Microstructural characterization of calcined lithomarge geopolymer mortar

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at the

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Outline

- Introduction: Iron rich clay – Binder for the future
- Mortar mixes and testing
- Results: Morphology

  Mineralogical phases

  Functional group classification
- Conclusion

Why alternative cementitious binder?

- 4.2 Billion metric tonnes of OPC was produced globally in 2016 and steady growth is projected;
- 5% of the current global CO_2 production is attributed to Portland cement production;
- Geoplymers are among the alternatives with the potential of improving sustainability of the construction industry.

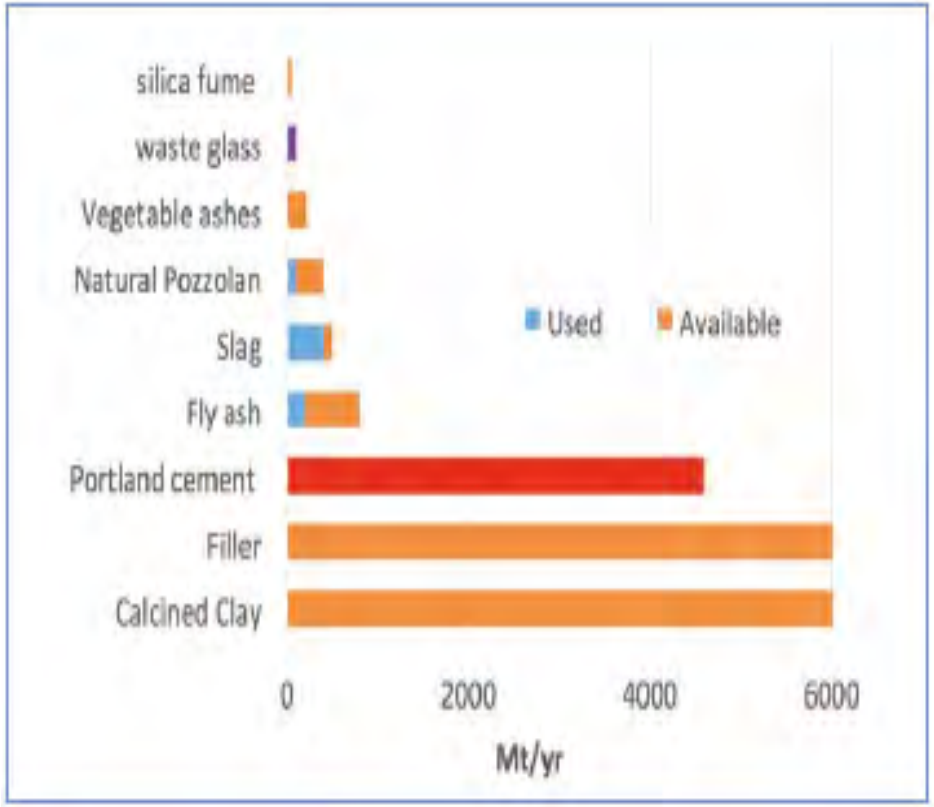
Table 1: Energy and CO2 savings of geopolymer cement; Source: (Davidovits, 2013)

Energy needs (MJ/tonne)	Calcination	Crushing	Silicate Sol.	Total	Reduction
Portland Cement	4270	430	0	4700	0
GP-cement, slag by-product	1200	390	375	1965	59%
GP-cement, slag manufacture	1950	390	375	2715	43%
CO_2 emissions (tonne)					
Portland Cement	1.000	0.020		1.020	0
GP-cement, slag by-product	0.140	0,018	0.050	0.208	80%
GP-cement, slag manufacture	0.240	0.018	0.050	0.308	70%

Typical cement plant

Clay based Geopolymers

- Previous studies focus largely on GGBS, PFA and Metakaolin as precursors that show promising results;
- Long term supply of these precursors may be hard to achieve in the future in many part of the world;
- Lithomarge clays are extensively available across the globe and can be viable alternative;



Utilization of SCMs; Source: Scrivener 2016

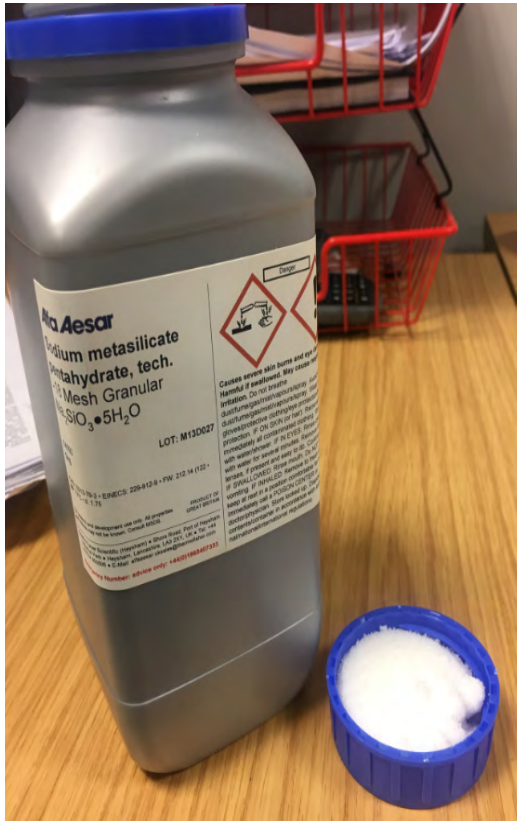
Materials

- Precursor: The flash Calcined lithomarge clay
- Fine aggregate: finer than 4.5 mm

