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RESTAURACIÓN DEL
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INSTITUTO UNIVERSITARIO DE INVESTIGACIÓN



UNIVERSITAT
POLITÈCNICA
DE VALÈNCIA

FESEM-EDX study of the crucible for melting furnace

R+D Project AICO/2021/095

“Developing strategies against climate change for the preventive conservation of Valencian railway-tramway heritage”

Financial support by *Consellería de Innovación, Universidades, Ciencia y Sociedad Digital. Dirección General de Ciencia e Investigación. Subvenciones para la realización de proyectos I+D+i desarrollados por grupos de investigación consolidados 2021*

Authors:

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Miguel F. Silva

Date:

Valencia, 2 November 2022

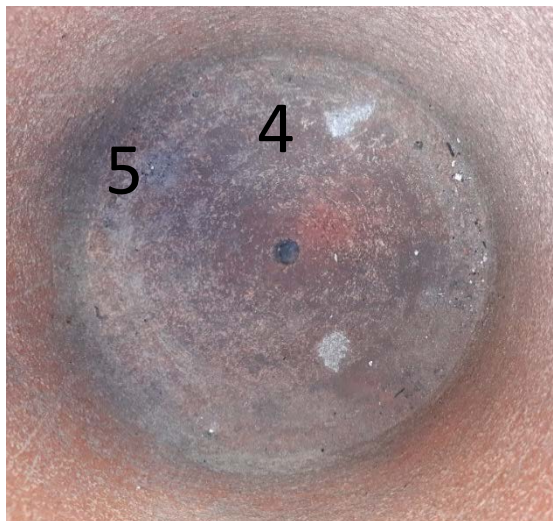
Instrumentation:

FESEM-EDX: Zeiss model ULTRA 55 that operates with an accelerating voltage of 20 kV in the electron source and working distance of the detector 2.9–4.0 mm.

Working conditions:

Microfragments previously examined by optical microscopy were mounted on aluminum disks adapted to be placed in the vacuum chamber of the FESEM-EDX. All the analyzed samples were carbon-coated to avoid localized charging and distortion or reflection of the electron beam.

Sampling points



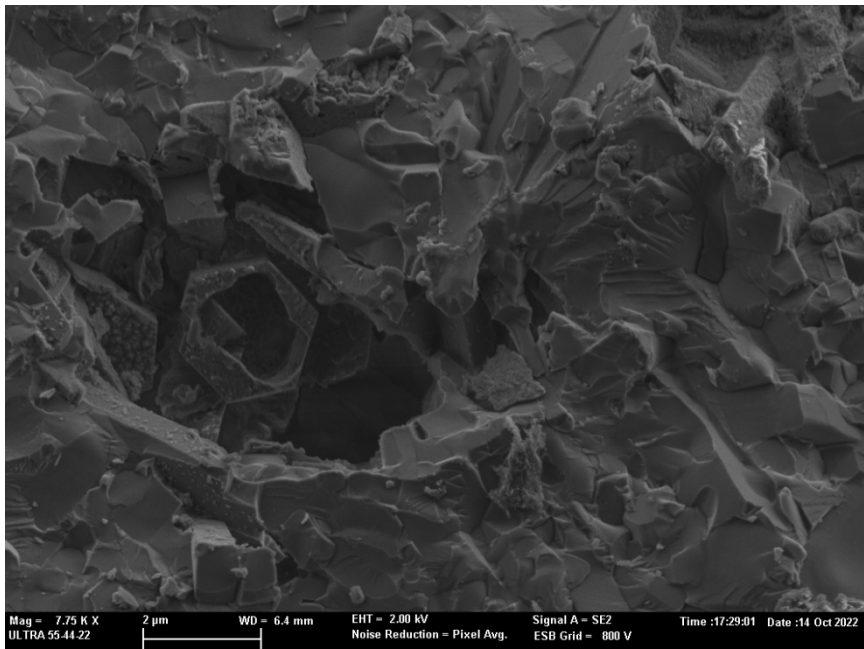
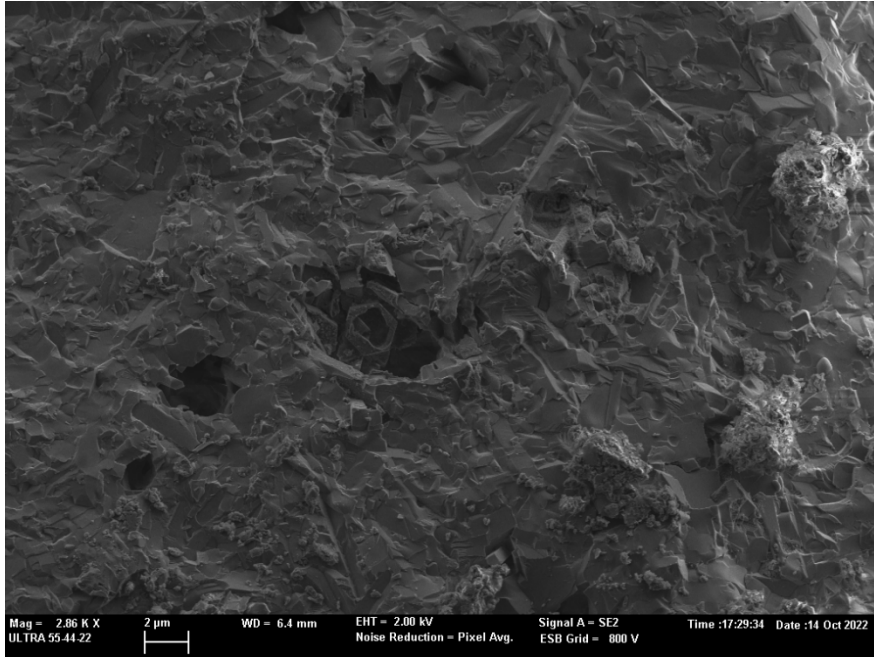
Sample: Refractory ceramic material

Ref: 1

Sampling point: external side



Secondary electron images

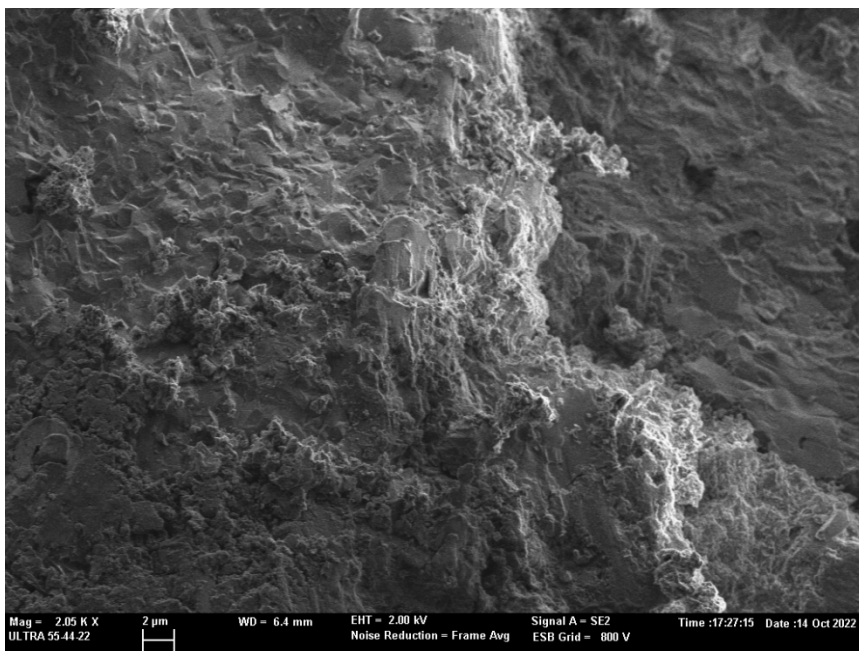
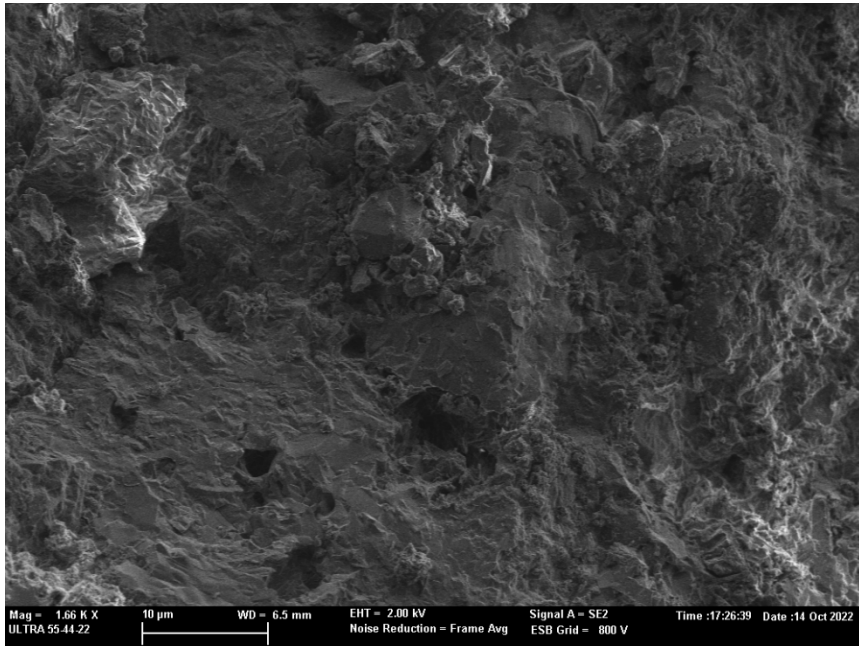


Vitrified micromorphology of the ceramic paste. In the center of the image an euhedral grain with hexagonal habit is ascribed to quartz.

Sample: Refractory ceramic material

Ref: 1

Sampling point: external side

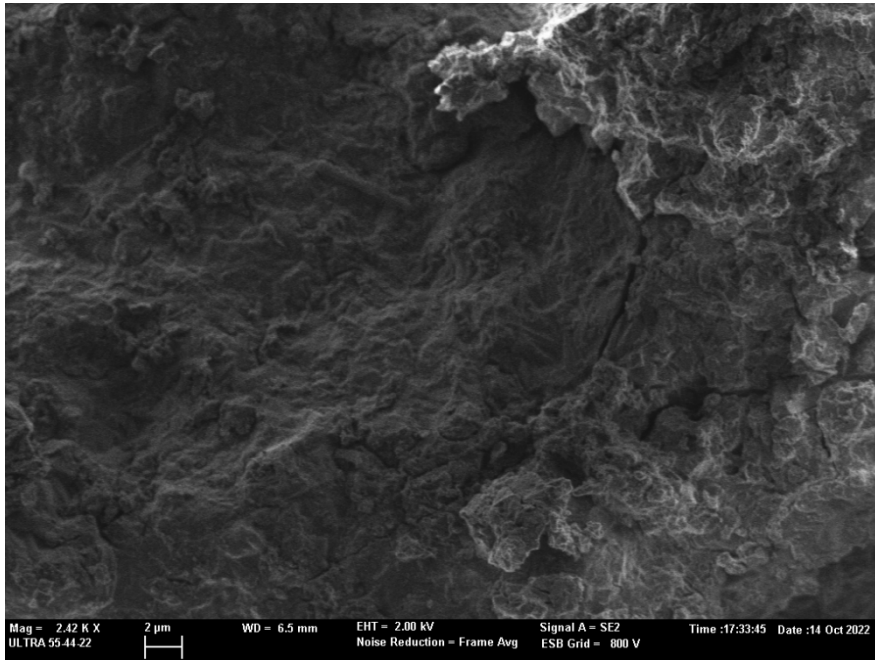


Vitrified micromorphology of the ceramic paste. Some polyedric grains protrude the vitrified paste. The euhedral shape is associated with neoformation mineral phases.

Sample: Refractory ceramic material

Ref: 1

Sampling point: external side



Vitrified micromorphology of the ceramic paste. Some poliedric grains protrude the vitrified paste. The euhedral shape is associated with neoformation mineral phases.

