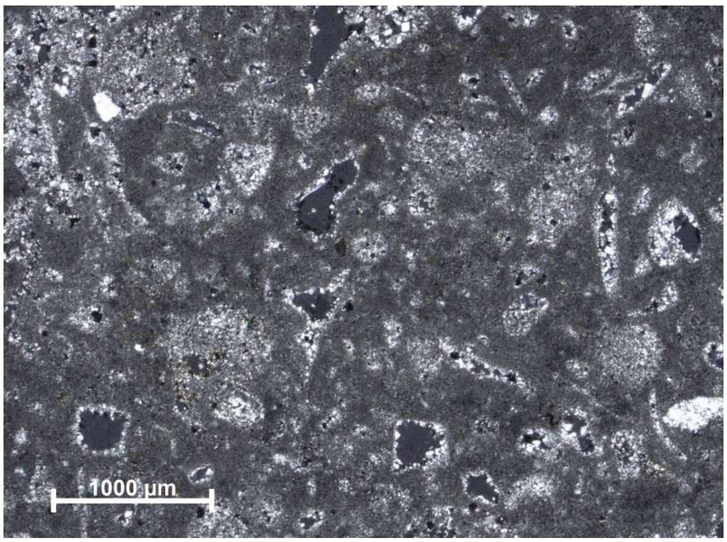
The stone is made of dolomite with a little amount of calcite and quartz. According to this composition it can be classified as dolomitic rock



S 2 - XRD diffraction pattern

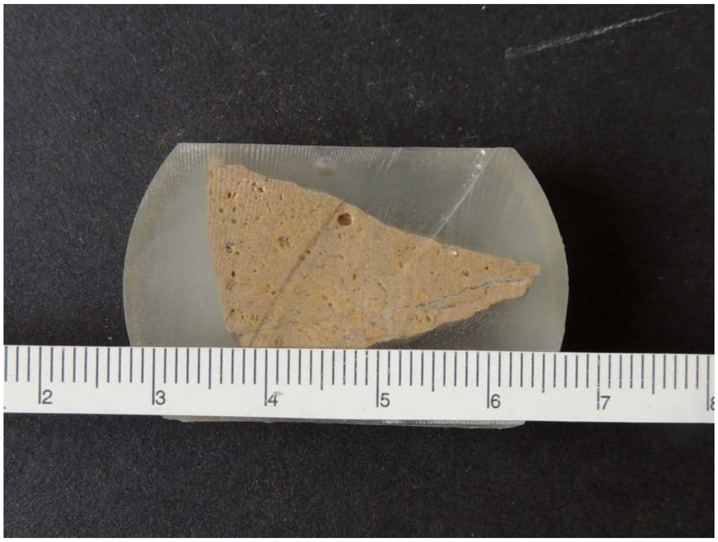
Petrographic observation in thin section

The stone shows a microsparitic texture with rare intraclasts and quartz grains. Macropores of subrounded shape are abundant with presence of dolomite crystallization.
According to the petrographic aspect the rock can be classified as "intraclasts bearing micrite" (Dunham 1962) and mudstone (Folk 1959).
According to the mineralogical composition it can be classified as a dolomite rock.



S 2 - Thin section image, transmitted light, crossed nicols

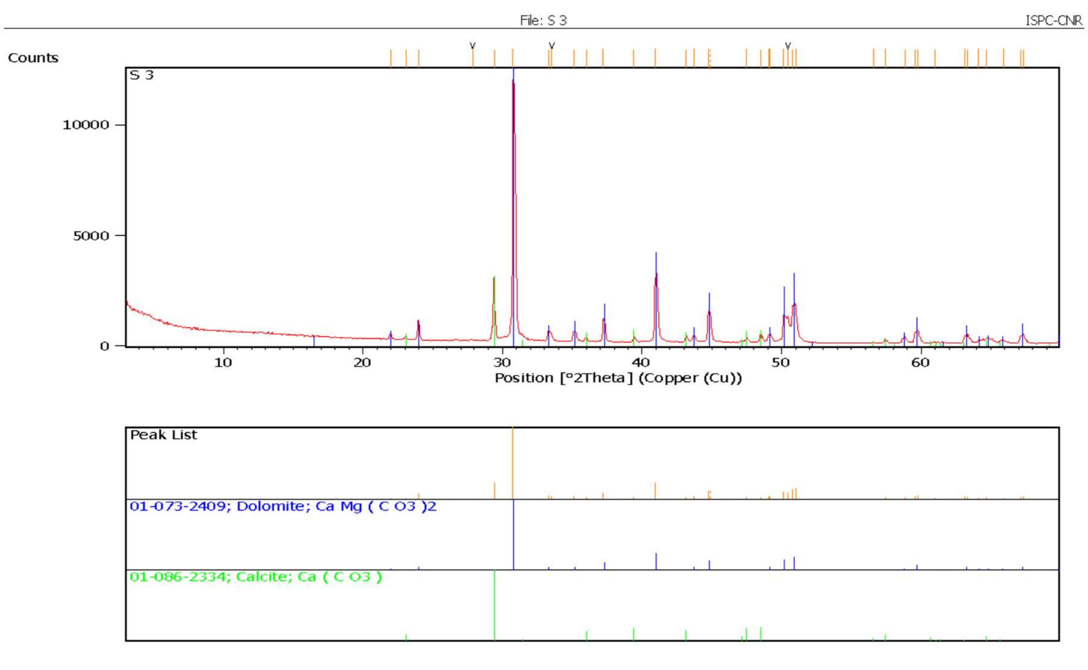
S3 West side Basement stone



S 3 - Cross section image

<u>*X ray diffraction analysis*</u>

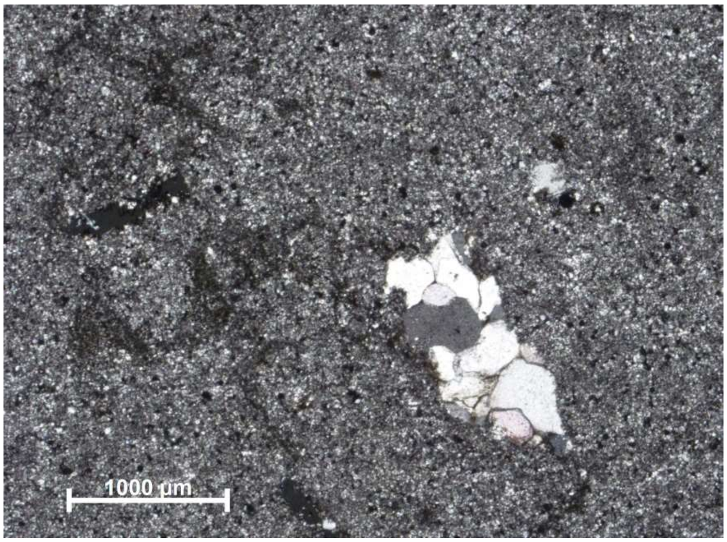
The stone is made of dolomite with a little amount of calcite and quartz. According to this composition it can be classified as dolomitic rock



S 3 - XRD diffraction pattern

Petrographic observation in thin section

The stone shows a microsparitic texture with rare intraclasts constituted by sparitic calcite with dimension of 400-600 µm. Calcite veins are present. Macropores are scarce. According to the petrographic aspect the rock can be classified as “intraclasts bearing micrite” (Dunham 1962) and mudstone (Folk 1959).
According to the mineralogical composition it can be classified as a dolomite rock



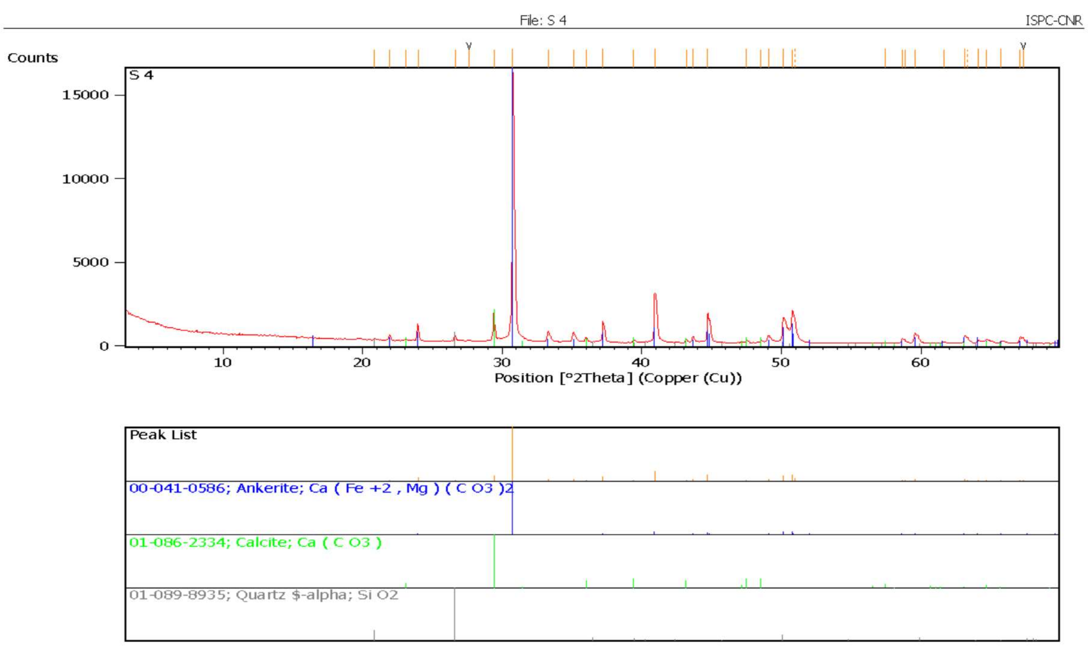
S 3 - Thin section image, transmitted light, crossed nicols

S4 Interior Wall Stone

S 4 - Cross section image

X ray diffraction analysis

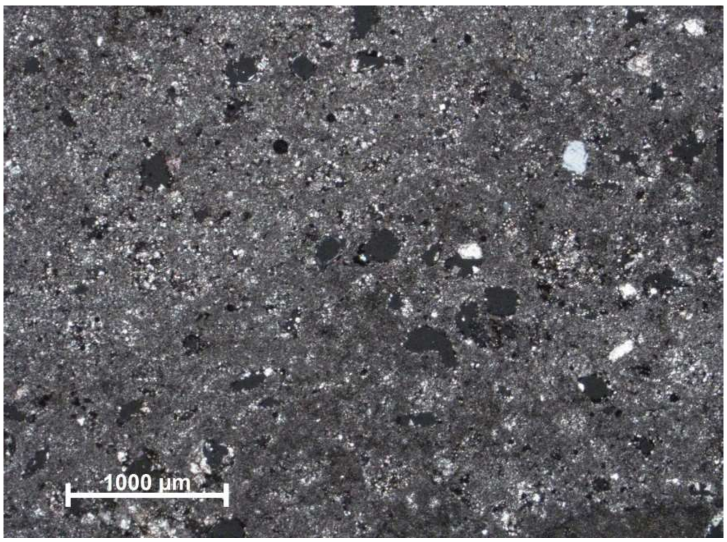
The stone is made of dolomite with a little amount of calcite and quartz. According to this composition it can be classified as dolomitic rock



S 4 - XRD diffraction pattern

Petrographic observation in thin section

The stone shows a microsparitic texture with rare intraclasts and quartz grains. Macropores of subrounded shape are abundant with presence of dolomite crystallization.
According to the petrographic aspect the rock can be classified as "intraclasts bearing micrite" (Dunham 1962) and mudstone (Folk 1959).
According to the mineralogical composition it can be classified as a dolomite rock.



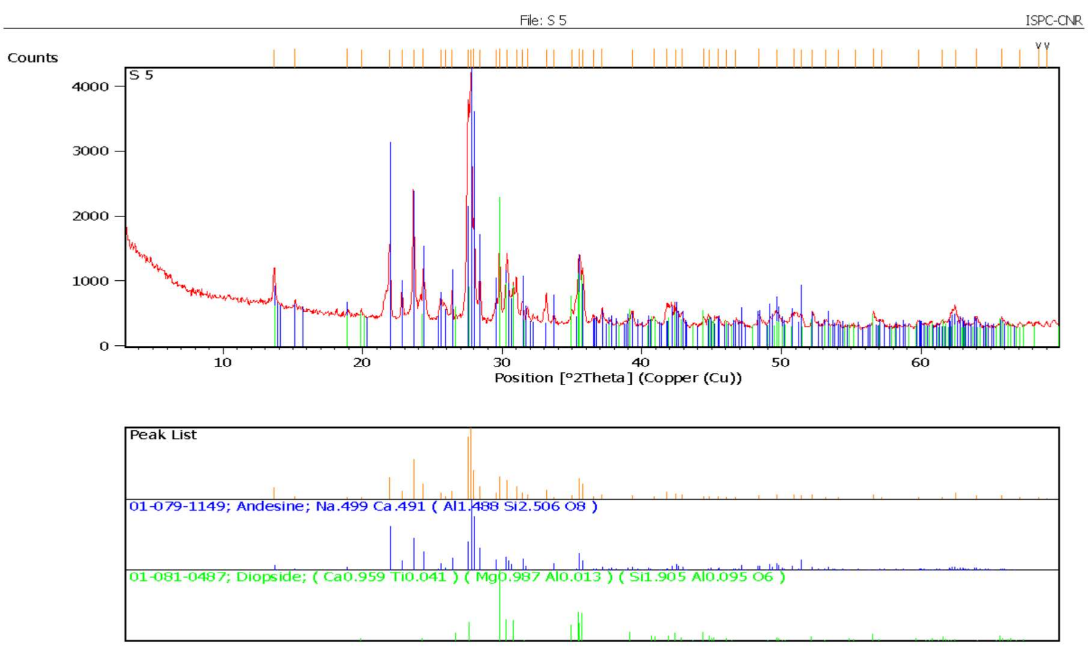
S 4 - Thin section image, transmitted light, crossed nicols

S5 Provided Stone slab roof stone

S 5 - Cross section image

X ray diffraction analysis

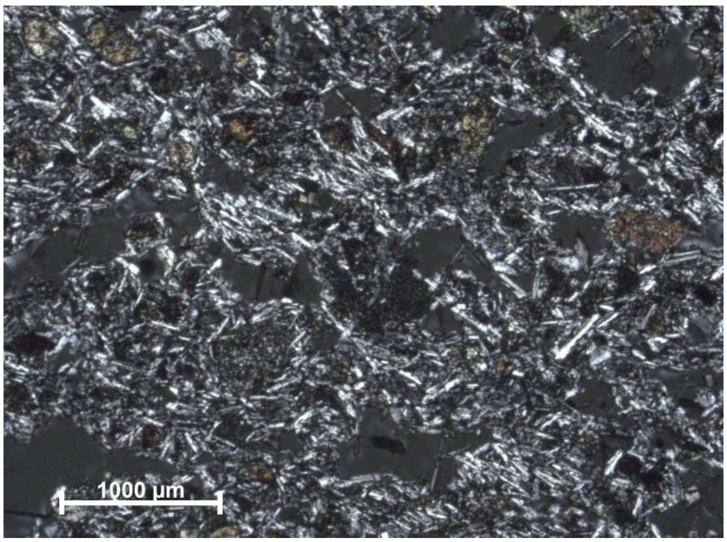
The crystalline phases present in the stone are feldspars (andesine) and pyroxenes (diopside). This stone can be referred to an effusive rock (andesite/basalt).



S 5 - XRD diffraction pattern

Petrographic observation in thin section

The stone shows a trachytic texture constituted by small aligned feldspars and phenocrysts constituted by pyroxenes. The macroporosity is abundant with pores of irregular shape. According to the mineralogical composition and petrographic aspect the rock can be referred to an effusive rock (andesite/basalt).



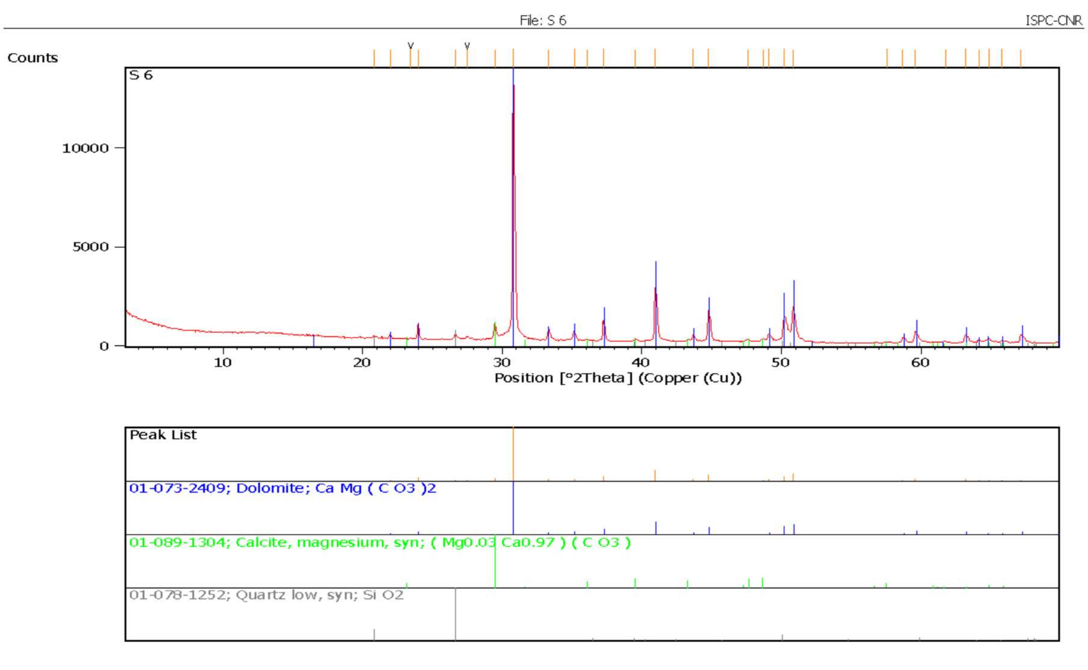
S 5 - Thin section image, transmitted light, crossed nicols

S6 Apse South-East side Wall stone

S 6 - Cross section image

X ray diffraction analysis

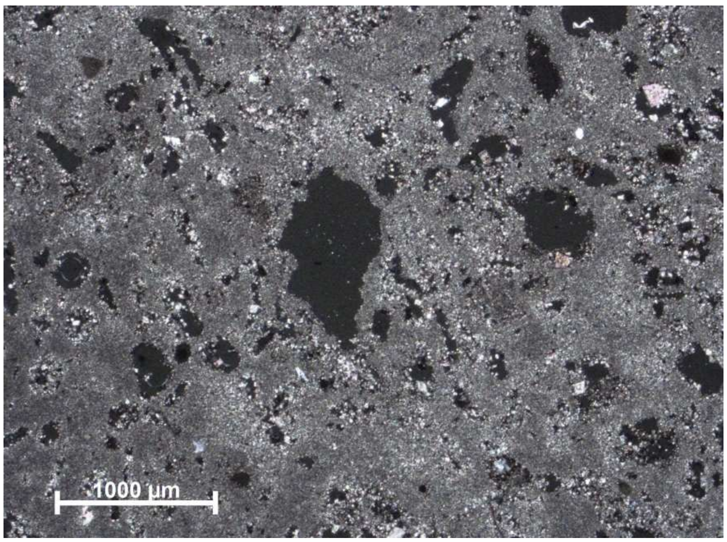
The stone is made of dolomite with a little amount of calcite and quartz. According to this composition it can be classified as dolomitic rock



S 6 - XRD diffraction pattern

Petrographic observation in thin section

The stone shows a microsparitic texture with rare intraclasts and quartz grains. Macropores of subrounded shape are abundant with presence of dolomite crystallization.
According to the petrographic aspect the rock can be classified as “intraclasts bearing micrite” (Dunham 1962) and mudstone (Folk 1959).
According to the mineralogical composition it can be classified as a dolomite rock



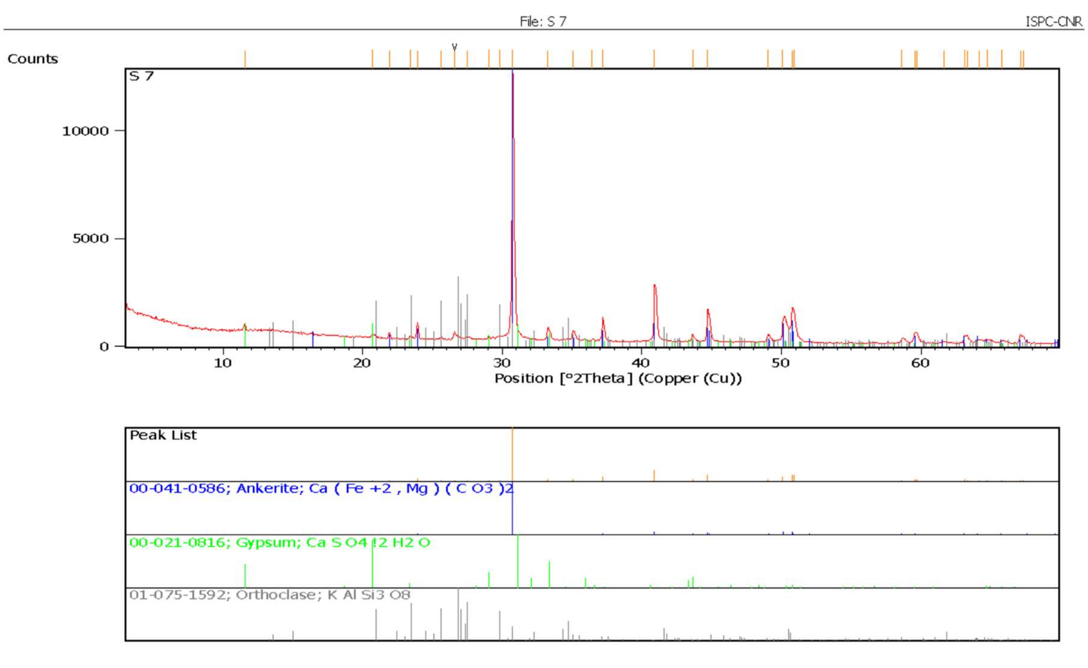
S 6 - Thin section image, transmitted light, crossed nicols

S7 Cornice North side Cornice stone

S 7 - Cross section image

X ray diffraction analysis

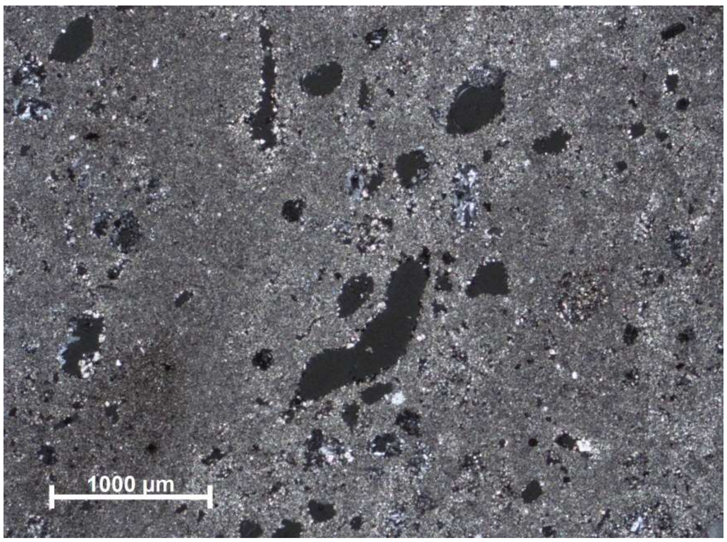
The stone is made of ankerite, with traces of feldspars (orthoclase) and gypsum. Ankerite [$Ca(Fe,Mg)(CO_3)_2$] is a dolomite where part of magnesium has been replaced by iron ferroan dolomite). According to this composition it can be still classified as dolomitic rock.