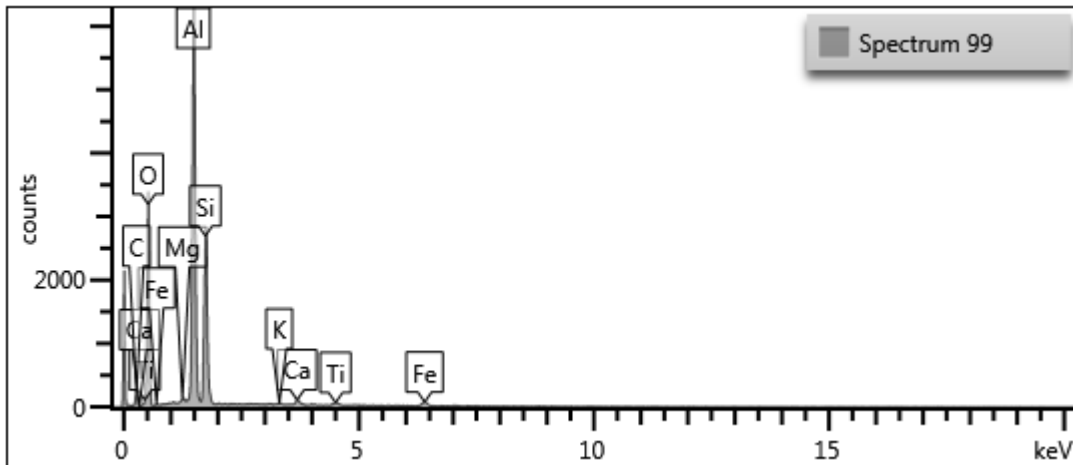
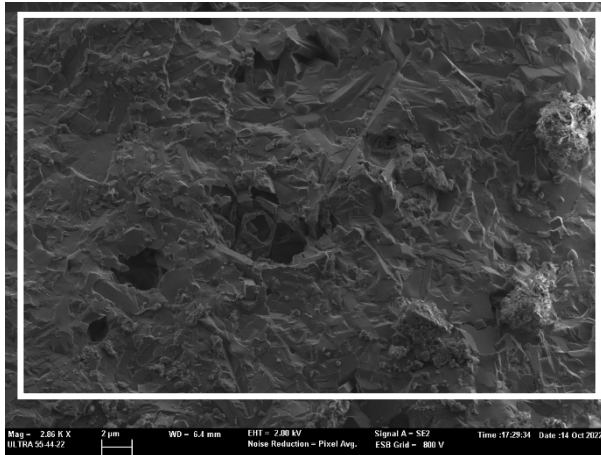
Sample: Refractory ceramic material

Ref: 1

Sampling point: external side



Chemical composition: clayey minerals and quartz that form the ceramic body



Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %	Standard Label
O		48,45		62,36			
Mg	K series	0,3	0,06	0,25	MgO	0,49	MgO
Al	K series	29,83	0,21	22,77	Al ₂ O ₃	56,36	Al ₂ O ₃
Si	K series	17,92	0,19	13,14	SiO ₂	38,34	SiO ₂
K	K series	0,23	0,06	0,12	K ₂ O	0,27	KBr
Ca	K series	0,78	0,07	0,4	CaO	1,09	Wollastonite
Ti	K series	0,6	0,08	0,26	TiO ₂	1	Ti
Fe	K series	1,9	0,13	0,7	FeO	2,45	Fe
Total:		100		100		100	

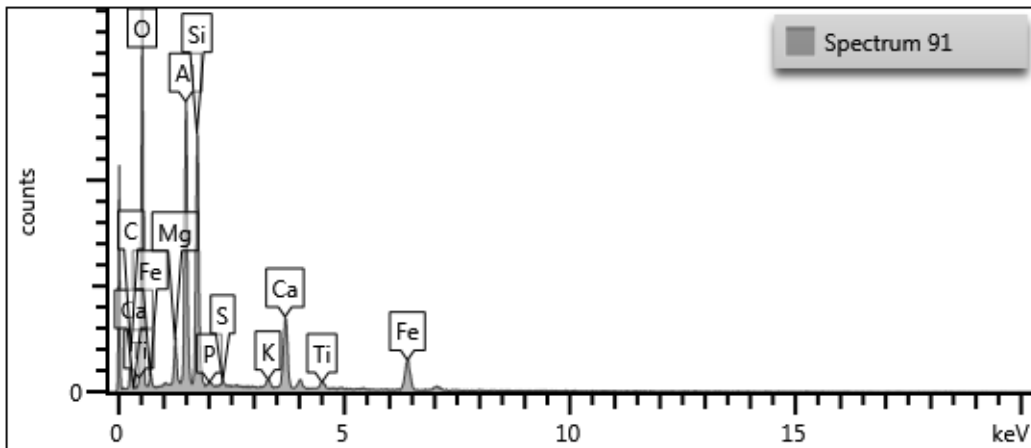
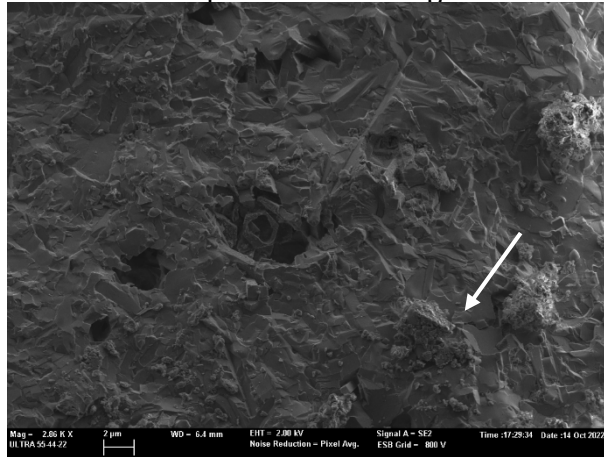
Sample: Refractory ceramic material

Ref: 1

Sampling point: external side



Chemical composition: clayey minerals and quartz that form the ceramic body, accompanied of calcite grain



Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %	Standard Label
O		43,78		60,73			
Mg	K series	2,95	0,11	2,69	MgO	4,88	MgO
Al	K series	16,75	0,19	13,78	Al ₂ O ₃	31,66	Al ₂ O ₃
Si	K series	16,97	0,2	13,41	SiO ₂	36,3	SiO ₂
P	K series	0,36	0,08	0,26	P ₂ O ₅	0,82	GaP
S	K series	0,34	0,07	0,23	SO ₃	0,85	FeS ₂
K	K series	0,77	0,07	0,44	K ₂ O	0,93	KBr
Ca	K series	7,71	0,15	4,27	CaO	10,79	Wollastonite
Ti	K series	1,13	0,1	0,52	TiO ₂	1,88	Ti
Fe	K series	9,25	0,24	3,68	FeO	11,9	Fe
Total:		100		100		100	

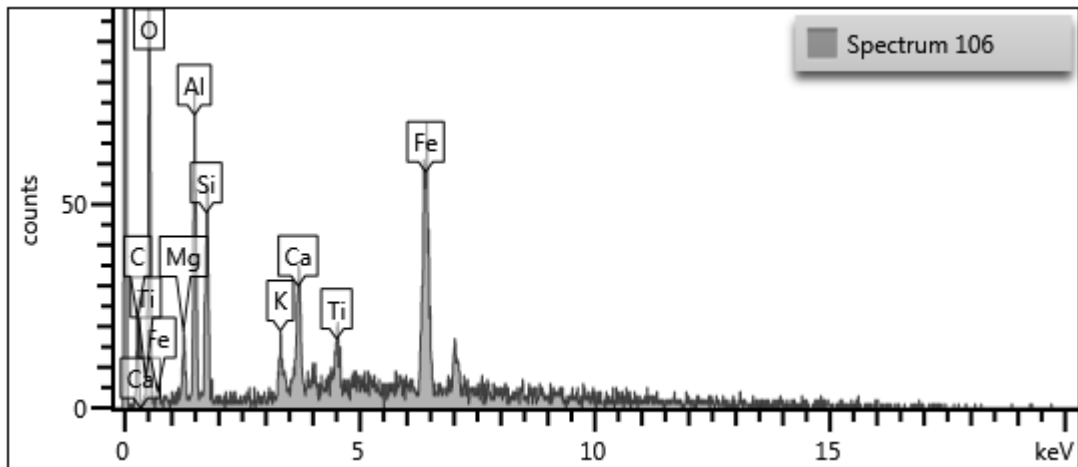
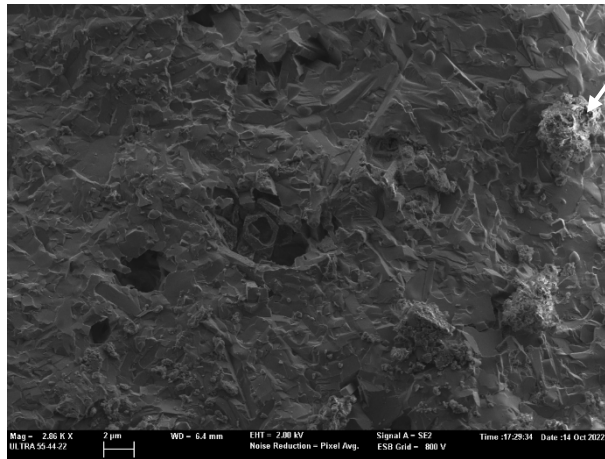
Sample: Refractory ceramic material

Ref: 1

Sampling point: external side



Chemical composition: Fe-rich nodule



Element	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %	Standard Label
O	34		56,56			
Mg	2,57	0,53	2,81	MgO	4,25	MgO
Al	10,23	0,74	10,1	Al ₂ O ₃	19,34	Al ₂ O ₃
Si	7,1	0,64	6,73	SiO ₂	15,19	SiO ₂
K	2,06	0,44	1,4	K ₂ O	2,48	KBr
Ca	5,95	0,64	3,95	CaO	8,32	Wollastonite
Ti	3,68	0,73	2,05	TiO ₂	6,14	Ti
Fe	34,41	1,65	16,4	FeO	44,27	Fe
Total:	100		100		100	

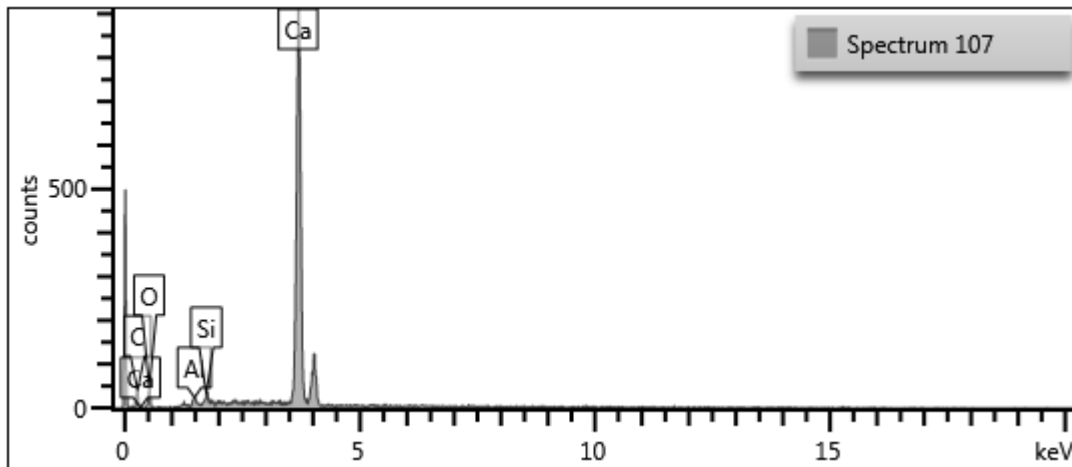
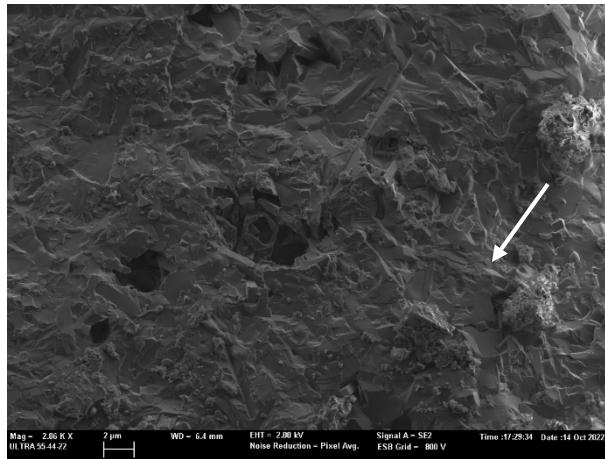
Sample: Refractory ceramic material

Ref: 1

Sampling point: external side



Chemical composition: calcite



Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %	Standard Label
O		29,31		50,66			
Al	K series	0,85	0,18	0,87	Al ₂ O ₃	1,6	Al ₂ O ₃
Si	K series	0,91	0,19	0,89	SiO ₂	1,94	SiO ₂
Ca	K series	68,94	0,62	47,58	CaO	96,46	Wollastonite
Total:		100		100		100	

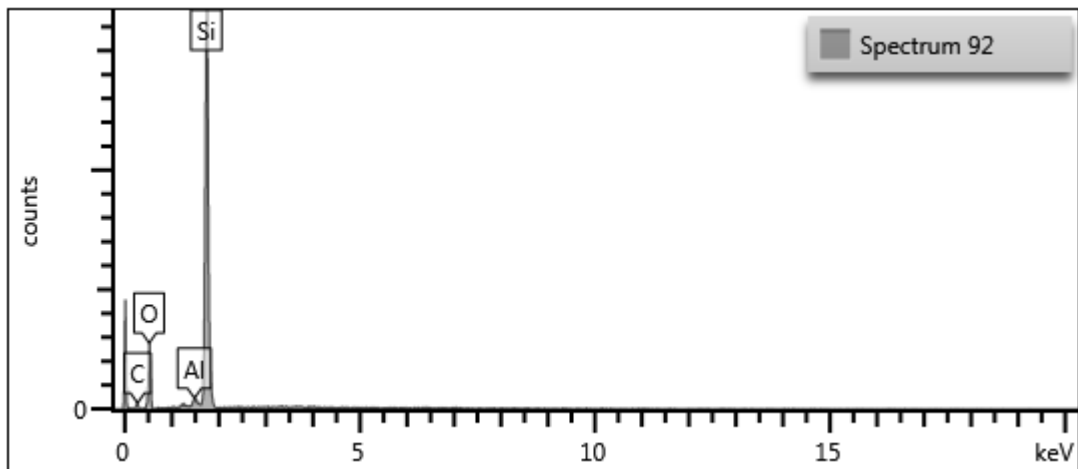
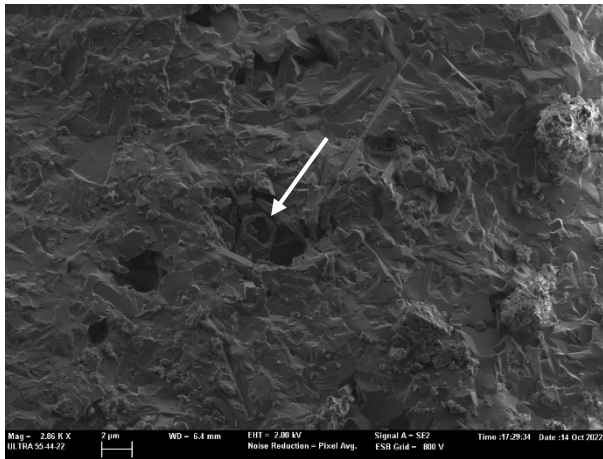
Sample: Refractory ceramic material