S 7 - XRD diffraction pattern

Petrographic observation in thin section

The stone shows a microsparitic texture with rare intraclasts and quartz grains. Macropores of subrounded shape are abundant with presence of dolomite crystallization.
According to the petrographic aspect the rock can be classified as "intraclasts bearing micrite" (Dunham 1962) and mudstone (Folk 1959).
According to the mineralogical composition it can be classified as a dolomite rock



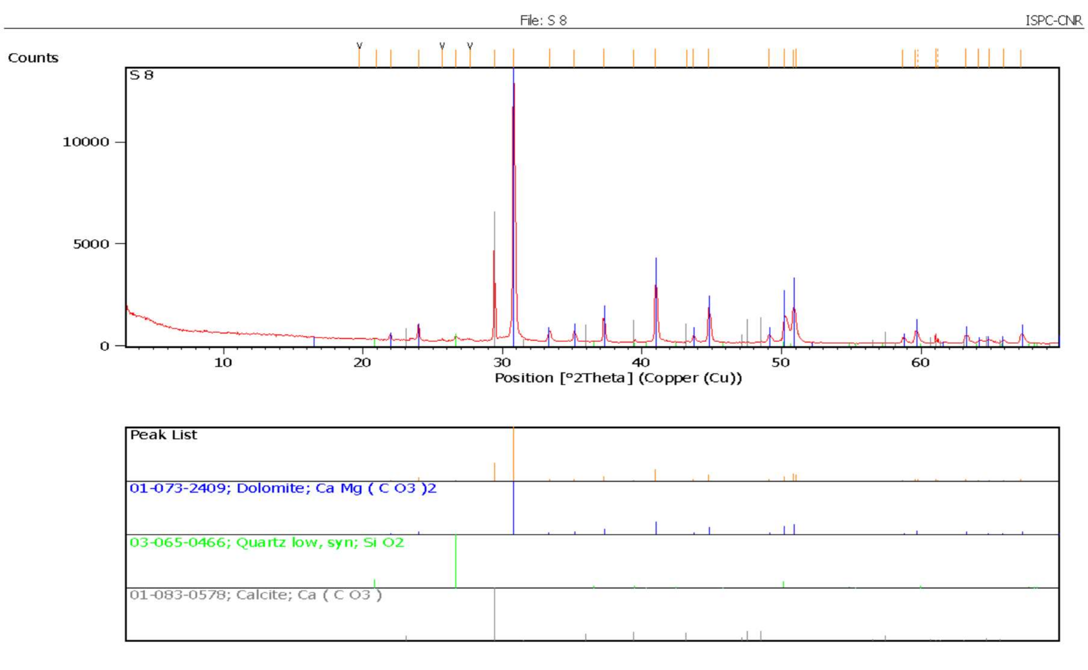
S 7 - Thin section image, transmitted light, crossed nicols

S8 Cornice West side Cornice stone

S 8 - Cross section image

X ray diffraction analysis

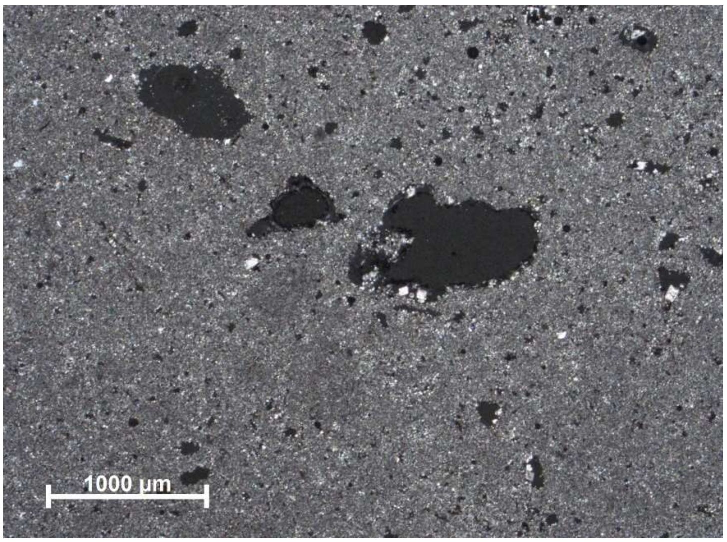
The stone is made of dolomite with a little amount of calcite and quartz. According to this composition it can be classified as dolomitic stone



S 8 - XRD diffraction pattern

<u>Petrographic observation in thin section</u>

The stone shows a microsparitic texture with rare intraclasts and quartz grains. Macropores of subrounded shape are abundant with presence of dolomite crystallization.
According to the petrographic aspect the rock can be classified as “intraclasts bearing micrite” (Dunham 1962) and mudstone (Folk 1959).
According to the mineralogical composition it can be classified as a dolomite rock



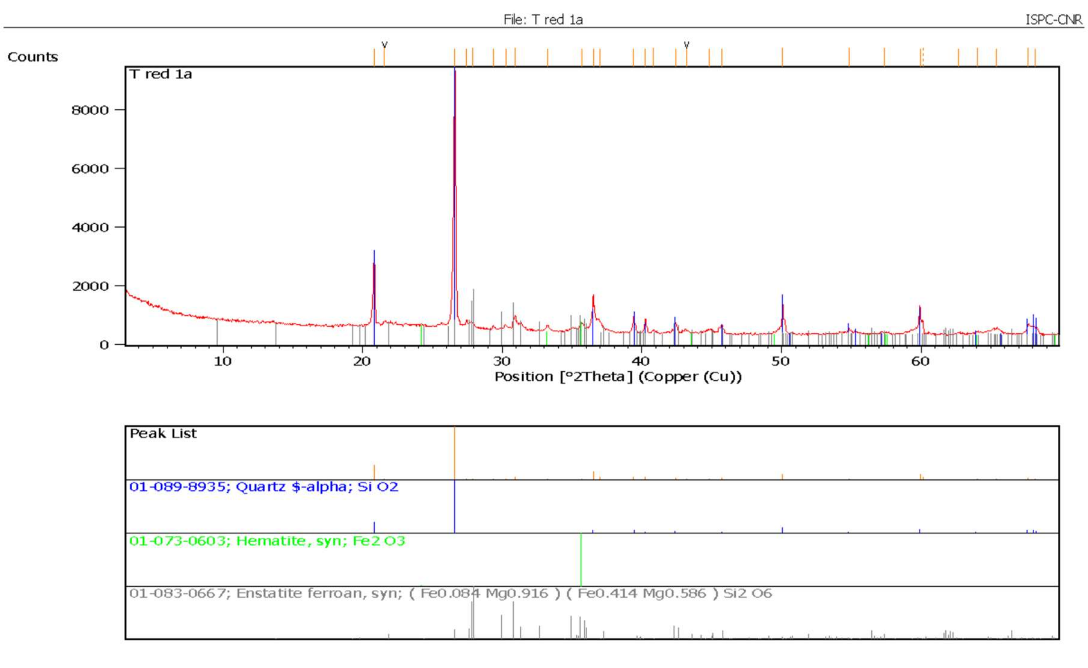
S 8 - Thin section image, transmitted light, crossed nicols

T (red) 1a   Provided old tile - XII sec

T (red) 1a - Cross section image

<u>X ray diffraction analysis</u>

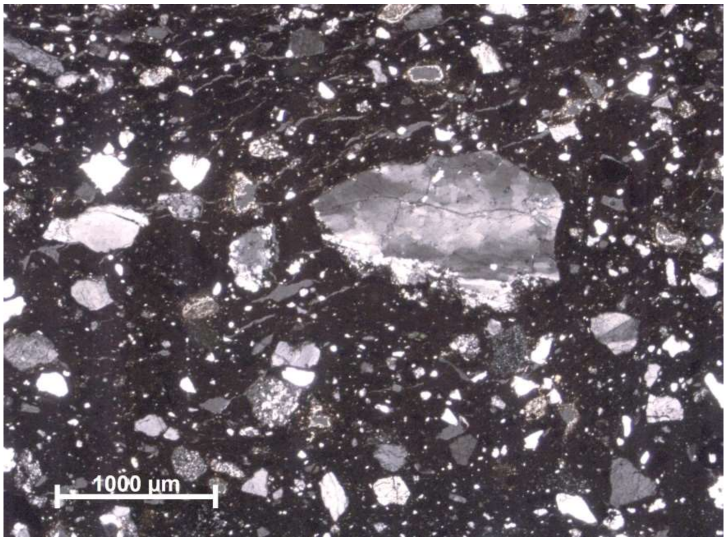
The crystalline phases present in the tile are quartz, hematite and pyroxenes (enstatite)



T (red) 1a - XRD diffraction pattern

Petrographic observation in thin section

The tile shows an opaque groundmass with rare bonherz (iron rich lumps). The framework is abundant, made of quartz and secondarily pyroxenes. The grain-size distribution is bimodal (100 - 200 µm and 400-500µm) with grains of angular shape. The macropores show an elongated shape.



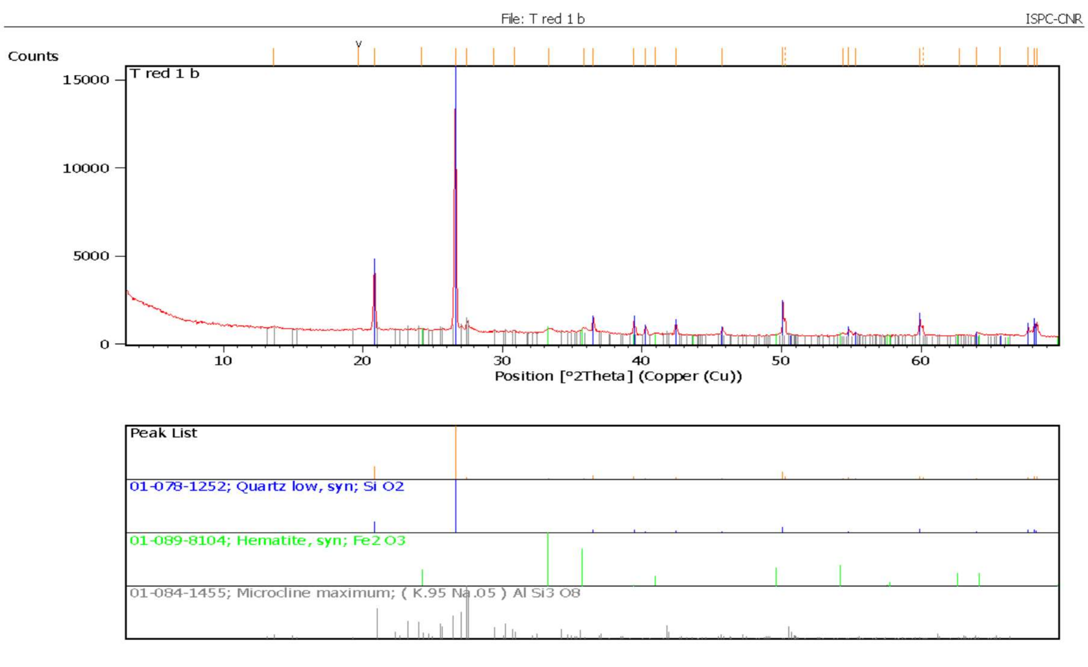
T (red) 1a - Thin section image, transmitted light, crossed nicols

T (red) 1 b   Provided old tile - XII sec

T (red) 1b - Cross section image

X ray diffraction analysis

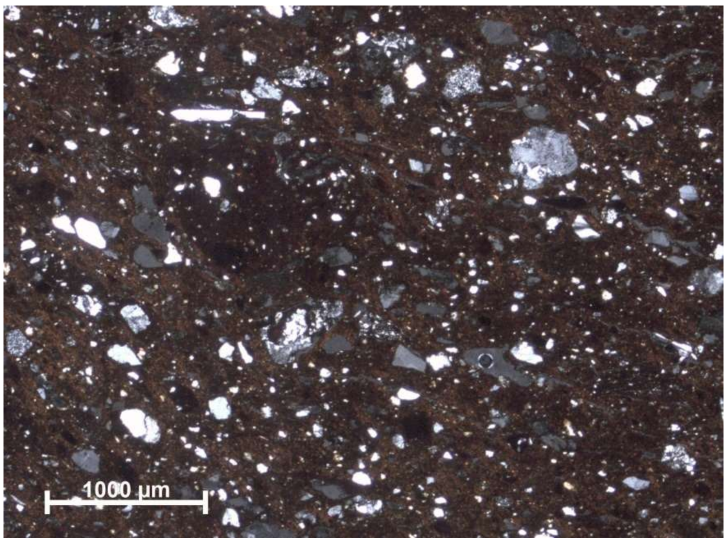
The crystalline phases present in the tile are quartz, hematite and feldspars (microcline)



T (red) 1b - XRD diffraction pattern

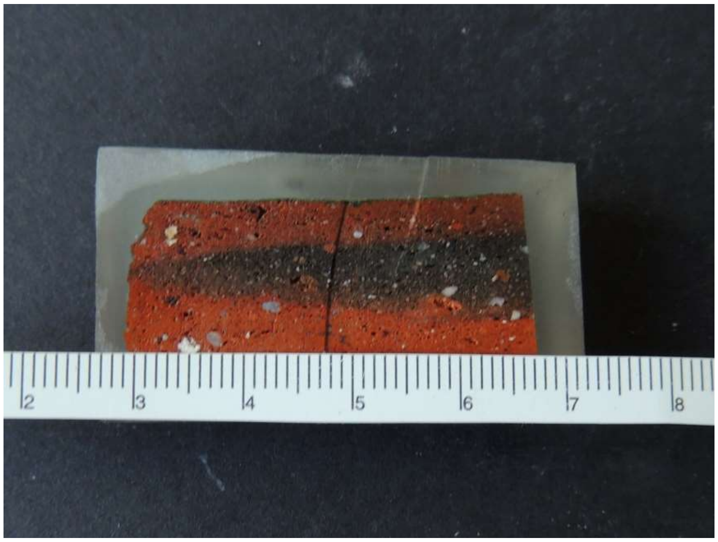
Petrographic observation in thin section

The tile shows a birefringent groundmass with abundant bonherz (iron rich lumps). The framework is abundant, made of quartz and feldspars. The grain-size distribution is bimodal (100 - 200 µm and 400-500µm) with grains of angular shape. The macropores show an elongated shape.



T (red) 1b - Thin section image, transmitted light, crossed nicols

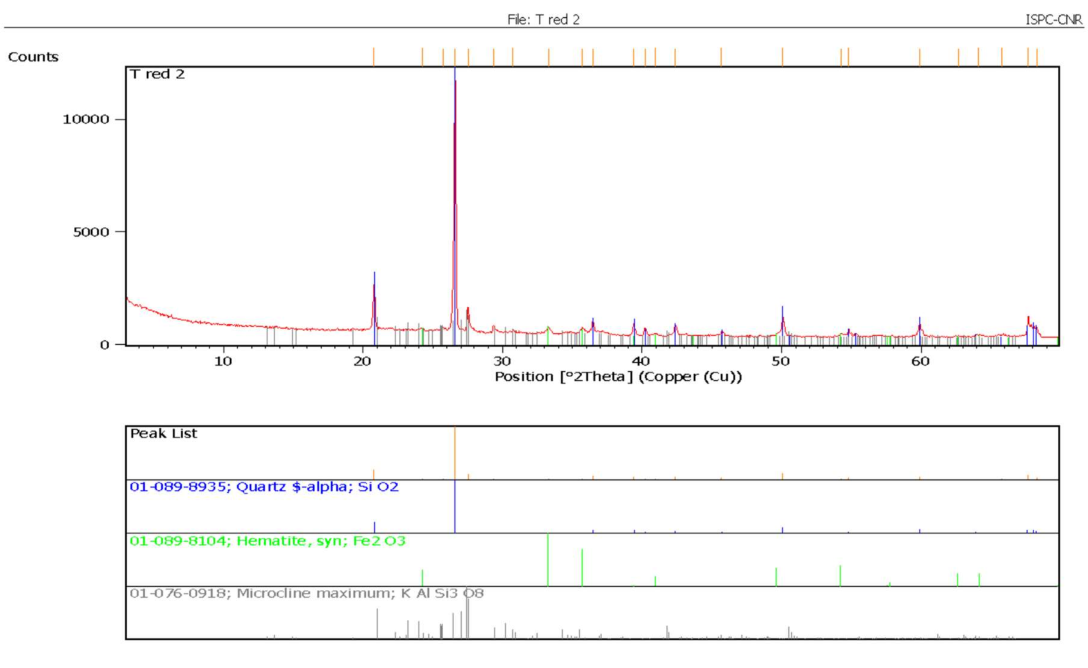
T (red) 2 Interior old tile - XVI sec



T (red) 2 - Cross section image

X ray diffraction analysis

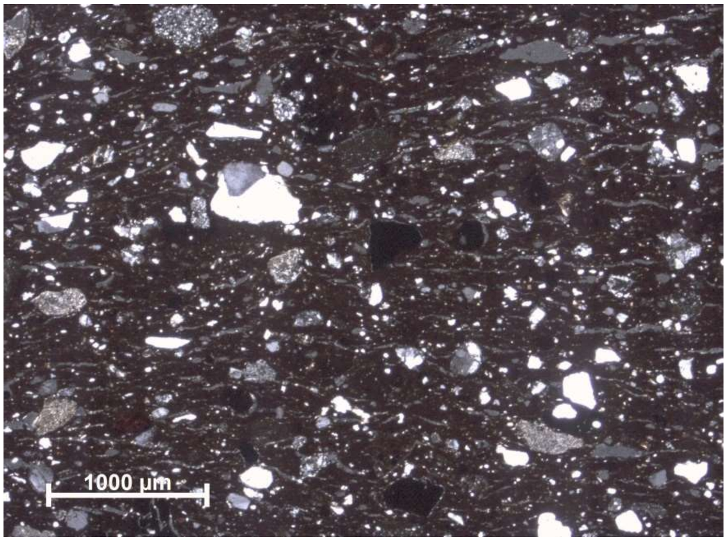
The crystalline phases present in the tile are quartz, hematite and feldspars (microcline)



T (red) 2 - XRD diffraction pattern

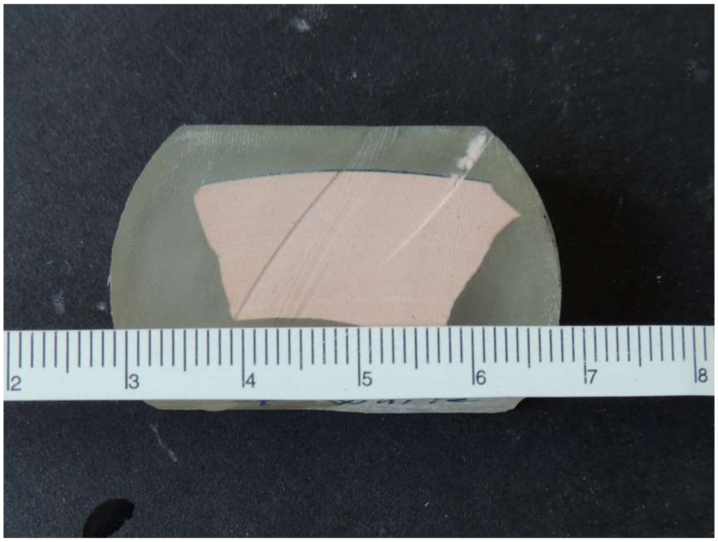
Petrographic observation in thin section

The tile shows an opaque groundmass with rare bonherz (iron rich lumps). The framework is abundant, made of quartz and secondarily pyroxenes. The grain-size distribution is bimodal (100 - 200 µm and 400-500µm) with grains of angular shape. The macropores show an elongated shape



T (red) 2 - Thin section image, transmitted light, crossed nicols

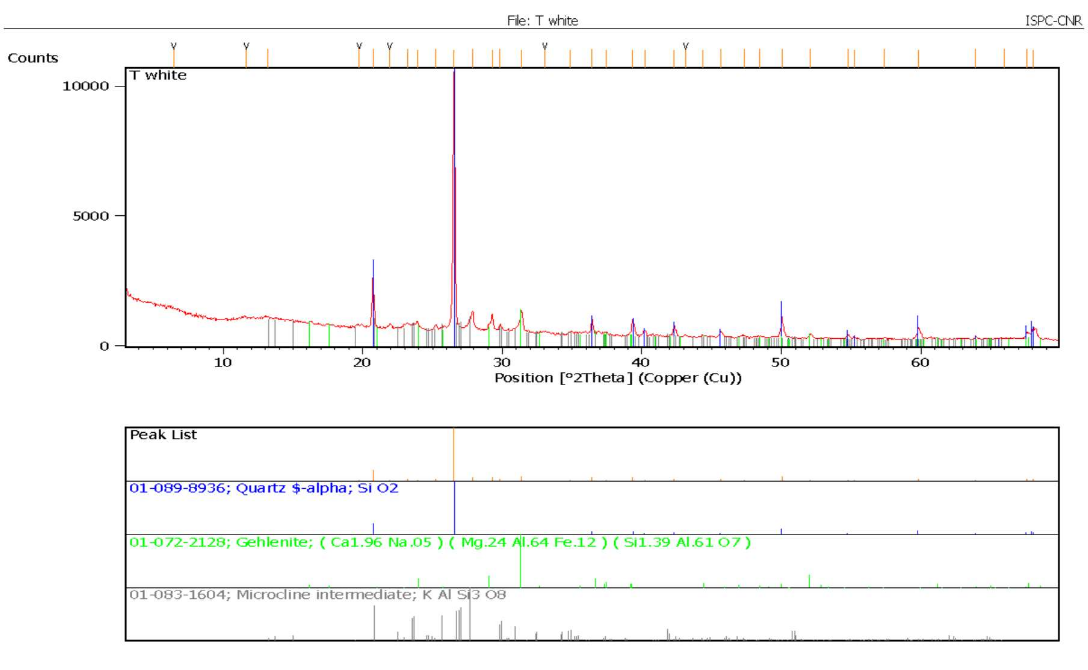
T (white)   Provided new white tile



T w - Cross section image

<u>*X ray diffraction analysis*</u>

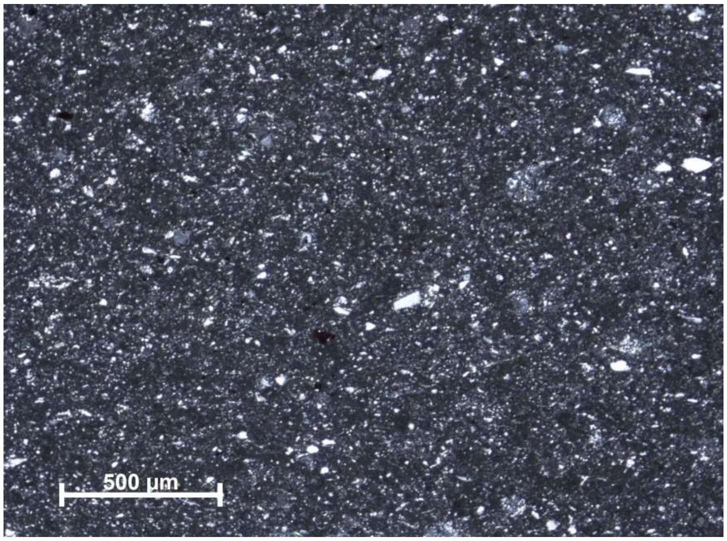
The crystalline phases present in the tile are quartz, feldspars (microcline) and gehlenite. This mineral forms when a marly clay is fired to realize earthenwares



T w - XRD diffraction pattern

Petrographic observation in thin section

The tile shows a slightly birifrengent groundmass. The framework is scarce, made of quartz and feldspars. The grain-size distribution is unimodal (50-100µm) with grains of angular shape. The macropores are scarce, of regular shape