Ref: 1

Sampling point: external side



Chemical composition: quartz grain



Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %	Standard Label
O		53,17		66,58			
Al	K series	0,71	0,16	0,53	Al2O3	1,34	Al2O3
Si	K series	46,12	0,48	32,89	SiO2	98,66	SiO2
Total:		100		100		100	

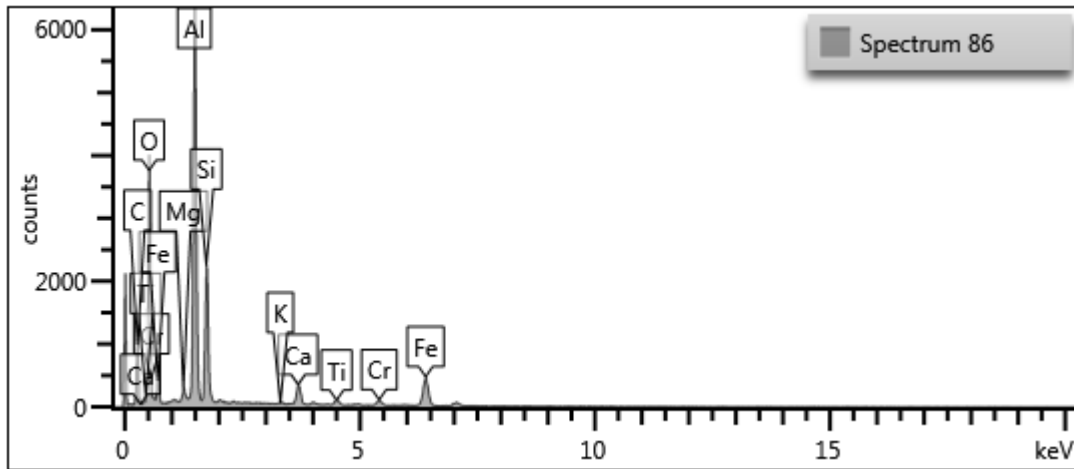
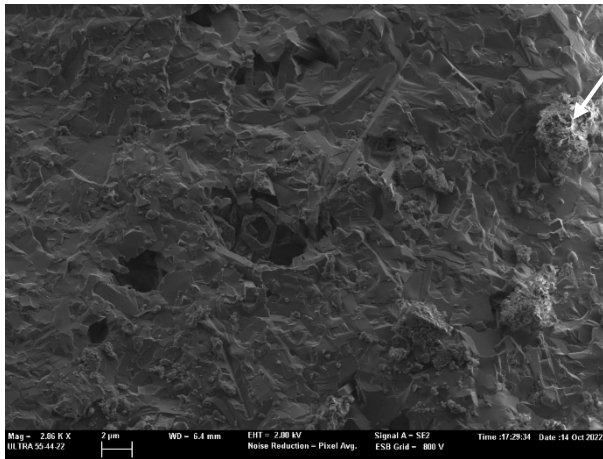
Sample: Refractory ceramic material

Ref: 1

Sampling point: external side



Chemical composition: altered ceramic body

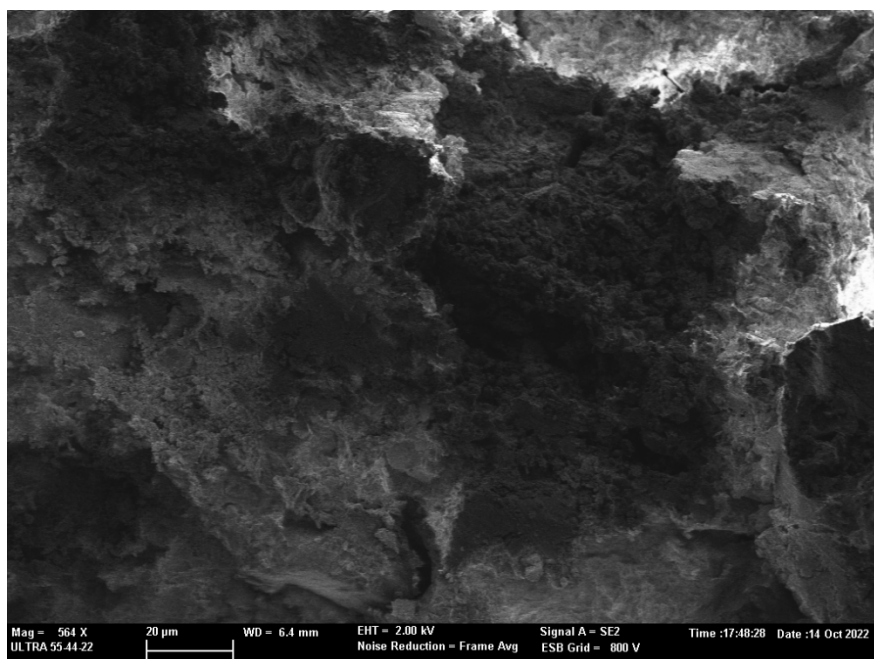
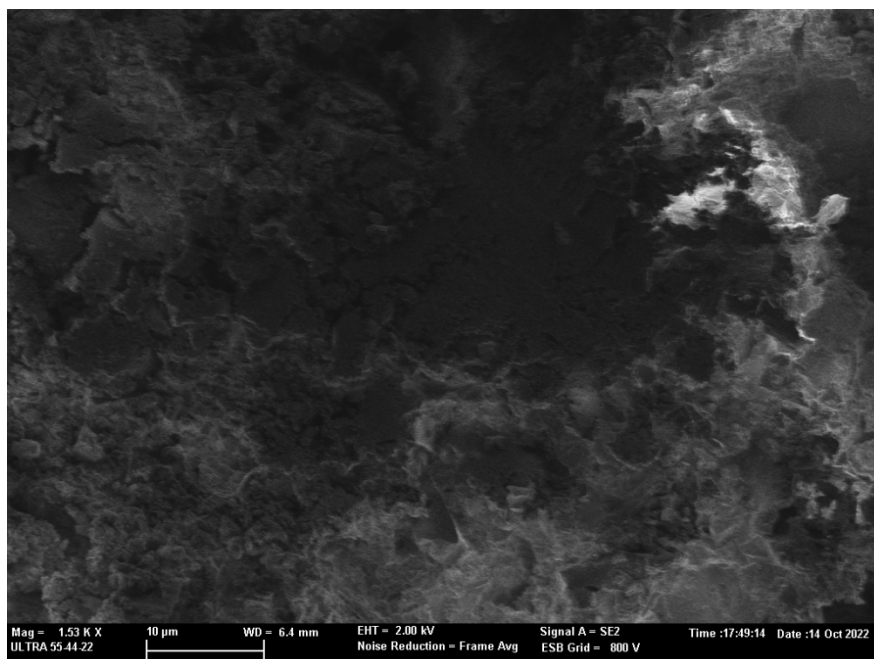


Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %	Standard Label
O		44,28		60,76			
Mg	K series	0,9	0,07	0,81	MgO	1,49	MgO
Al	K series	27,33	0,2	22,24	Al ₂ O ₃	51,64	Al ₂ O ₃
Si	K series	12,47	0,16	9,75	SiO ₂	26,68	SiO ₂
K	K series	0,18	0,05	0,1	K ₂ O	0,22	KBr
Ca	K series	2,58	0,08	1,41	CaO	3,61	Wollastonite
Ti	K series	0,97	0,08	0,45	TiO ₂	1,62	Ti
Cr	K series	1,25	0,09	0,53	Cr ₂ O ₃	1,83	Cr
Fe	K series	10,03	0,2	3,94	FeO	12,91	Fe
Total:		100		100		100	

Sample: Efflorescences
Ref: 2
Sampling point: external side

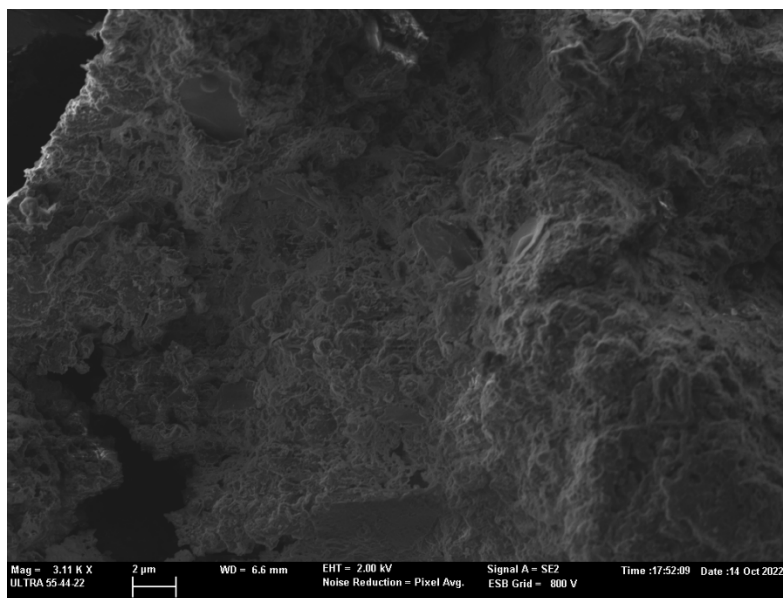
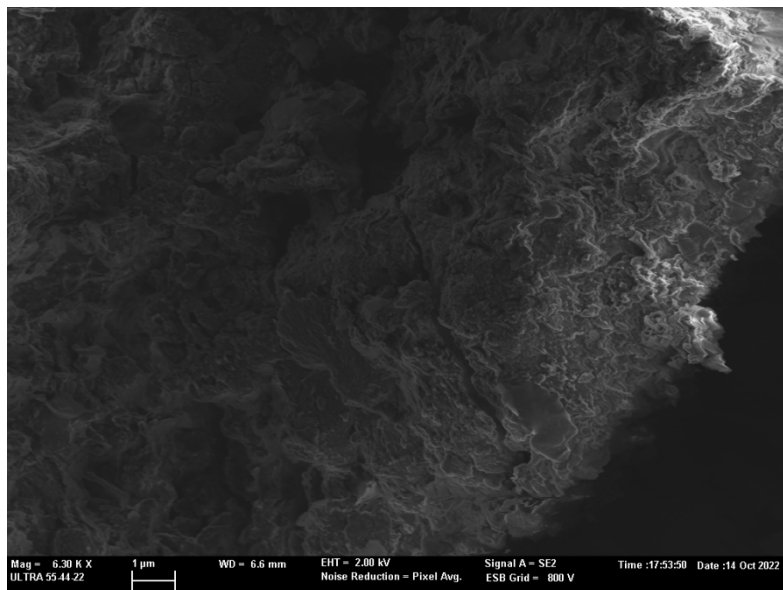


Secondary electron images



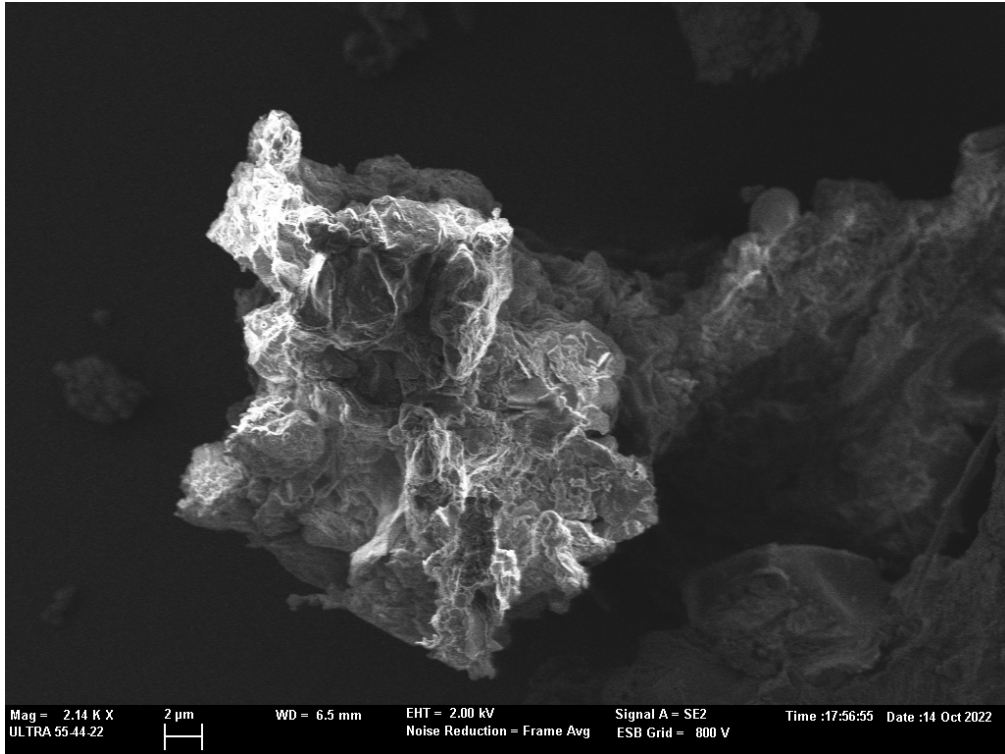
View at low magnification of White efflorescences formed on the outer surface in the bottom of the crucible