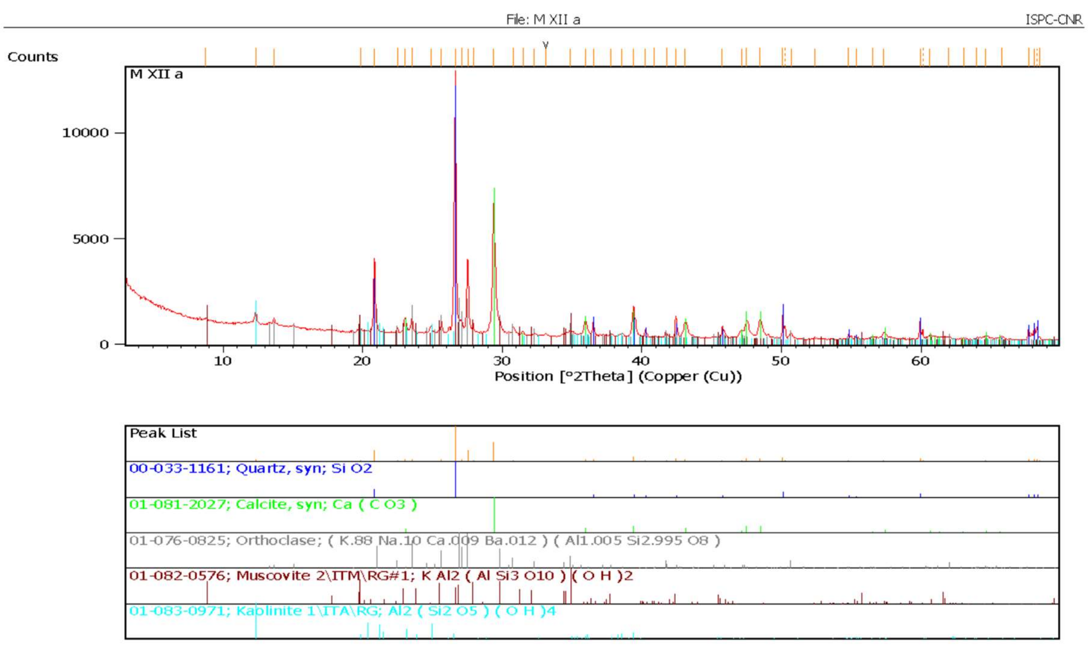
T w - Thin section image, transmitted light, crossed nicols

M.XII-a Provided, from XII tile mortar XII sec

M XII a - Cross section image

X ray diffraction analysis

The mortar is made of calcite, quartz, feldspars (orthoclase), micas (muscovite), with traces of clay minerals) kaolinite)



M XII a - XRD diffraction pattern

Petrographic observation in thin section

The mortar shows an abundant binder (Binder/Aggregate ~ 1/2) constituted by an air hardening lime with a micritic texture. The aggregate is made of quartz, feldspars, micas, and carbonate rock fragments. The grains are angular in shape with a unimodal grain-size distribution (400-800 µm). Lime lumps are present. The macropores are constituted by shrinkage fractures.

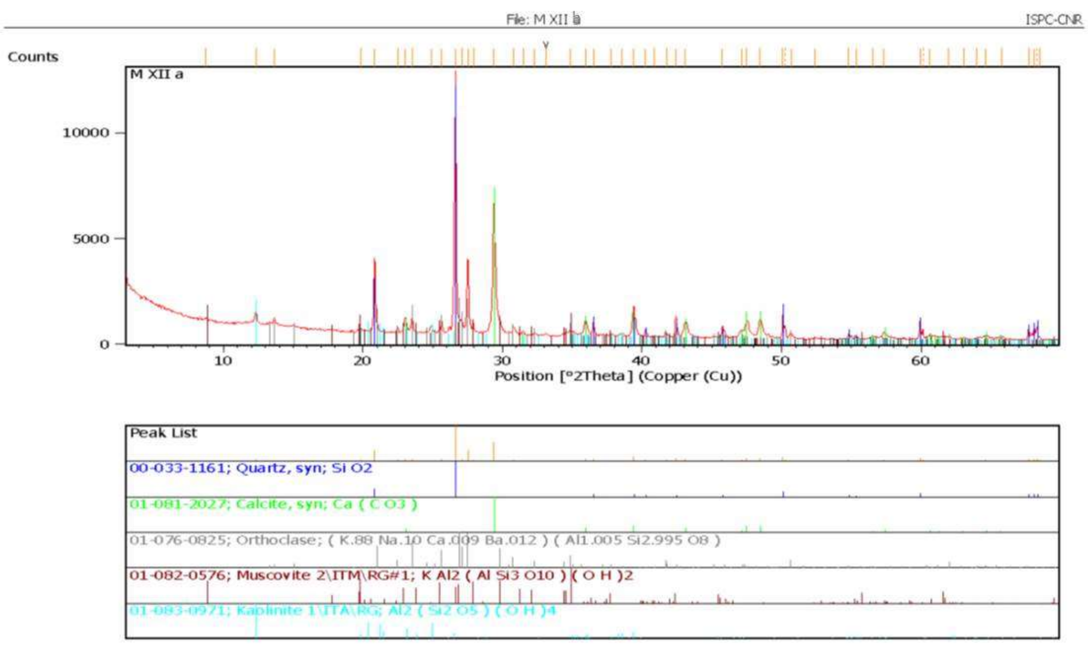
M XII a - Thin section image, transmitted light, crossed nicols

M.XII-b Provided, from XII tile mortar, to check if XII sec

M XII b - Cross section image

X ray diffraction analysis

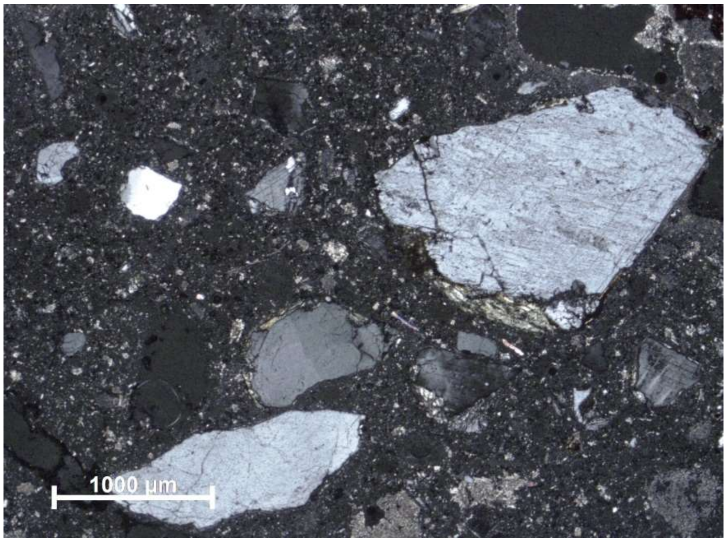
The crystalline phases present in the tile are quartz, hematite and feldspars (orthoclase)



M XII b - XRD diffraction pattern

Petrographic observation in thin section

The mortar shows an abundant binder (Binder/Aggregate ~ 1/2) constituted by an air hardening lime with a micritic texture. The aggregate is made of quartz, feldspars, micas, and carbonate rock fragments. The grains are angular in shape with a unimodal grain-size distribution (400-800 µm). Lime lumps are present. The macropores are constituted by shrinkage fractures.

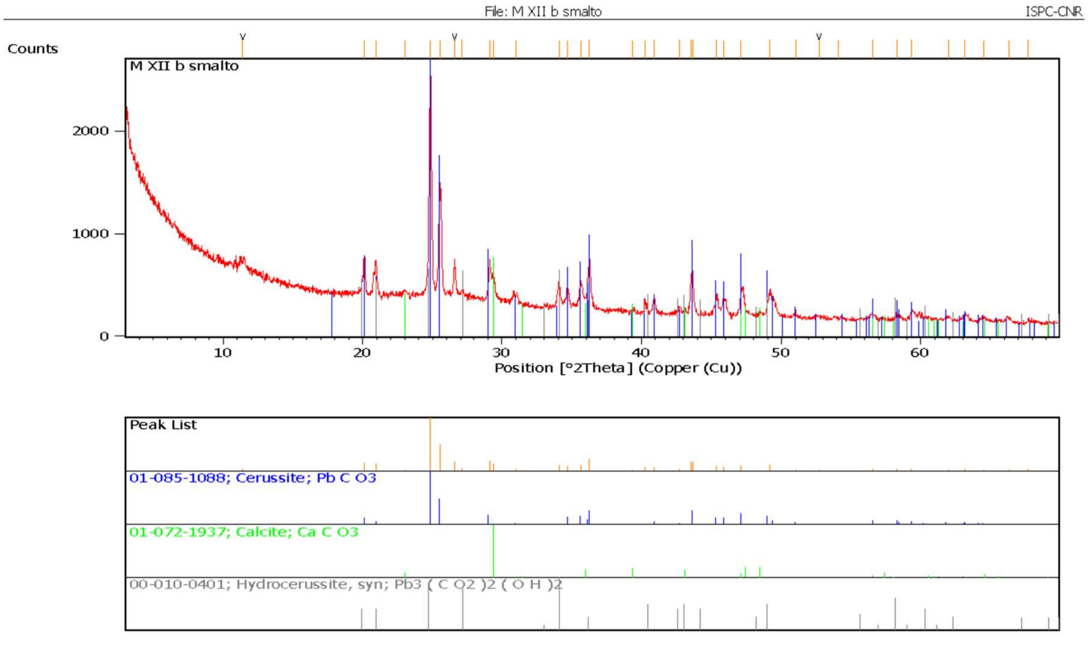


MXII b - Thin section image, transmitted light, crossed nicols

M.XII-b Provided, from XII tile mortar, to check if XII sec glaze

X ray diffraction analysis

The crystalline phases present in the glaze are cerussite –$PbCO_3$-, hydrocerussite-$Pb_3(CO_3)_2(OH)_2$- and calcite. The presence of hydrocerussite points out a decay process in the glaze.



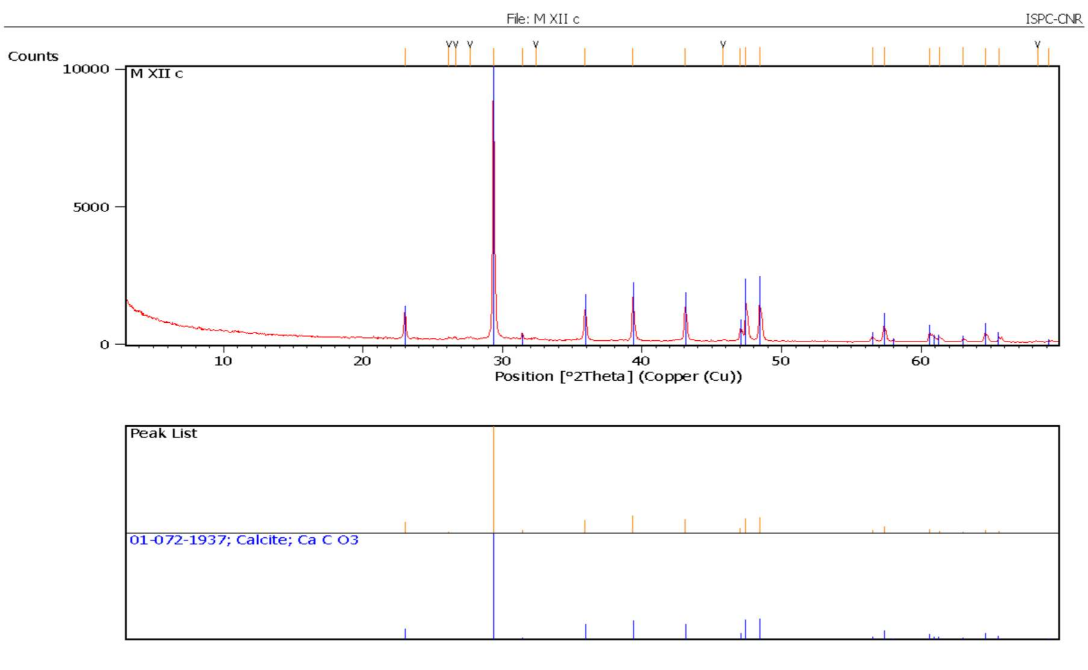
M XII b glaze - XRD diffraction pattern

M.XII-c   Provided mortar, to check if XII sec

M XII c - Cross section image

X ray diffraction analysis

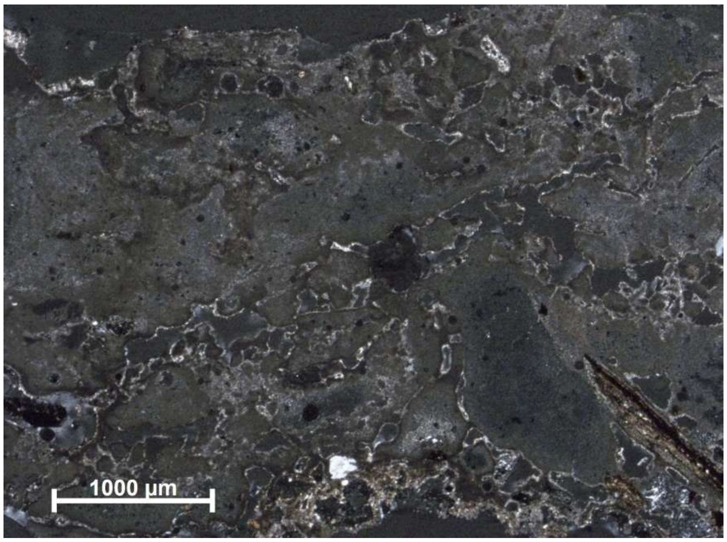
This mortar is made only of calcite



M XII c - XRD diffraction pattern

Petrographic observation in thin section

The sample is constituted by an air hardening binder without presence of aggregate. Lime lumps are present. The macropores are frequent, of irregular shape