Sample: Efflorescences
Ref: 2
Sampling point: external side



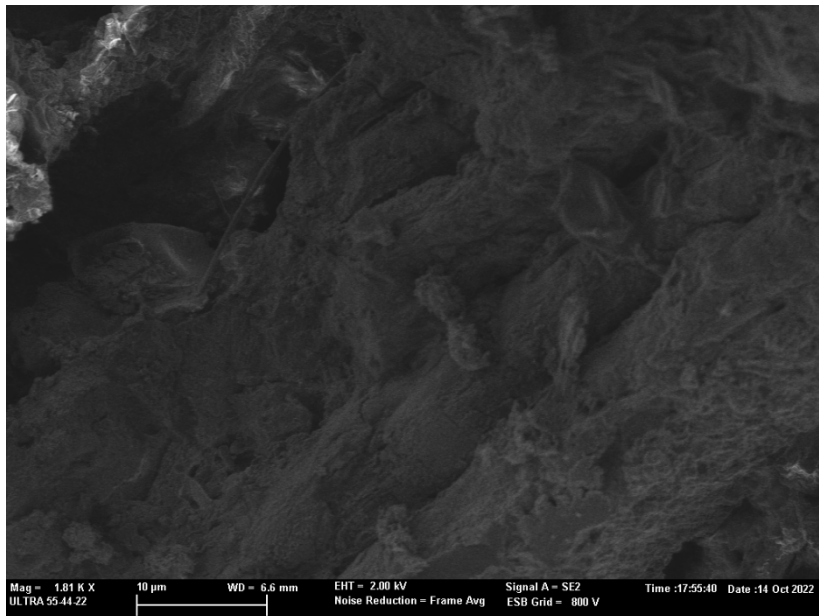
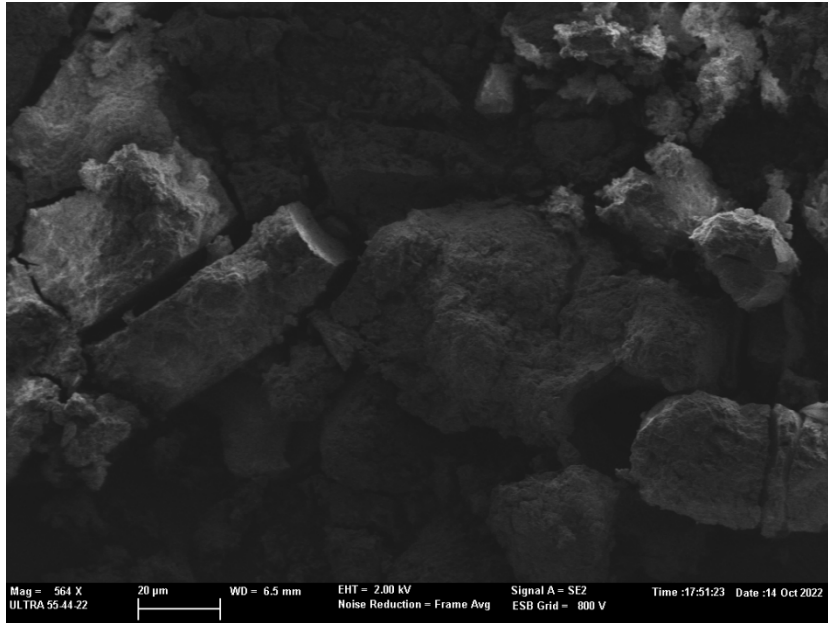
View at low magnification of the efflorescences

Sample: Efflorescences
Ref: 2
Sampling point: external side



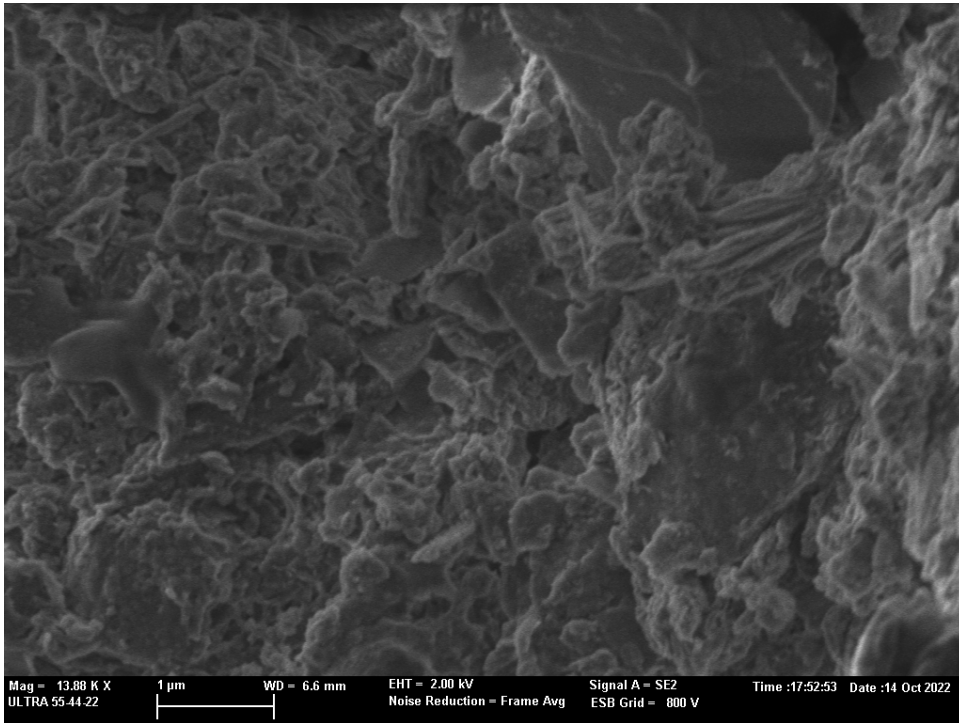
Detail of the morphology of the efflorescences

Sample: Efflorescences
Ref: 2
Sampling point: external side



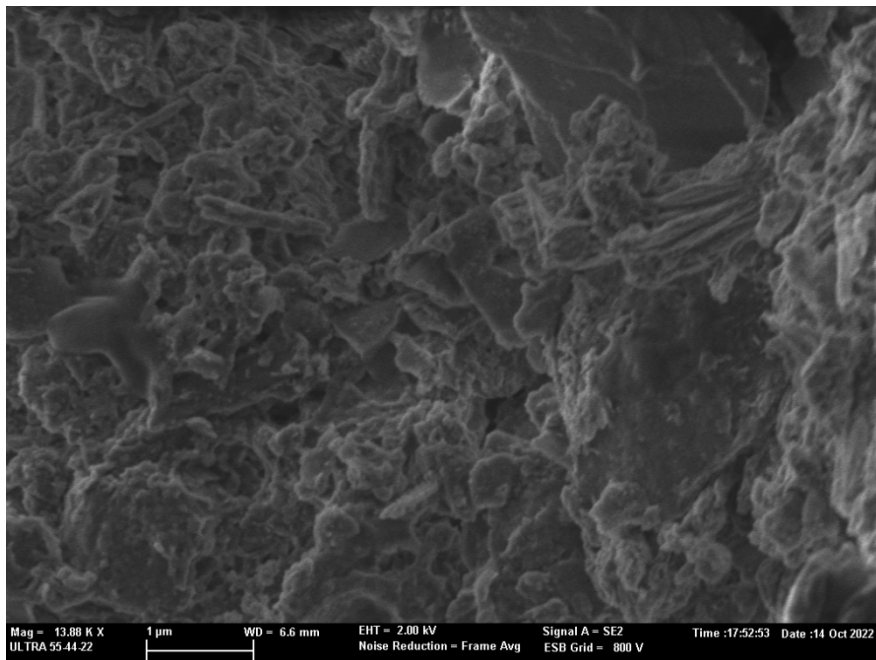
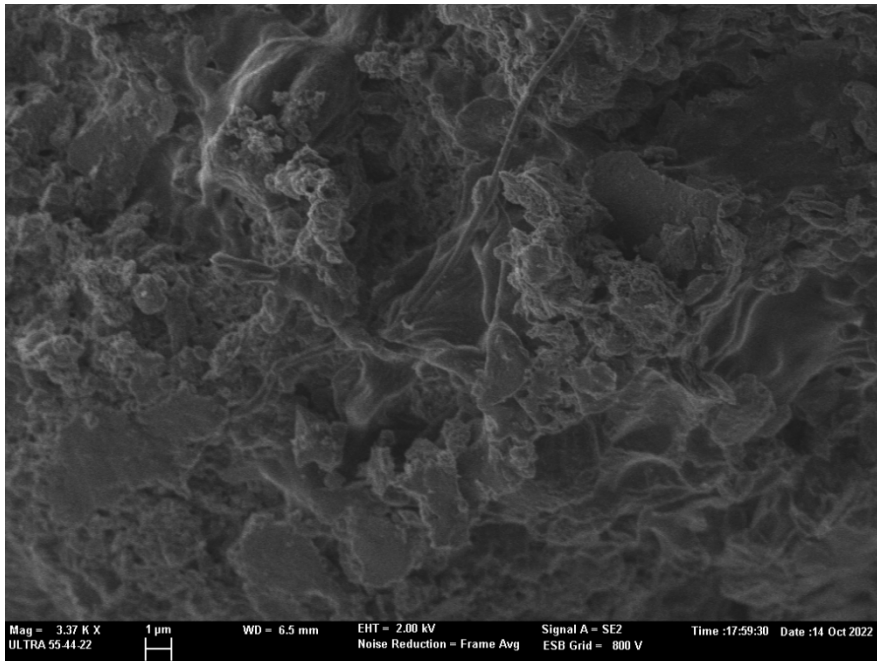
Images of aggregates with columnar shape

Sample: Efflorescences
Ref: 2
Sampling point: external side



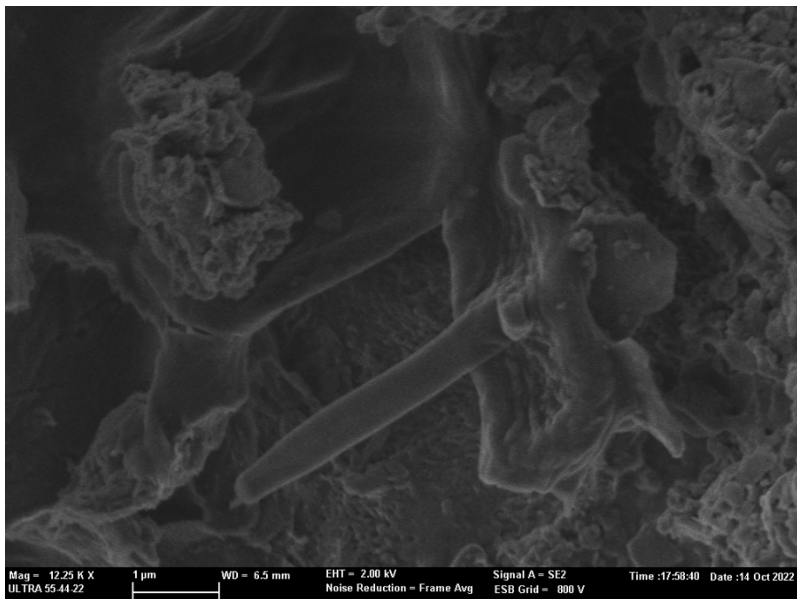
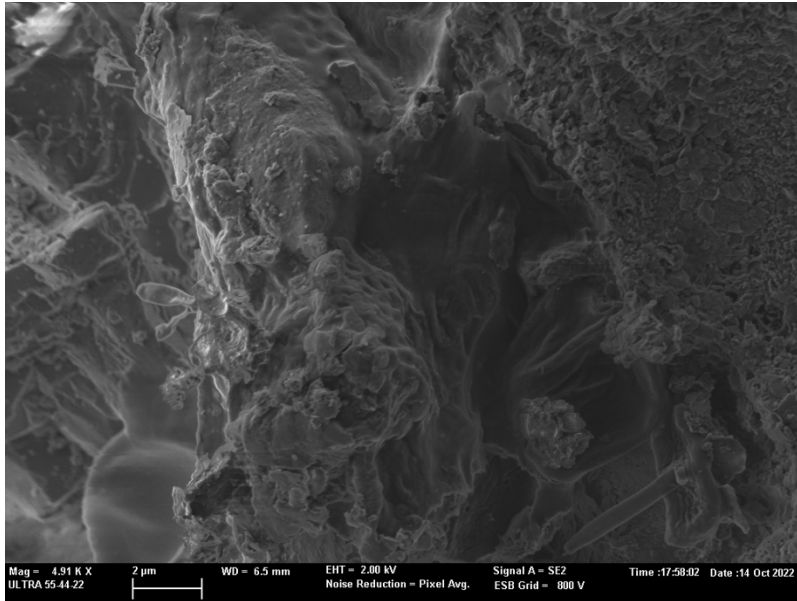
Images of organic material infiltrated in the efflorescences
mass of criptocrystalline morphology

Sample: Efflorescences
Ref: 2
Sampling point: external side



Spores and hyphae of fungi infiltrated in the efflorescences mass of criptocrystalline morphology

Sample: Efflorescences
Ref: 2
Sampling point: external side



Spores and hyphae of fungi infiltrated in the efflorescences mass of
criptocrystalline morphology