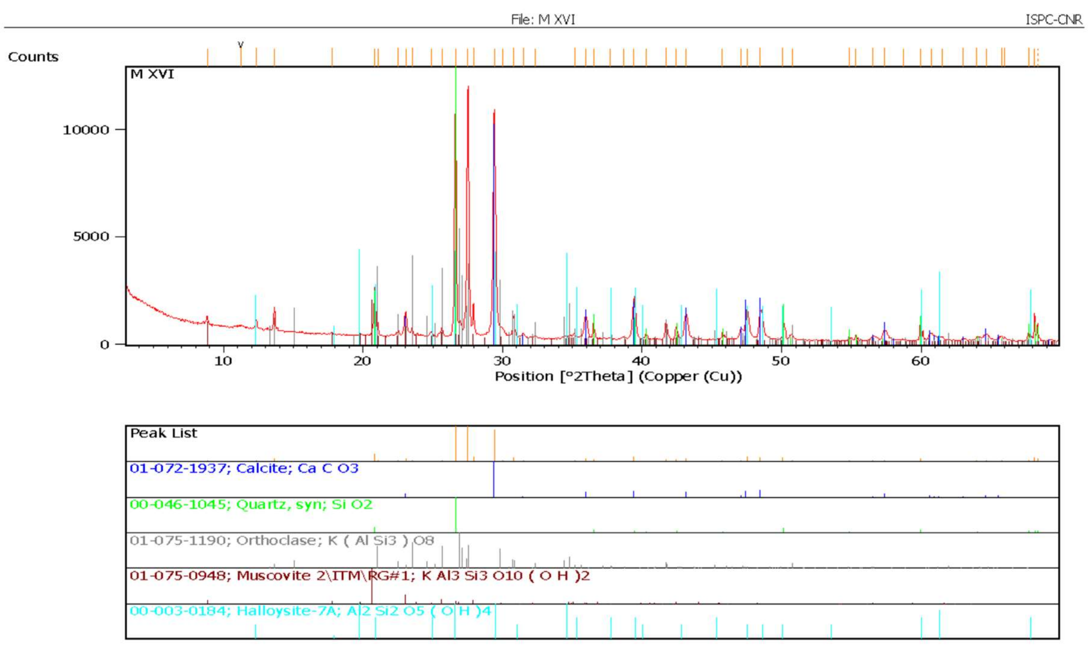
MXII c - Thin section image, transmitted light, crossed nicols

M.XVI *Provided mortar XVI sec*

M XVI - Cross section image: the mortar is in the left side

Xray diffraction analysis

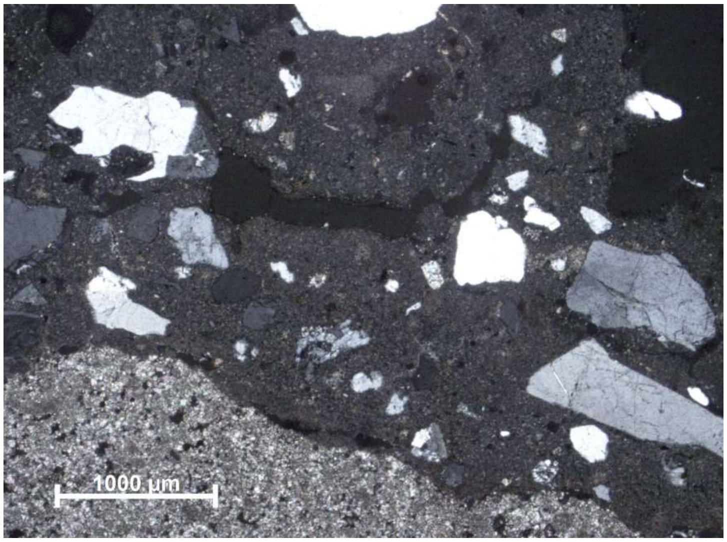
The mortar is made of calcite, quartz, feldspars (orthoclase), micas (muscovite) and clay minerals (halloysite)



M XVI - XRD diffraction pattern

Petrographic observation in thin section

The mortar shows an abundant binder (Binder/Aggregate ~ 1/2) constituted by an air hardening lime with a micritic texture. The aggregate is made of quartz, feldspars, micas, and carbonate rock fragments. The grains are angular in shape with a unimodal grain-size distribution (400-800 µm). Lime lumps are present. The macropores are constituted by shrinkage fractures



M XVI - Thin section image, transmitted light, crossed nicols

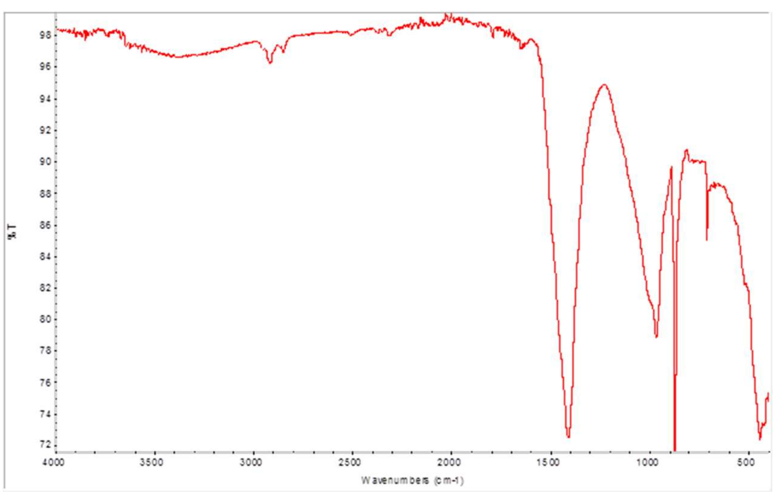
Results of the FTIR analyses

R2 b Saggio R2 Insulating, connection between roof and wall

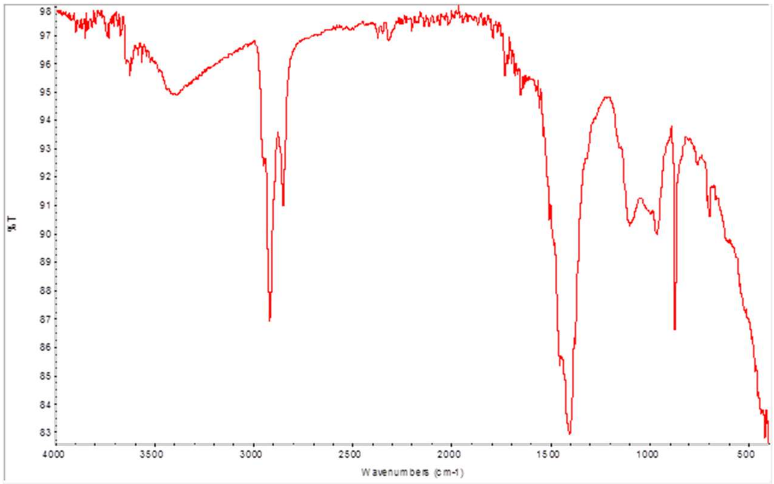
The sample appears as a thin, non-brittle, elastic fragment. Macroscopically it is possible to distinguish an external light brown layer, a blue layer and an internal non-continuous layer of adhesion with the material underlying the fragment.

Sample R2 b

The outer layer (spectrum 1) is basically composed of calcite and silicates. The blue layer (spectrum 2) shows typical C-H stretching and C-H bending signals of aliphatic compounds, which could indicate the presence of a paraffin wax.



R2 b – FTIR spectrum 1, external layer



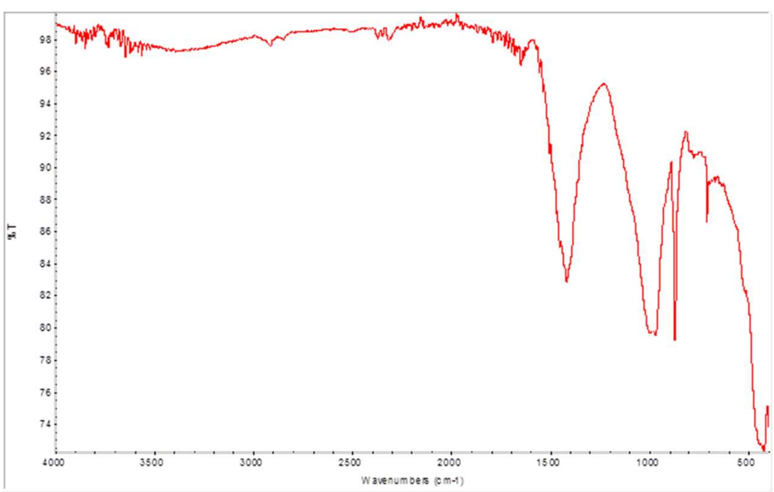
R2 b – FTIR spectrum 2, – intermediate blue layer

R3 b Saggio R3 Insulating, connection between roof and wall

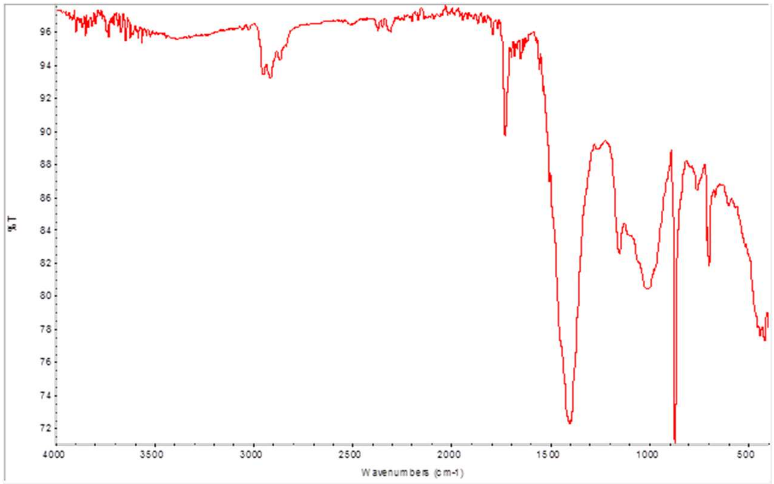
The sample appears as a thin, non-brittle, elastic fragment, similar to R2b. It is possible to distinguish an external light brown layer, a whitish layer, a blue layer and an internal non-continuous layer of adhesion with the material underlying the fragment.

Sample R3 b

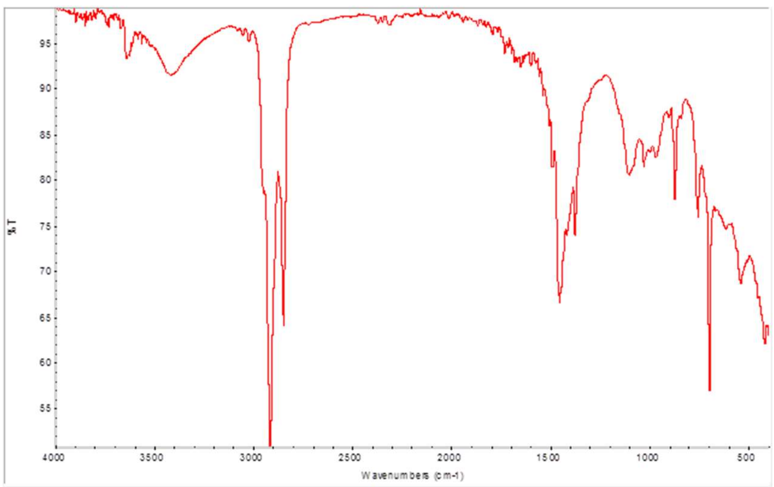
The outer layer (spectrum 3) is basically composed of calcite and silicates. The intermediate whitish layer (spectrum 4) shows a peak at 1733 cm-1 (carbonyl C = O), which could indicate the presence of a wax other than paraffinic, probably partially saponified. The blue layer (spectrum 5) shows typical C-H stretching and C-H bending signals of aliphatic compounds, which could indicate the presence of a paraffin wax.



R3 b – FTIR spectrum 3, – external layer



R3 b – FTIR spectrum 4, whitish intermediate layer



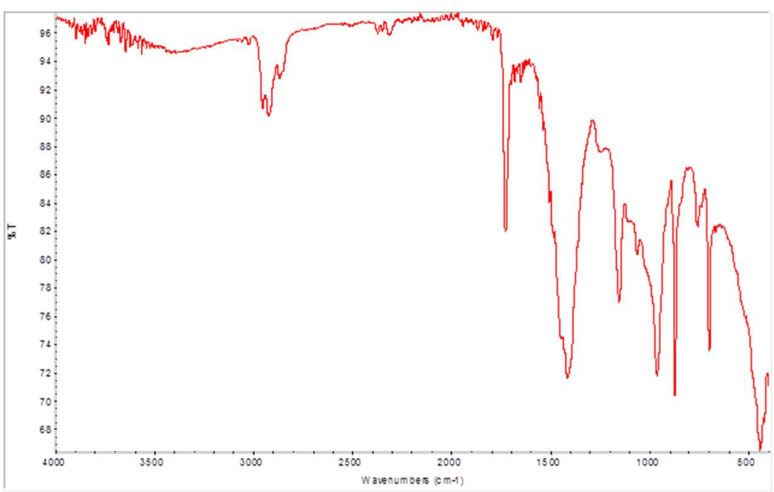
R3 b - FTIR spectrum 5 – blue layer

R3 c Saggio R3 Insulating, liquid (mortar + organic portion)

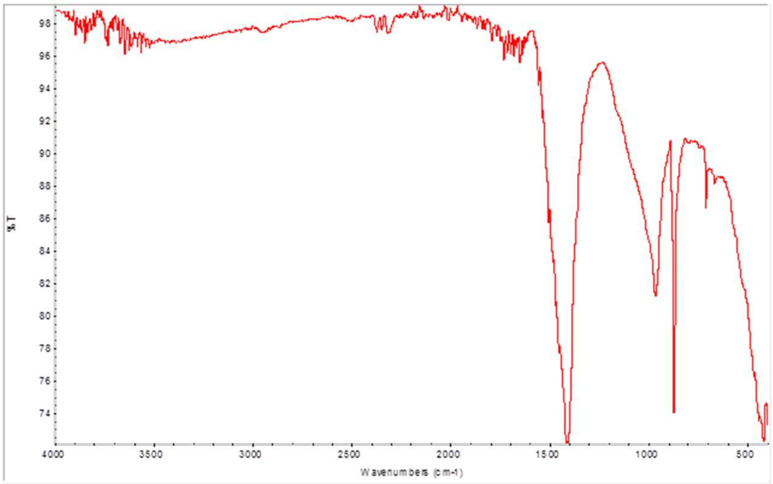
The sample consists of 3 irregularly shaped fragments, in which layers are not macroscopically distinguished as in the previously analysed samples. The fragments subjected to analysis are less elastic, and indeed tend to shatter under pressure from the measuring head of the instrument.

Sample R3 c

The outer layer (Spectrum 6) exhibits the signals of calcite and silicates, but also the signals of what could be a saponified wax. The inner layer (Spectrum 7) is basically calcite and silicates.



R3c - FTIR spectrum 6 – external layer



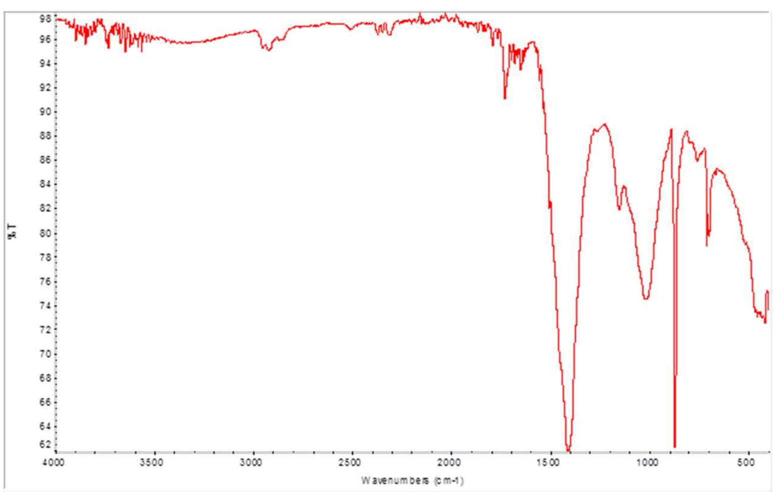
R3c – FTIR spectrum 7 – internal layer

R3 d   Saggio R3   Glue

The sample consists of 2 irregularly shaped fragments, in which layers are not macroscopically distinguished.

Sample R3 d

The fragments subjected to analysis are less friable than the R3c sample, but the FT-IR spectrum of the outer layer (Spectrum 8) shows the presence of what could be traces of wax.



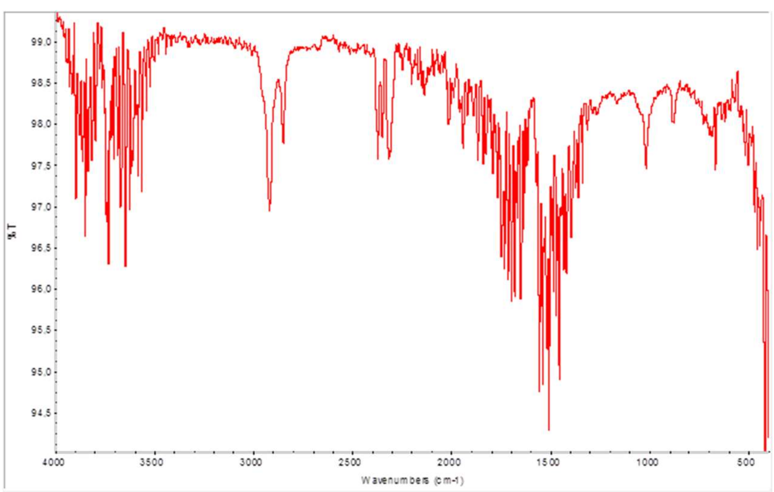
R3 d – FTIR spectrum 8 – external layer

S2 Drum near R3 Wall

The sample, previously analysed from a mineralogical-petrographic point of view, has a translucent brown surface treatment

Sample S2

The sample is not easy to analyse with the ATR accessory, and the resulting spectra are very noisy and therefore difficult to read. Nevertheless, some peaks attributable to organic compounds are visible, even if not better identifiable (Spectrum 9).



S2 - FTIR spectrum 9 – translucent brown surface

Discussion of the results

The mineralogical-petrographic study of the mortar, stones and tile samples made it possible to investigate their composition.

Mortars

Samples R1d, R2a, R3a, R3b, R3c, F2 show a Binder/Aggregate ratio of 1/2- 1/3 with an impure aspect of possible hydraulic nature. The aggregate is made of quartz, feldspars, micas, fragments of porphyric, granitic and basaltic rocks. The grains are from subrounded to angular with a bimodal grain-size distribution (300-500 µm and 1– 2 mm). The macropores are from irregular to sub-spherical in shape.

Samples R1a and R1b are quite similar to the previous group with the difference of a binder of pure aspect still of hydraulic nature

Sample R1e shows an abundant binder (Binder/Aggregate 1/1-1/2) made of air hardening lime. The aggregate is made of brick fragments, quartz, feldspars. The grains are angular in shape with a bimodal grain-size distribution (100-300 µm and 800 µm –1.5 mm). Rare lime lumps are present. The macropores are sub-spherical in shape.

Samples M XII a, MXII b, M XVI show an abundant binder (Binder/Aggregate ~ 1/2) constituted by air hardening lime. The aggregate is made of quartz, feldspars, micas, and carbonate rock fragments. The grains are angular in shape with a unimodal grain-size distribution (400-800 µm). Lime lumps are present. The macropores are constituted mostly by shrinkage fractures.

Sample M XIIc is constituted by an air hardening binder without presence of aggregate

Tiles

Samples MXII b, T red 1a, T red 1b, T red 2 show an abundant framework with a bimodal grain-size distribution (100 - 200 µm and 400-500µm), and a variable amount of bonherz (iron rich lumps). The macropores show an elongated shape. The groundmass is opaque except for T red 1b with a birefringent aspect due to a lower firing temperature.

Sample R1 c shows a slightly birefringent groundmass rich in bonherz (iron rich lumps) with an abundant framework of fine grain-size

Tw shows a slightly birifrengent groundmass and scarce framework of fine grain size (50-100µm) The macropores are scarce, of regular shape. The presence of gehlenite in the mineralogical composition points out the use of a marly clay as raw material.

Stones

Samples S2, S4, S6, S7, S8 show a microsparitic texture with rare intraclasts and quartz grains. Macropores of subrounded shape are abundant with presence of dolomite crystallization.

According to the petrographic aspect the rock can be classified as “intraclasts bearing micrite” (Dunham 1962) and mudstone (Folk 1959). According to the mineralogical composition it can be classified as a dolomite rock.

Sample S1, from the petrographical point of view, can be classified as the sample of the previous group but calcite is prevalent in the mineralogical composition therefore is defined as dolomitic limestone. Another difference is that the macropores are rare.

Also sample S3, from the petrographical point of view, can be classified as the sample of the previous group, but it is characterized by the presence of intraclasts of sparry calcite.

Sample S5 shows a trachytic texture constituted by small aligned feldspars and phenocrysts constituted by pyroxenes. The macroporosity is abundant with pores of irregular shape. According to the mineralogical composition and petrographic aspect the rock can be referred to an effusive rock (andesite/basalt).

Sample F1 is a pumice (effusive rock) constituted by a completely vitreous groundmass and porphyroblasts with dimensions of 400-800 µm, made of prevailing feldspars (andesine, anorthoclase) and secondarily by pyroxenes. Suspheric pores are abundant with dimension of 400-500 µm.

Insulating materials

In sample R2 b, the outer layer is basically composed of calcite and silicates, the blue layer is a paraffin wax

In sample R3 b, the outer layer is basically composed of calcite and silicates, the intermediate whitish layer is a wax other than paraffinic, probably partially saponified, the blue layer is a paraffin wax.

In sample R3 c, the outer layer is composed by calcite and silicates together with a saponified wax. The inner layer is basically calcite and silicates

In sample R3 d there are traces of wax.

The translucent brown surface treatment of sample S2 is an organic compounds that has not been possible to identify

References

Dunham R.J. (1962) “Classification of Carbonate Rocks According to Depositional Texture”, in: *Classification of Carbonate Rocks*, Ham, W.E., Ed.; AAPG, Tulsa, USA, 108-121

Folk R. L.(1959) “Practical petrographic classification of limestones”, *Am Ass. Pet Geol Bull*, 43, 1-38

Comment on the results of Laboratory analyses

Authors:

Prof. Arch. Ugo Tonietti;

Arch. PhD. Sara Stefanini

Florence, December 23rd 2021

A crucial aim of the mineralogical-petrographic study of the mortar, stones and tile samples consists in the investigation of their composition that allows the chemical-physical characterisation. Such information is decisive for an assessment of the condition of the materials employed in the artefact, for an understanding of what was implemented in the last intervention on the monastery, and to guide the identification of a new conservation design.