Sample: Efflorescences
Ref: 2
Sampling point: external side

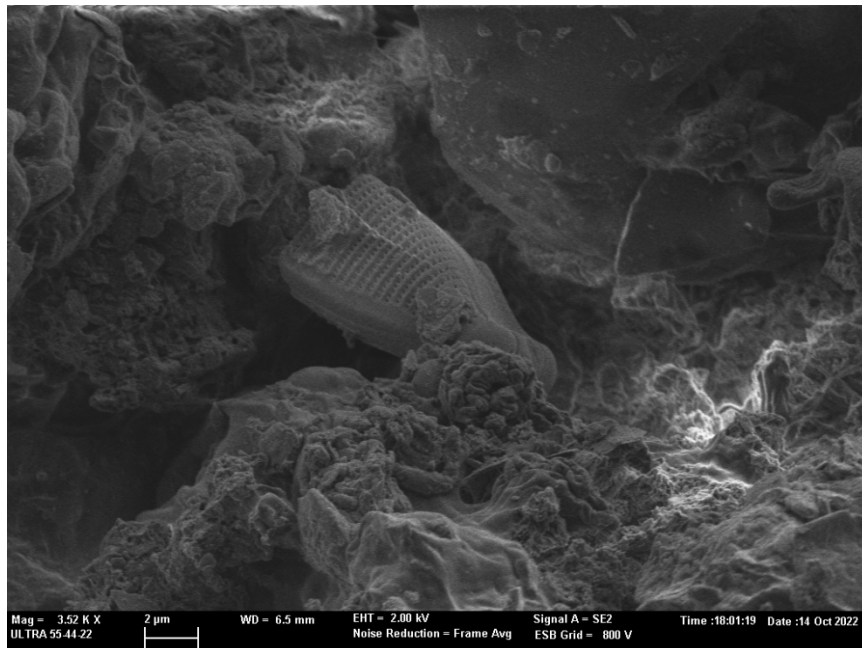
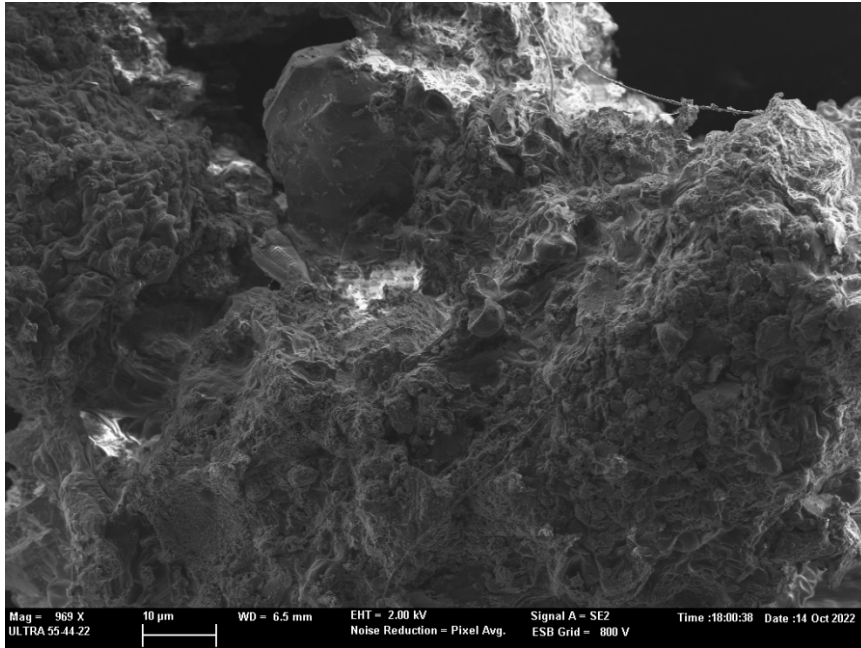
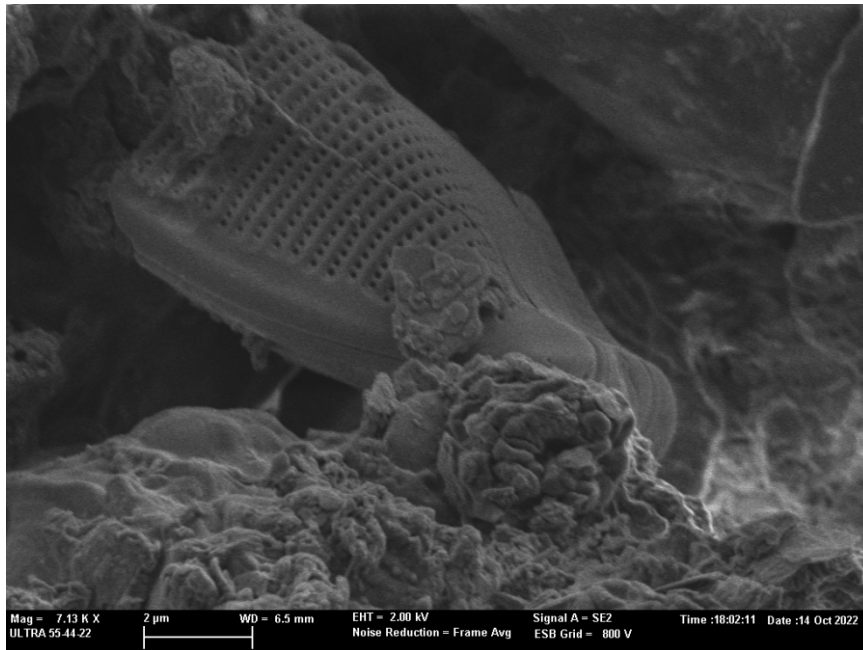


Image of an algae of diatomea type

Sample: Efflorescences
Ref: 2
Sampling point: external side



The morphology of the diatomea ascribes this specimen to navicula type

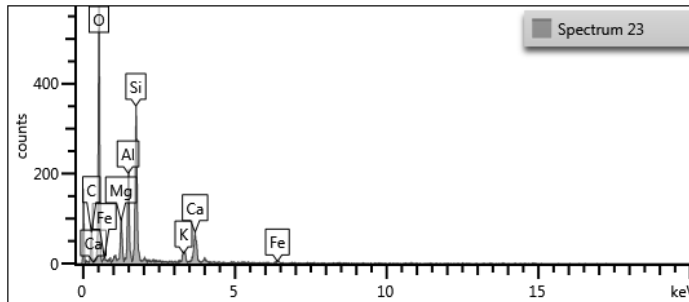
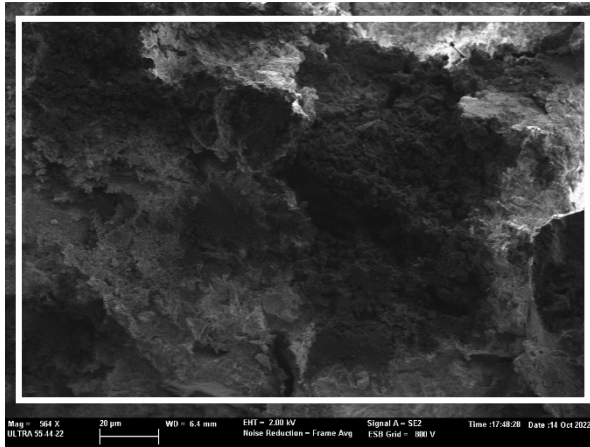
Sample: Efflorescences

Ref: 2

Sampling point: external side



Chemical composition: clayey minerals



Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %	Standard Label
O		46,03		60,96			
Mg	K series	6,07	0,43	5,29	MgO	10,07	MgO
Al	K series	13,1	0,55	10,29	Al ₂ O ₃	24,76	Al ₂ O ₃
Si	K series	23,17	0,69	17,48	SiO ₂	49,57	SiO ₂
K	K series	2,58	0,34	1,4	K ₂ O	3,11	KBr
Ca	K series	7,63	0,5	4,04	CaO	10,68	Wollastonite
Fe	K series	1,41	0,41	0,54	FeO	1,82	Fe
Total:		100		100		100	
Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %	Standard Label
O		38,41		57,86			
Mg	K series	3,96	0,13	3,93	MgO	6,57	MgO
Al	K series	2,06	0,1	1,84	Al ₂ O ₃	3,89	Al ₂ O ₃
Si	K series	7,93	0,15	6,8	SiO ₂	16,96	SiO ₂
P	K series	5,51	0,14	4,29	P ₂ O ₅	12,64	GaP
S	K series	1,1	0,08	0,83	SO ₃	2,76	FeS ₂
K	K series	0,91	0,08	0,56	K ₂ O	1,1	KBr
Ca	K series	38,59	0,27	23,21	CaO	54	Wollastonite
Ti	K series	0,35	0,1	0,18	TiO ₂	0,59	Ti
Fe	K series	1,17	0,15	0,5	FeO	1,5	Fe
		100		100		100	

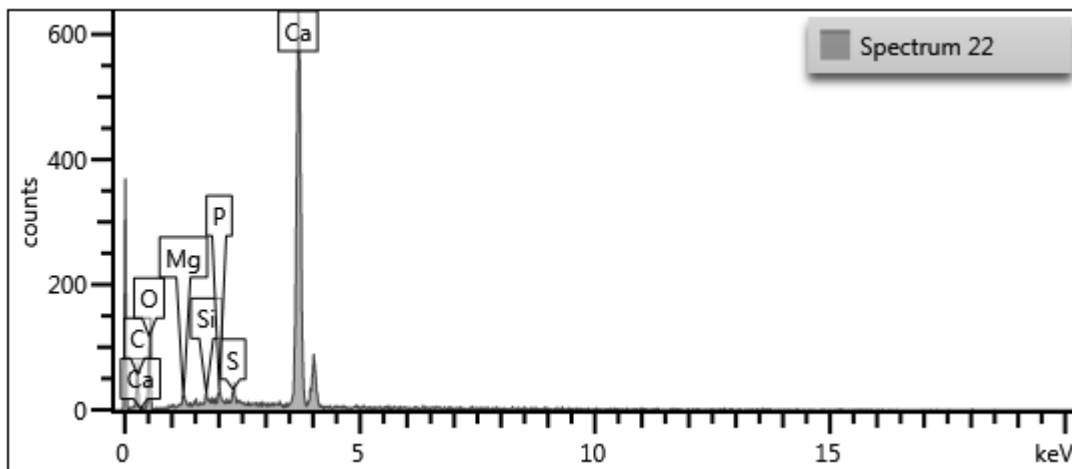
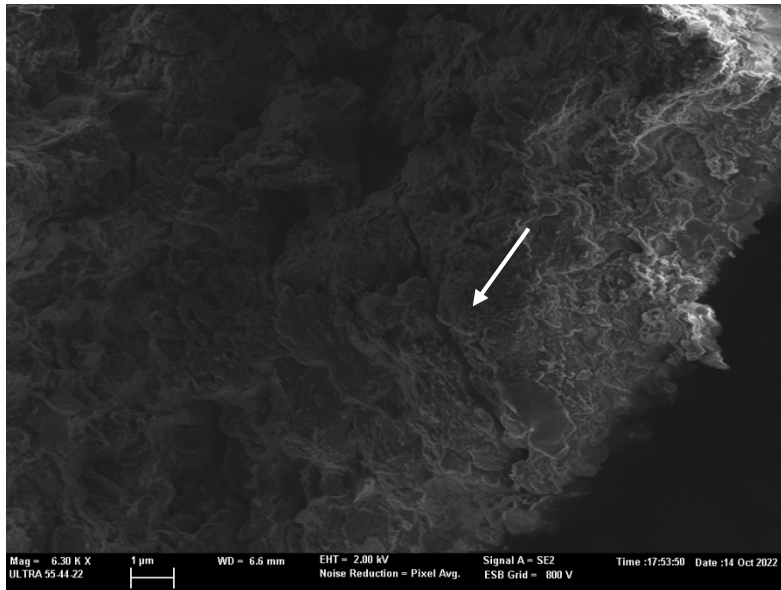
Sample: Efflorescences

Ref: 2

Sampling point: external side



Chemical composition: calcite and dolomite accompanied of calcium hydroxyapatite, gypsum, clayey minerals



Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %	Standard Label
O		31,07		52,41			
Mg	K series	1,08	0,3	1,2	MgO	1,79	MgO
Si	K series	0,78	0,23	0,75	SiO2	1,67	SiO2
P	K series	1,23	0,27	1,07	P2O5	2,82	GaP
S	K series	1,46	0,26	1,23	SO3	3,64	FeS2
Ca	K series	64,38	0,82	43,34	CaO	90,07	Wollastonite
Total:		100		100		100	

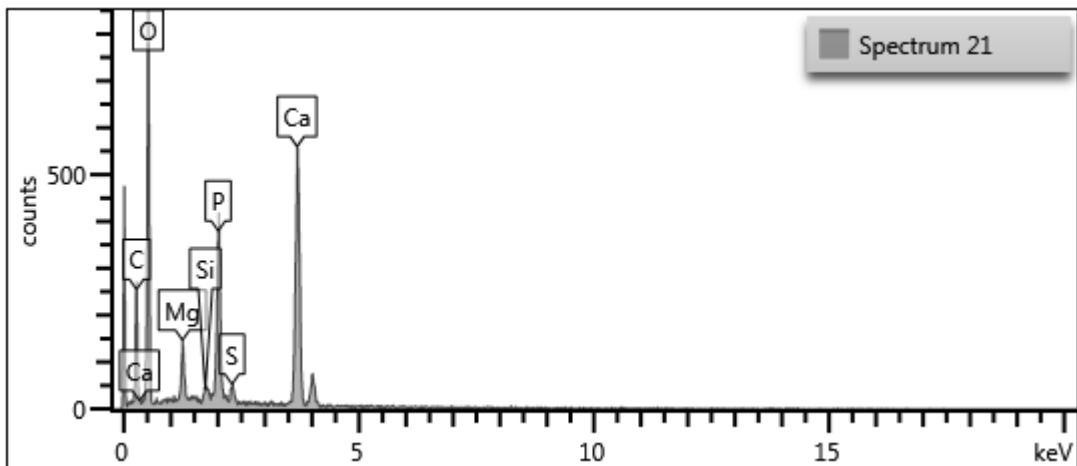
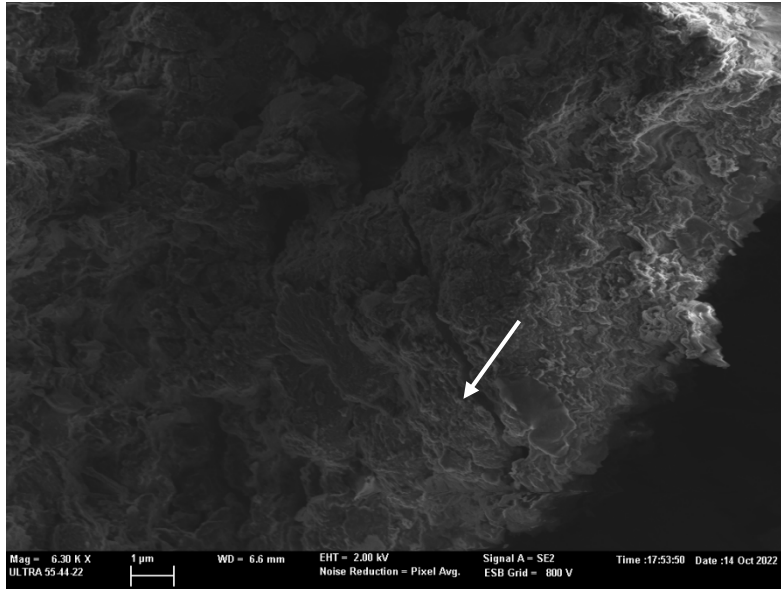
Sample: Efflorescences