It would be useful to try to comment on the results of the tests, analysing the connection with the behaviour exhibited by the monastery in recent times (with particular attention to the weaknesses that have emerged).

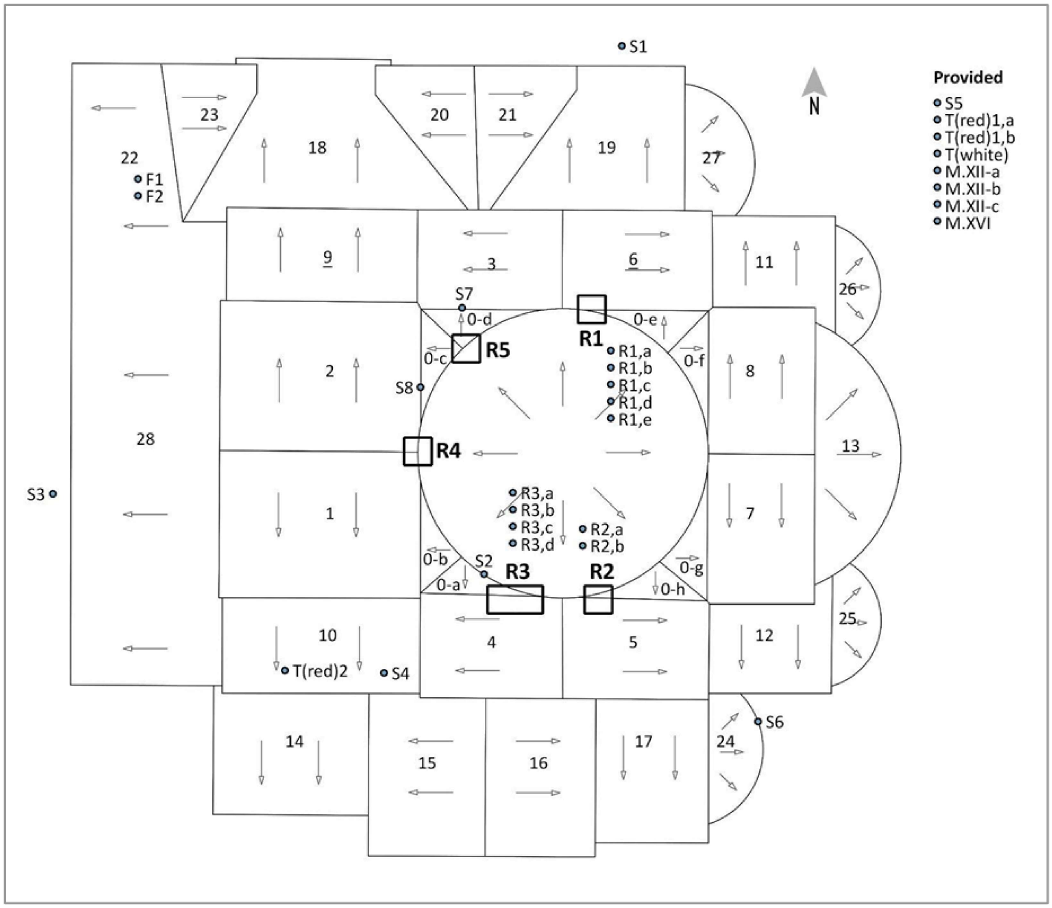


Figure 1 - Map of the localisation of the sondages and the samples taken during the June 2021 mission.

Figure 2 - Samples taken on-site during the June 2021 mission.

Sample	**NAME**	**Pick-up point**	**Description**
1	**R1a**	Sondage R1	Tile filling mortar
2	**R1b**	Sondage R1	Bedding mortar (under tiles)
3	**R1c**	Sondage R1	New red tile
4	**R1d**	Sondage R1	Mortar - connection between roof tile and wall
5	**R1e**	Sondage R1	Compound used for the recomposition of voids in the stones
6	**R2a**	Sondage R2	Mortar at the connection roof/wall
7	**R2b**	Sondage R2	Textile waterproofing insulating at the connection roof/wall
8	**R3a**	Sondage R3	Bedding mortar (under tiles)
9	**R3b**	Sondage R3	Textile waterproofing insulating at the connection roof/wall
10	**R3c**	Sondage R3	Waterproofing insulating, liquid layer
11	**R3d**	Sondage R3	Glue
12	**F1**	Under-roof pitch n. 22	Pumice, filling of the under-roof n.22
13	**F2**	Under-roof pitch n. 22	Mortar, filling of the under-roof n.22
14	**S1**	Collected Northside	Wall Stone
15	**S2**	Drum near R3	Wall Stone
16	**S3**	Westside	Basement Stone
17	**S4**	Interior	Wall Stone
18	**S5**	Provided	Stone slab, 12th-13th century
19	**S6**	Apse South-East side	Wall Stone
20	**S7**	Cornice Northside	Cornice Stone
21	**S8**	Cornice Westside	Cornice Stone
22	**T(red)1a**	Provided	Old tile, 12th century
23	**T(red)1b**	Provided	Old tile, 12th century
24	**T(red)2**	Interior	Old tile,16th century
25	**T(white)**	Provided	New white tile
26	**M.XIIa**	Provided, From 12th tile	Bedding mortar (under tiles), 12th century
27	**M.XIIb**	Provided, From 12th tile	Bedding mortar (under tiles), 12th century
28	**M.XIIc**	Provided, From stone slab	Bedding mortar (under tiles), 12th century
29	**M.XVI**	Provided, From 16th tile	Bedding mortar (under tiles), 16th century

Table 1 - Complete set of samples

Mortars

The analyzes show a substantial difference between the samples of the historical mortars (M.XIIa, M.XIIb, M.XIIc, M.XIV) and the samples of the more recent mortars (R1a, R1b, R1e, R1d, R2a, R3a, R3b, R3c, F2): the former group is composed of aerial mortar, while the latter one from hydraulic or hydraulicized mortar - sample R1e.
In particular, the samples R1d, R2a, R3a, R3b, R3c, F2 show a more impure binder than the samples R1a, R1b which show a lower hydraulic capacity.
However, it should be emphasized that, despite the hydraulic properties of modern mortars, they cannot represent an obstacle to the filtration of water and humidity due to the porous consistency of the dough. For this reason, the mortar layer cannot be entrusted with the task of protecting the building from rain.

Historical samples exhibit an abundant air-hardening lime binder, which makes them more porous than modern mortars. This fact might indicate that the ancient builders did not use the mortar to waterproof the roof, a task that may have been assigned to other construction devices. It would be appropriate to investigate such an issue with the help of a historian expert of the technological solution adopted on the roofs in that age.

As regards the sample R1e, representative of the material employed for the restoration of damaged and exfoliated surfaces at the base of the drum, it has to be noted the composition based on an aerial mortar made hydraulic through the insertion of brick fragments.

Tiles

The tile sample belonging to a recent white tile production characterized by a white body - sample T(white) – owes its peculiar character to the fact that it was produced, by firing, at low temperatures, a very marly clay (that is, containing a lot of calcite). During cooking, therefore, a high quantity of calcium oxide developed. Such oxide was not able to react entirely with the silicon coming from clay minerals (made amorphous in cooking). Therefore, there is still some unbound calcium oxide. Unfortunately, this oxide tends to continue the hydration and carbonation process once it is applied in a humid environment, with the consequence that the dough, continuing to hydrate itself, increases in volume and flakes. This fact may explain the severe damage exhibited by these typologies of glazed tiles. The marly component is at the origin of the white colouring.

The tile sample belonging to a recent production characterized by a red body - sample R1c - was fired at a non-optimal temperature. It is a sample with an abundant sandy framework, cooked at a not very high temperature, so it does not have very high cohesion. The clay is not too worked. The presence of iron-rich lumps in the sample indicates a lack of seasoning of the earth used in the production of the material. In fact, the clay must be seasoned for months before being used in the production of building materials; the seasoning causes the lumps to be lost.

The samples from ancient tiles - samples M.XIIb, T(red)1a, T(red)1b, T(red)2 - are produced by a more effective cooking process. This fact can be deduced from the opacity of the groundmass that has lost its crystalline appearance. On the contrary, in modern samples, the dough is slightly birefringent, due to the presence of crystalline elements.

Stones

The stone samples taken from the walls (drum, women's gallery and apse) appear to be variants of the same type of rock, i.e. dolomite. Sample S1 is also the same type of rock, even if it has not been dolomitized. The S3 stone sample, taken from the plinth at the entrance of the Church, always belongs to the dolomites rocks, even if it exhibits a more crystallized structure than the other samples.
Sample S5, the representative of the stone slabs used in ancient times for roofing, is a basalt, volcanic rock. This type of rock turns out to be excellent for its resistance to meteoric actions and therefore for use on roofs.

The F1 sample, taken from the filling material of the under roof of pitch n. 22, is an effusive rock too, known as pumice, which is characterized by the presence of many closed pores into which water does not enter, making it waterproof. Anyway, it should be noted that the mortar, with which these stones have been put in place, is not a waterproof material.

Insulating Materials

The samples consist of several layers. The external layer of calcite and silicates is probably belonging to the mortar that has remained attached to the insulation.

Paraffin waxes are generally durable materials.

The real problem in the analysed case is how the insulation material was applied: the absolute thinness of the thickness and its very modest extension makes its use as insulation insignificant.

Tests on Porosity and Bulk density

Sample	Description	Porosity %	Bulk volume gr/cm^3
R1a	Tile filling mortar	22	1.87
R1b	Bedding mortar (under tiles)	25	1.86
R1d	Mortar - connection between roof tile and wall	22	1.91
R1e	Compound used for the recomposition of voids in the stones	34	1.62
R2a	Mortar at the connection roof/wall	23	1.91
R3a	Bedding mortar (under tiles)	23	1.91
R3c	Mortar taken together with waterproofing insulating liquid layer	26	1.91
F1	Pumice, filling of the under-roof n.22	13	1.13
F2	Mortar, filling of the under-roof n.22	22	1.86
T(red)1a	Old tile, 12th century	15	1.94
T(red)1b	Old tile, 12th century	23	1.92
T(red)2	Old tile, 16th century	13	2.05
M.XIIb	Old tile, 12th century - sample with mortar	20	2.00
R1c	New red tile	25	1.86
T(white)	New white tile	31	1.6
S1	Wall Stone - carbonatic stone	12	2.40
S2	Wall Stone - carbonatic stone	15	2.21
S3	Basement Stone - carbonatic stone	7	1.94
S4	Wall Stone - carbonatic stone	18	2.07
S5	Stone slab, 12th-13th century - volcanic stone	11	2.16
S6	Wall Stone - carbonatic stone	15	2.08
S8	Cornice Stone - carbonatic stone	14	2.10

Table 2 - Physical characteristics of the samples: determination of the water accessible porosity and bulk volume through the hydrostatic balance method. The analysis of porosity was not carried out on the samples not shown in the table due to the small quantity of the material.

Trying to better understand the results of the tests, in particular as regards the behaviour of some materials crucial for explaining their capacity to contrast the water infiltration or the resistance to humidity and meteoric actions, we asked CNR-ISPC to carry out an in-depth study on porosity and bulk density. Table n.2 shows such a study.

The porosity values tell us that the mortars exhibit, as expected and obvious, a high porosity accessible to water. The result is a remarkable water permeability of the material.

More interesting is the evaluation of porosity for the tiles: in fact, the new ones exhibit a porosity value (25% for the red type and 31% for the white type) greater than the average value (17.75 %) for the old ones. This is a further indicator of the weakness of the new tiles to exposure to water.

The bulk density (i.e. the weight for unit of volume) is a parallel indicator that puts in evidence the incidence of voids in the material (for the new tiles: average value of 1.73 vs 1.98 for the old ones).