Ref: 2

Sampling point: external side



Chemical composition: calcium hydroxyapatite, accompanied of calcite, dolomite, gypsum



Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %	Standard Label
O		41,05		60,37			
Mg	K series	4,99	0,36	4,83	MgO	8,27	MgO
Si	K series	0,88	0,21	0,74	SiO ₂	1,88	SiO ₂
P	K series	15,43	0,46	11,72	P ₂ O ₅	35,35	GaP
S	K series	1,65	0,23	1,21	SO ₃	4,11	FeS ₂
Ca	K series	36,01	0,62	21,14	CaO	50,39	Wollastonite
Total:		100		100		100	

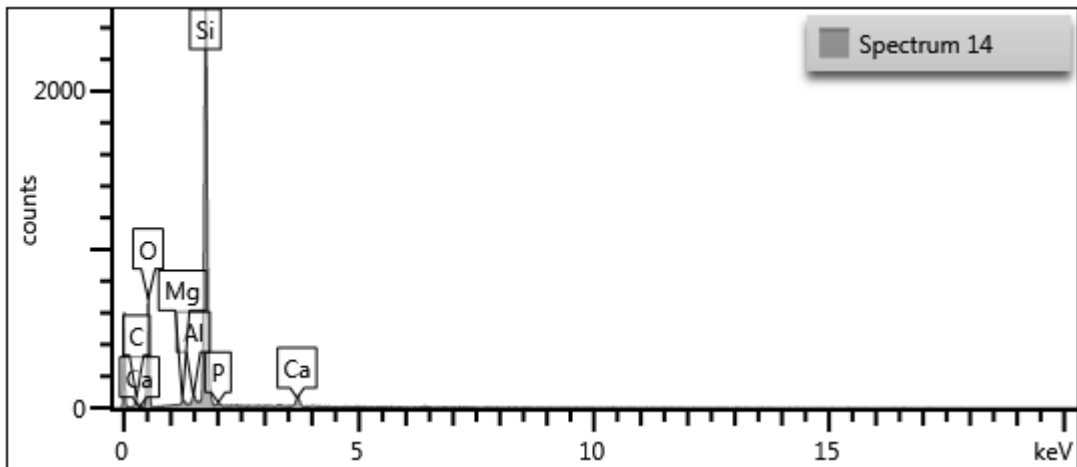
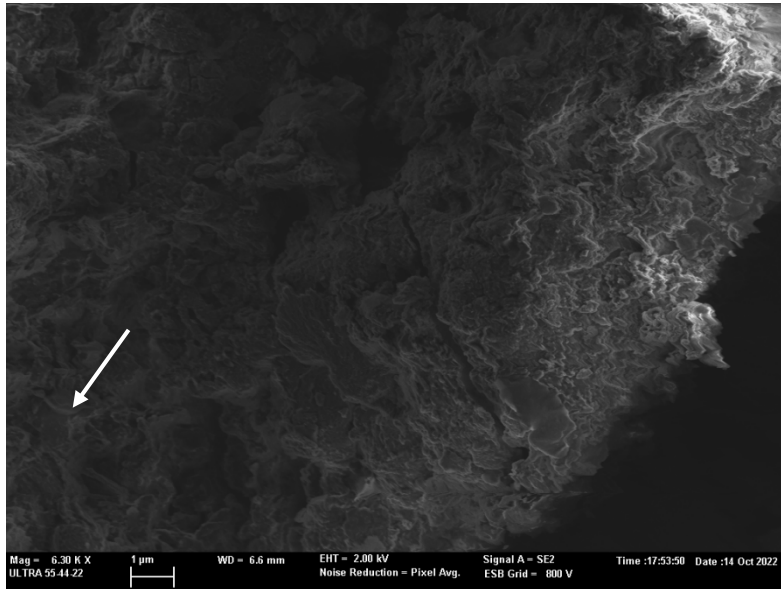
Sample: Efflorescences

Ref: 2

Sampling point: external side



Chemical composition: quartz accompanied of hydroxyapatites and clayey minerals



Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %	Standard Label
O		52,28		66,09			
Mg	K series	0,5	0,12	0,41	MgO	0,82	MgO
Al	K series	0,83	0,13	0,63	Al ₂ O ₃	1,58	Al ₂ O ₃
Si	K series	43,48	0,41	31,31	SiO ₂	93,01	SiO ₂
P	K series	0,57	0,15	0,38	P ₂ O ₅	1,32	GaP
Ca	K series	2,34	0,17	1,18	CaO	3,28	Wollastonite
Total:		100		100		100	

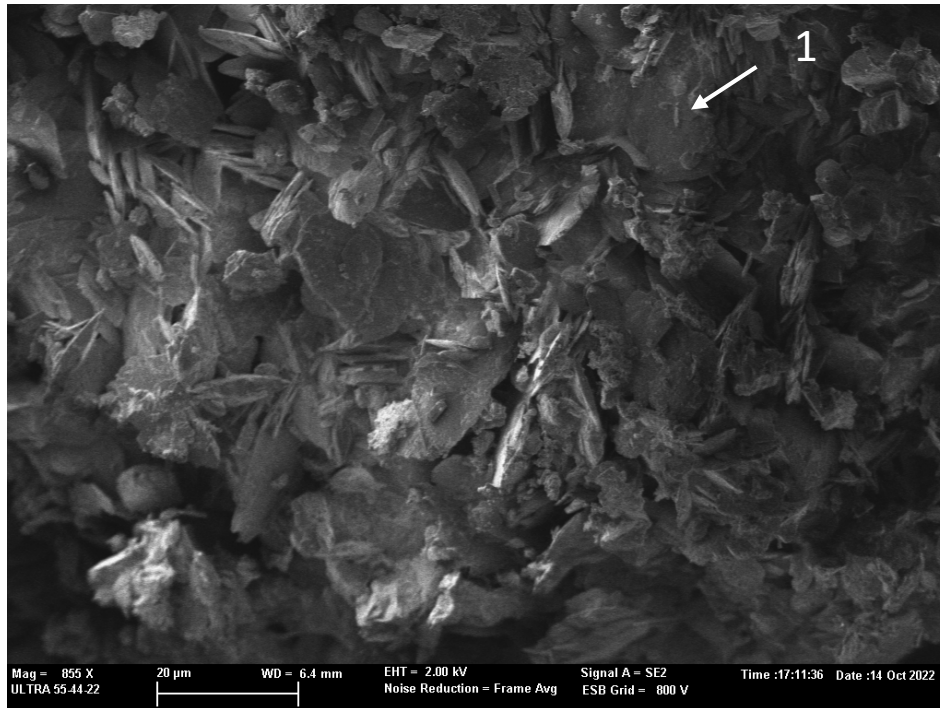
Sample: filling material in hole

Ref: 3

Sampling point: inner surface



Secondary electron images



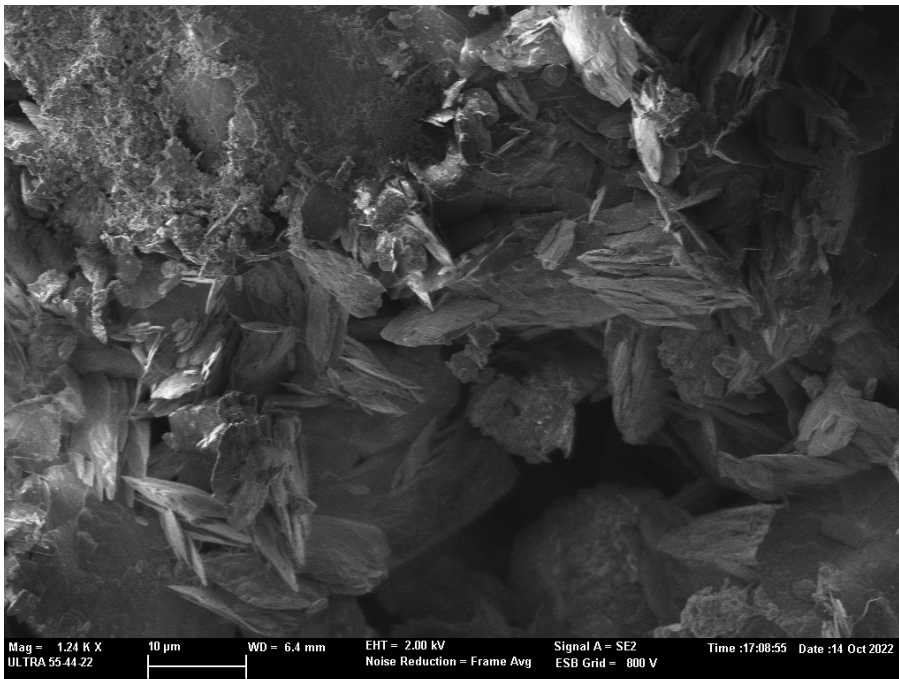
Cryptocrystalline mass of subeuhedral grains of gypsum with typical tabular shape forming aggregates. Acicular needles of gypsum (1).

Description: laminar crystals of gypsum forming plano-parallel and rose-like clusters that also include cryptocrystalline quartz. The gypsum crystals are flattened on the c axis, fanning open in radiating rosettes or planar aggregates. These rosette crystal habit tends to occur when the crystals form in dryness sandy conditions.

Sample: filling material in hole
Ref: 3
Sampling point: inner surface



Secondary electron images

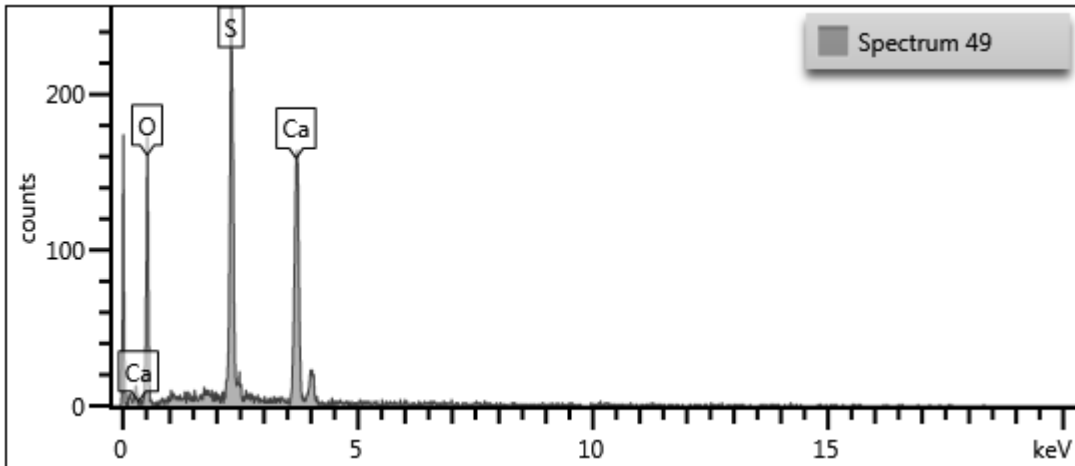
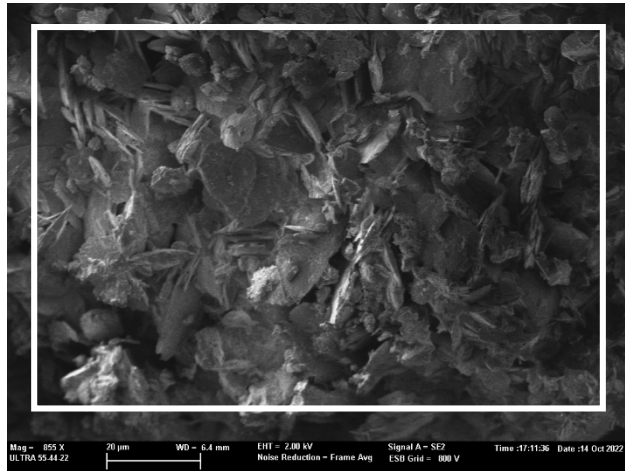


Description: lamellar crystals of gypsum forming plano-parallel and rose-like clusters that also include cryptocrystalline quartz. The gypsum crystals are flattened on the c axis, fanning open in radiating rosettes or planar aggregates. These rosette crystal habit tends to occur when the crystals form in dryness sandy conditions.

Sample: filling material in hole
Ref: 3
Sampling point: inner surface



Chemical composition: gypsum

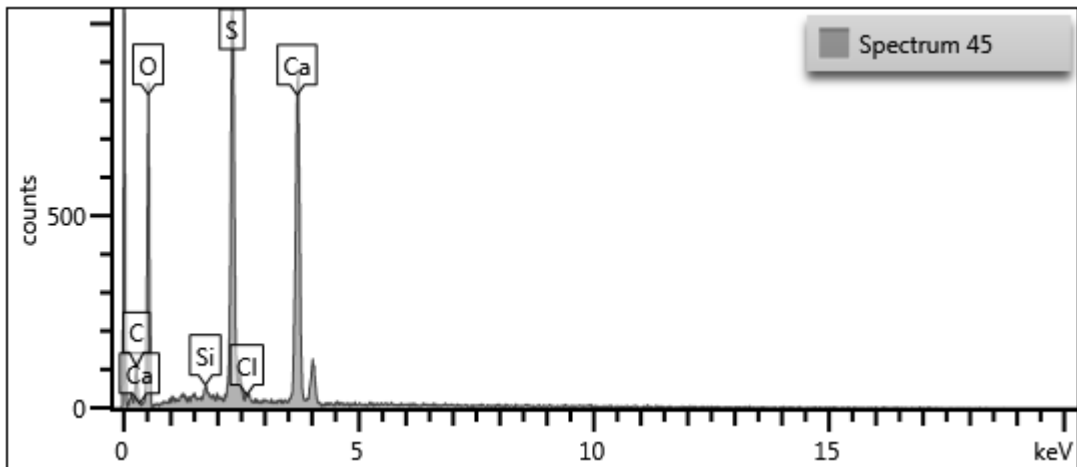
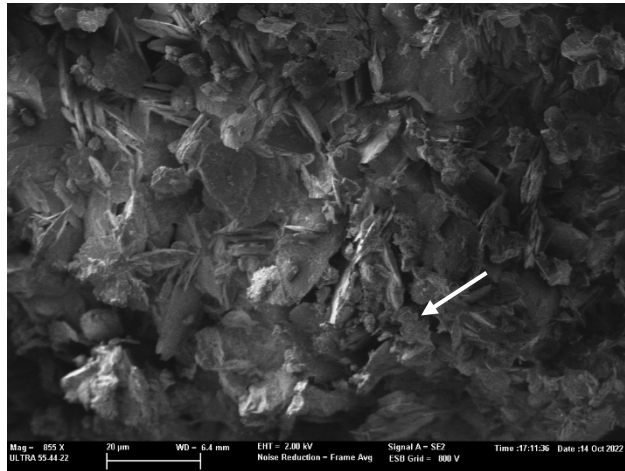


Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %	Standard Label
O		46,86		66,56			
S	K series	23,36	0,76	16,56	SO3	58,34	FeS2
Ca	K series	29,78	0,88	16,88	CaO	41,66	Wollastonite
Total:		100		100		100	

Sample: filling material in hole
Ref: 3
Sampling point: inner surface



Chemical composition: gypsum accompanied of chloride salts and siliceous minerals

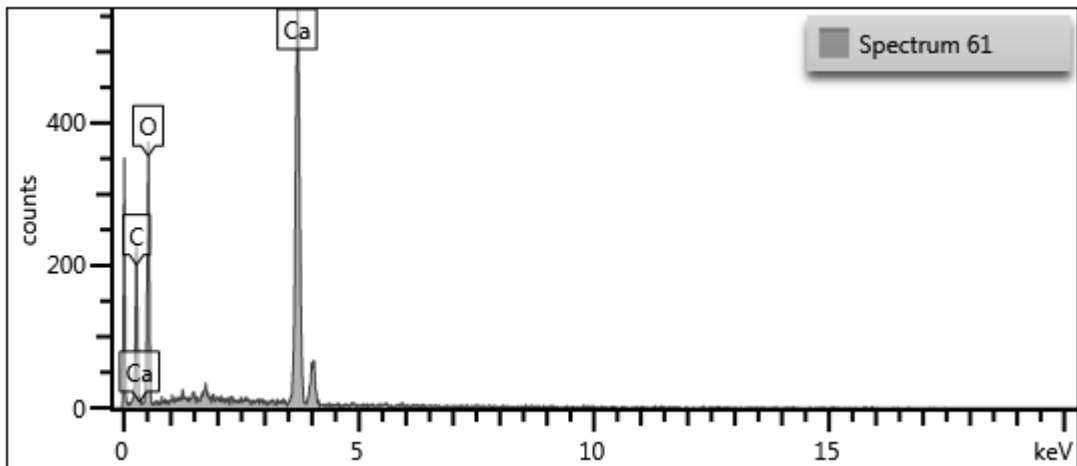
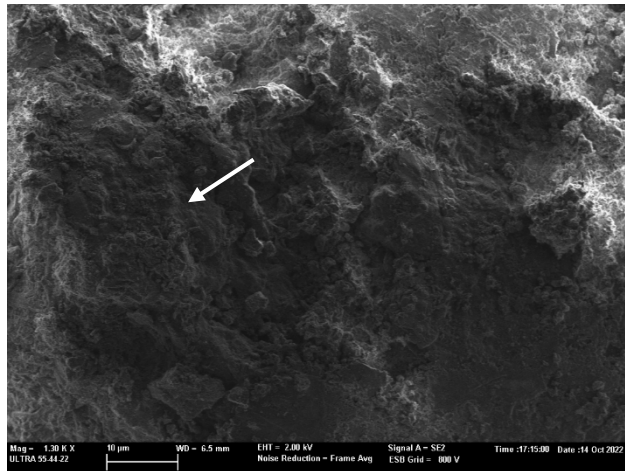


Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %	Standard Label
O		45,56		65,5			
Si	K series	0,54	0,12	0,44	SiO ₂	1,15	SiO ₂
S	K series	21,54	0,35	15,45	SO ₃	53,79	FeS ₂
Cl	K series	0,55	0,13	0,35		0	NaCl
Ca	K series	31,81	0,42	18,25	CaO	44,51	Wollastonite
Total:		100		100		99,45	

Sample: filling material in hole
Ref: 3
Sampling point: inner surface



Chemical composition: calcite



Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %	Standard Label
O		28,53		50			
Ca	K series	71,47	0,76	50	CaO	100	Wollastonite
Total:		100		100		100	

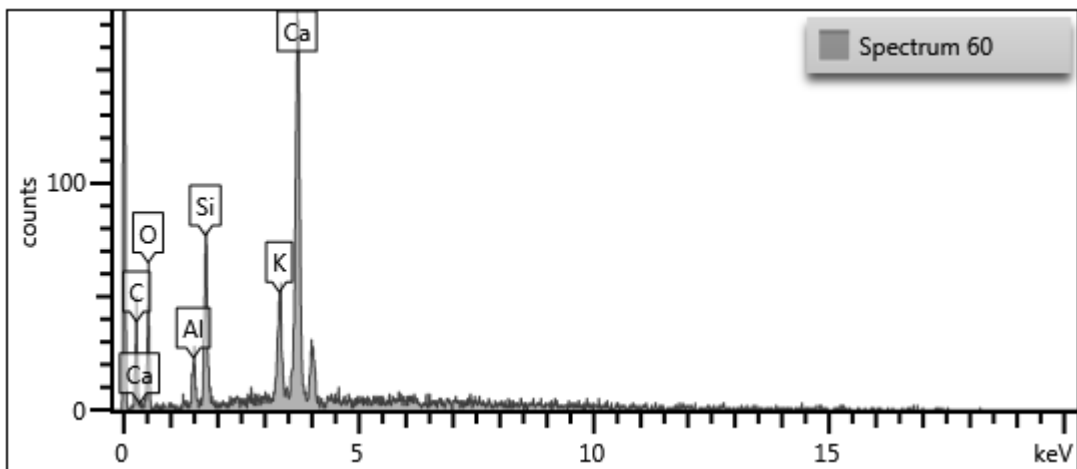
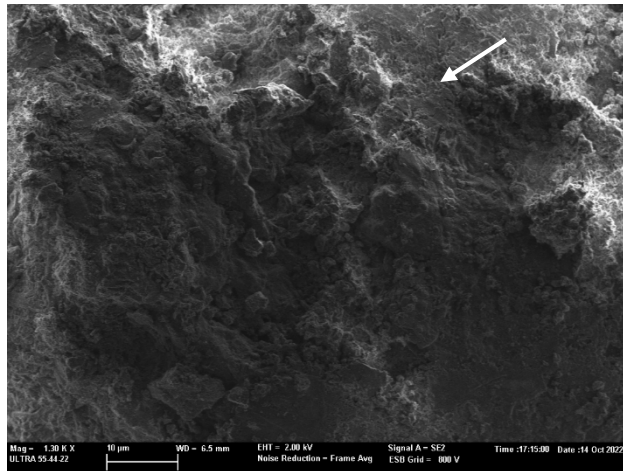
Sample: filling material in hole

Ref: 3

Sampling point: inner surface



Chemical composition: calcite accompanied of clayey minerals

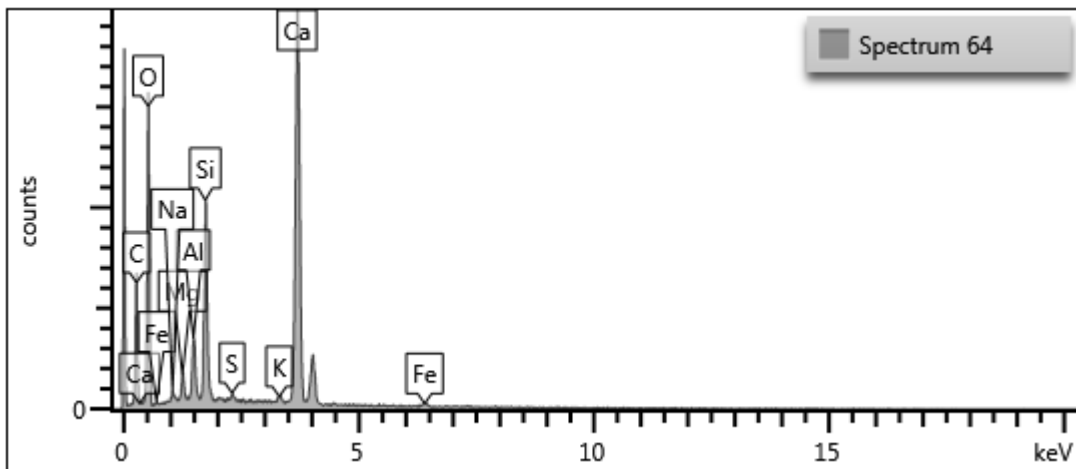
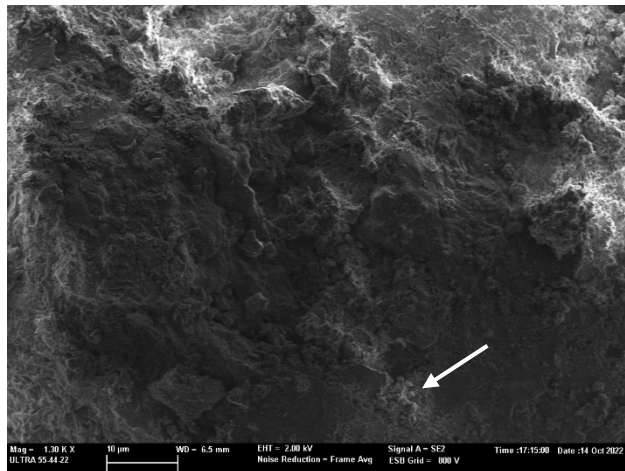


Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %	Standard Label
O		33,7		53,86			
Al	K series	3,22	0,47	3,05	Al ₂ O ₃	6,08	Al ₂ O ₃
Si	K series	9,97	0,7	9,07	SiO ₂	21,33	SiO ₂
K	K series	8,81	0,8	5,76	K ₂ O	10,61	KBr
Ca	K series	44,3	1,26	28,26	CaO	61,98	Wollastonite
Total:		100		100		100	

Sample: filling material in hole
Ref: 3
Sampling point: inner surface



Chemical composition: calcite accompanied of clayey minerals

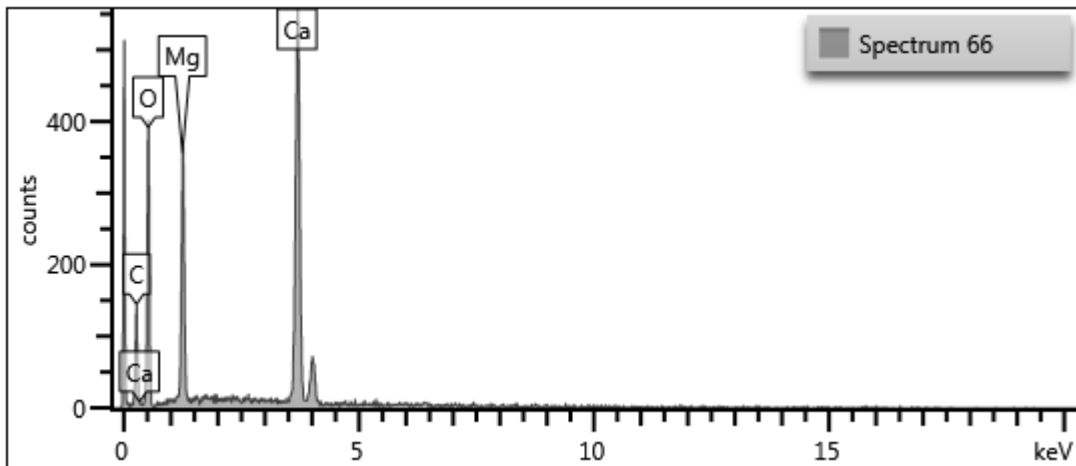
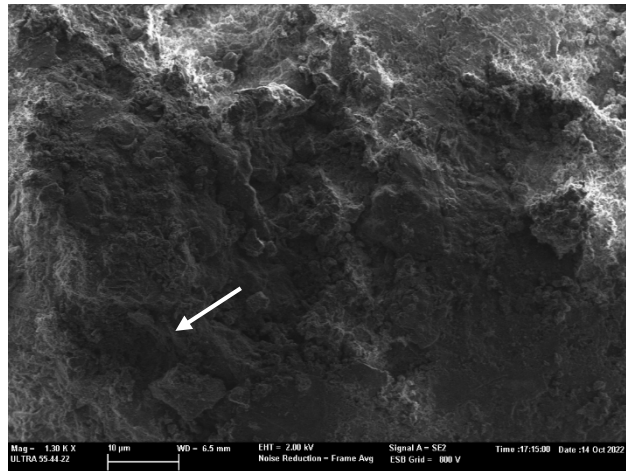


Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %	Standard Label
O		36,55		55,99			
Na	K series	0,77	0,17	0,82	Na ₂ O	1,04	Albite
Mg	K series	1,96	0,15	1,97	MgO	3,25	MgO
Al	K series	3,86	0,17	3,5	Al ₂ O ₃	7,29	Al ₂ O ₃
Si	K series	11,73	0,23	10,24	SiO ₂	25,1	SiO ₂
S	K series	0,39	0,1	0,3	SO ₃	0,98	FeS ₂
K	K series	0,62	0,11	0,39	K ₂ O	0,75	KBr
Ca	K series	42,96	0,38	26,27	CaO	60,11	Wollastonite
Fe	K series	1,16	0,21	0,51	FeO	1,49	Fe
Total:		100		100		100	

Sample: filling material in hole
Ref: 3
Sampling point: inner surface



Chemical composition: dolomite



Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %	Standard Label
O		32,33		50			
Mg	K series	20,55	0,6	20,91	MgO	34,07	MgO
Ca	K series	47,12	0,71	29,09	CaO	65,93	Wollastonite
Total:		100		100		100	

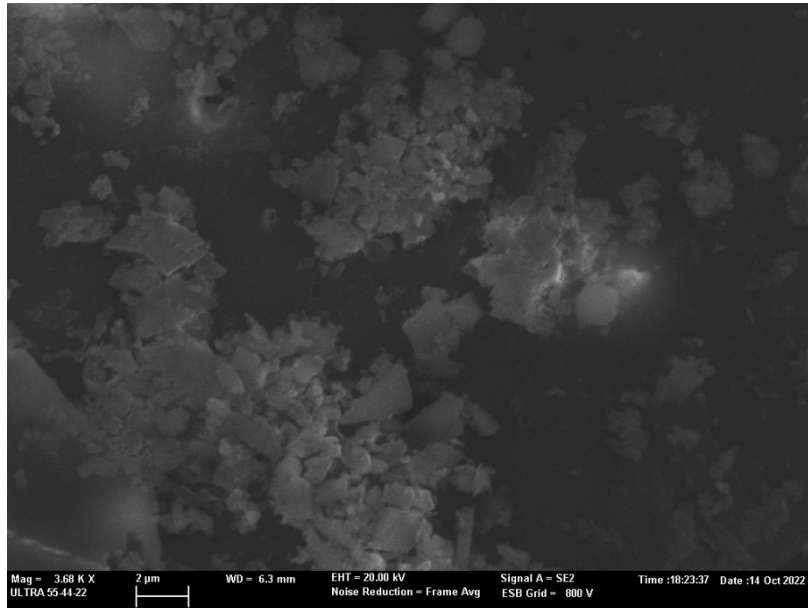
Sample: White spot in the bottom

Ref: 4

Sampling point: inner surface



Secondary electron images



Cryptocrystalline irregular deposits on the inner Surface in the bottom of the crucible

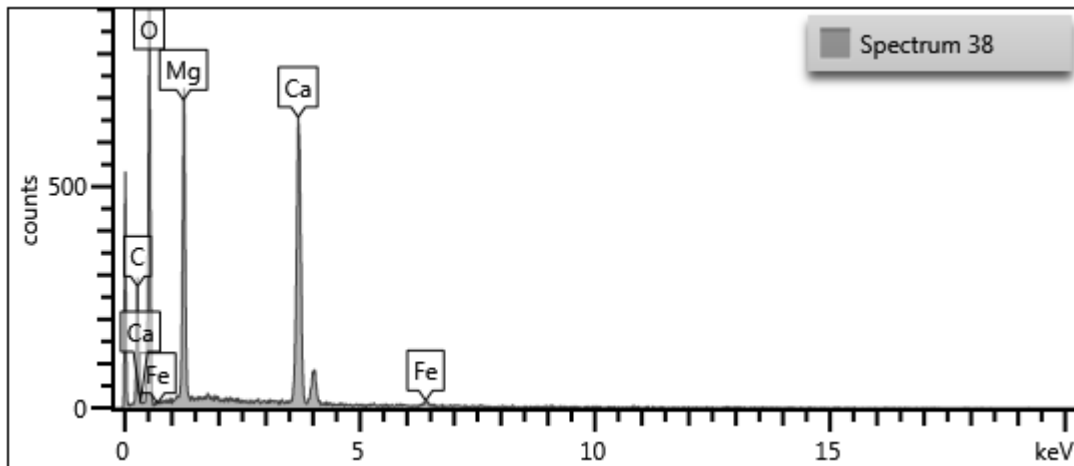
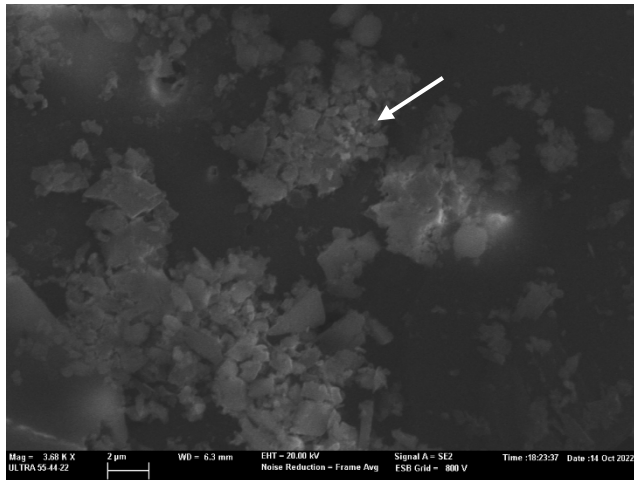
Sample: White spot in the bottom

Ref: 4

Sampling point: inner surface



Chemical composition: dolomite accompanied of iron oxide



Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %	Standard Label
O		33,23		50			
Mg	K series	26,17	0,54	25,92	MgO	43,4	MgO
Ca	K series	38,81	0,59	23,31	CaO	54,3	Wollastonite
Fe	K series	1,79	0,39	0,77	FeO	2,3	Fe
Total:		100		100		100	

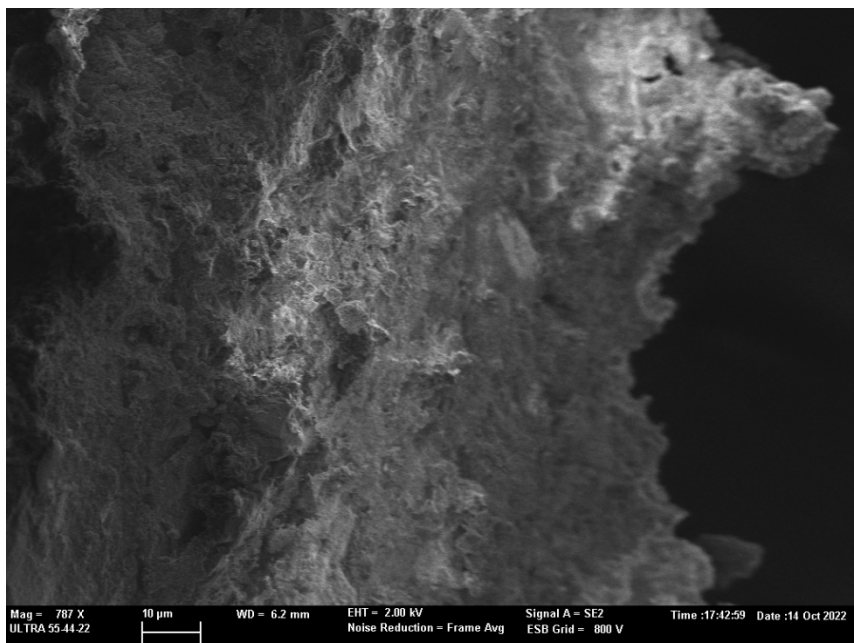
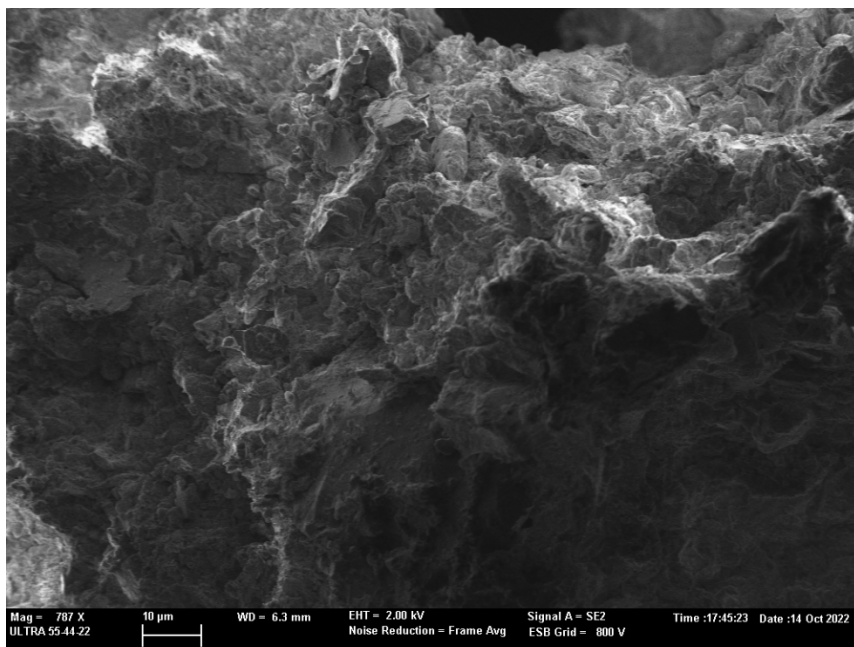
Sample: Brown-grey deposits

Ref: 5

Sampling point: inner side



Secondary electron images

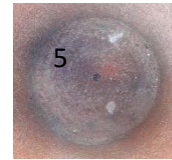


Cryptocrystalline aggregates of irregular grains deposited on the inner surface in the bottom of the crucible forming a black crust

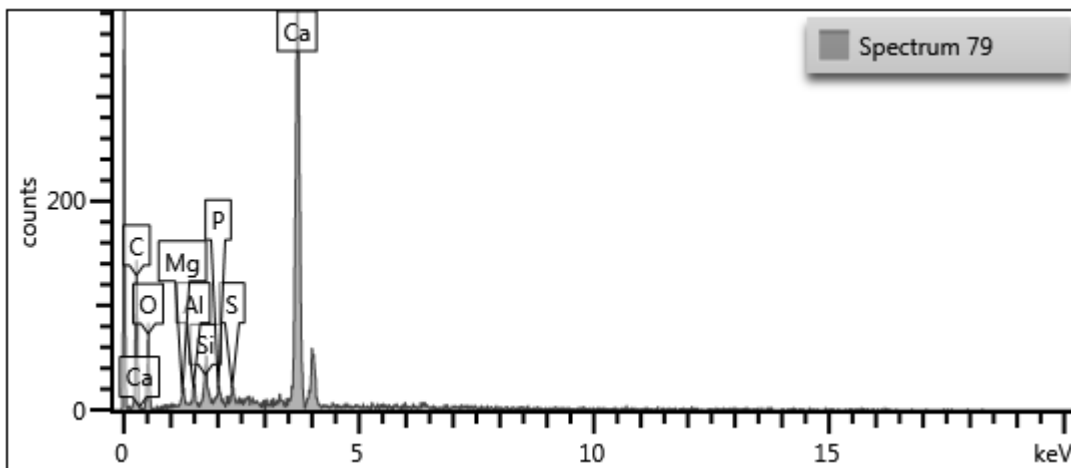
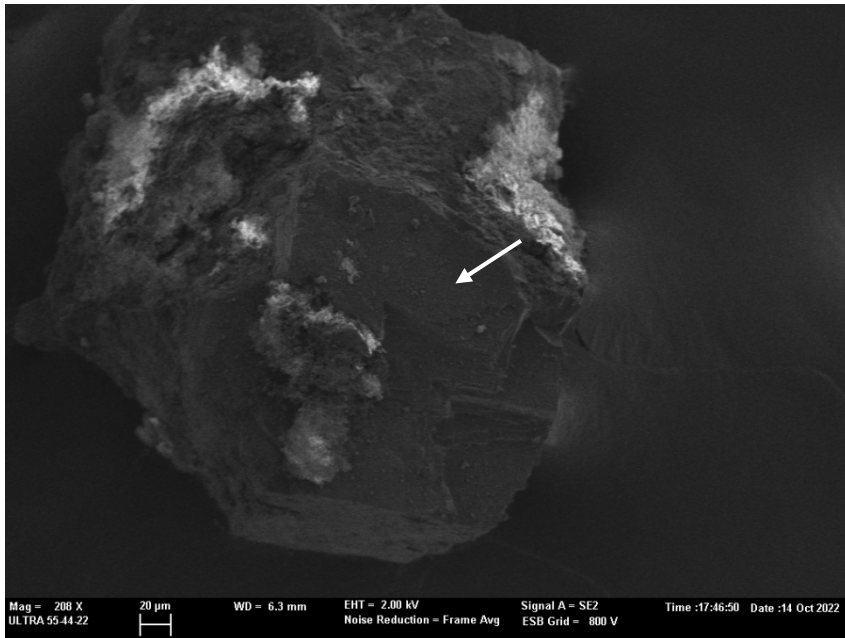
Sample: Brown-grey deposits

Ref: 5

Sampling point: inner side



Chemical composition: dolomite accompanied of clayey apatitic minerals



Element	Line Type	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %	Standard Label
O		32,86		53,66			
Mg	K series	1,61	0,36	1,73	MgO	2,68	MgO
Al	K series	1,89	0,32	1,83	Al2O3	3,58	Al2O3
Si	K series	3,12	0,39	2,9	SiO2	6,67	SiO2
P	K series	1,18	0,34	0,99	P2O5	2,7	GaP
S	K series	1,24	0,31	1,01	SO3	3,09	FeS2
Ca	K series	58,1	1,02	37,87	CaO	81,29	Wollastonite
Total:		100		100		100	