Oxide (% by weight)	SiO_2	Fe_2O_3	Al_2O_3	TiO_2	MgO	CaO	LOI
banahmeta	35.18	25.4	29.6	2.9	1.3	0.9	< 2%

The alkali metal source

- Na_2SiO_3 solution; MR = 2.05
- $Na_2SiO_3.5H_2O$
- NaOH



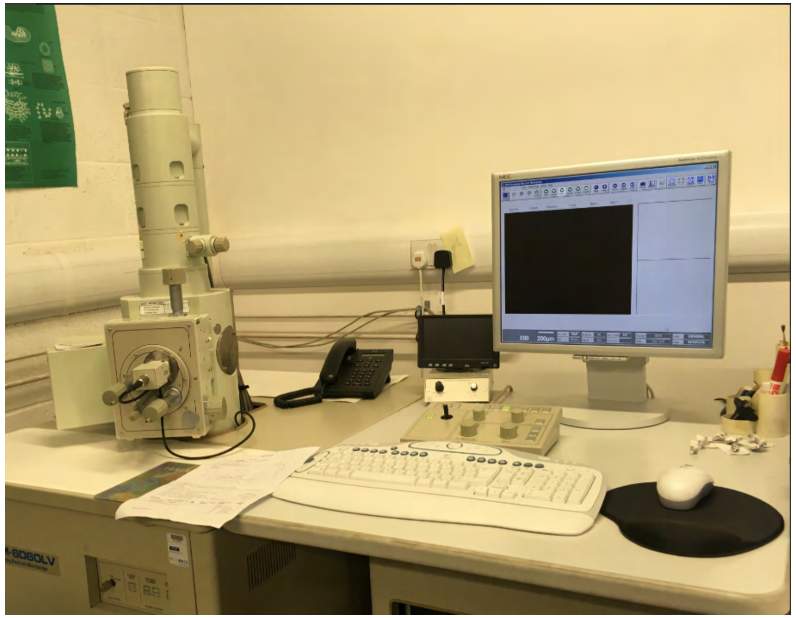
Mortar Mixes

Mass ratio of mixes

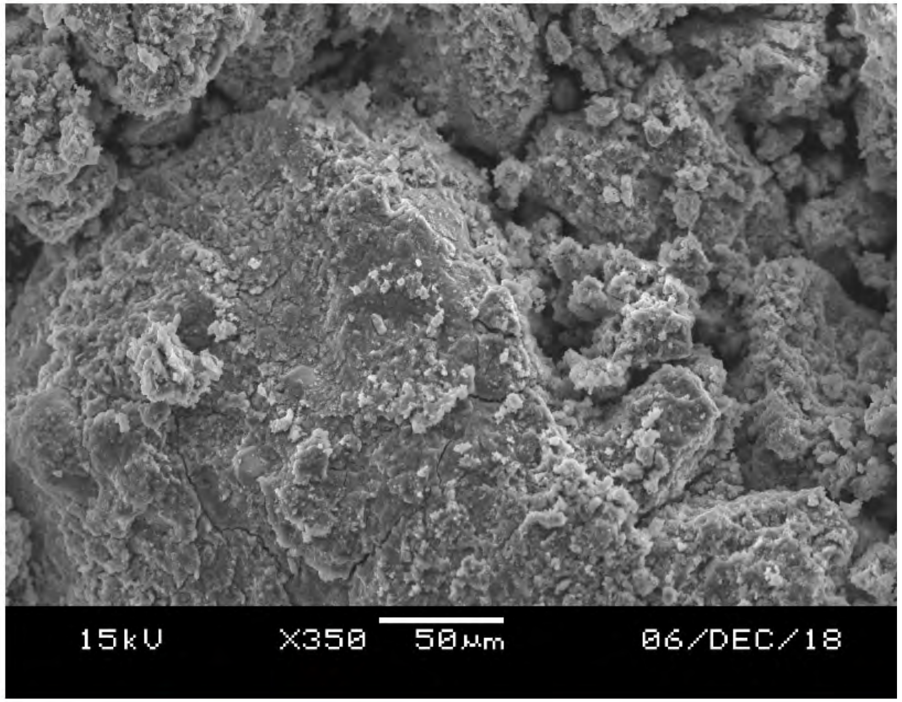
Group 1	Sand	CC – 32SH			CC – 44.1SSP		
L:B		Calcined clay	32% NaOH solution	Free water	Calcined clay	44.1% $Na_2SiO_3.5H_2O$	Free water
0.8	723	723	579	0	723	579	0
Group 2 (CC - 54.5SS)	Sand		Calcined clay		54.5% Na_2SiO_3		Free water
1	1221		297		297		84
1.5	1221		238		356		84

Mix - CC : SS ratio	Na_2O/Al_2O_3	SiO_2/Al_2O_3	Na_2O/SiO_2	H_2O/Na_2O
1	1	4.12	0.24	14.10
1.5	1.49	5.16	0.29	13.24

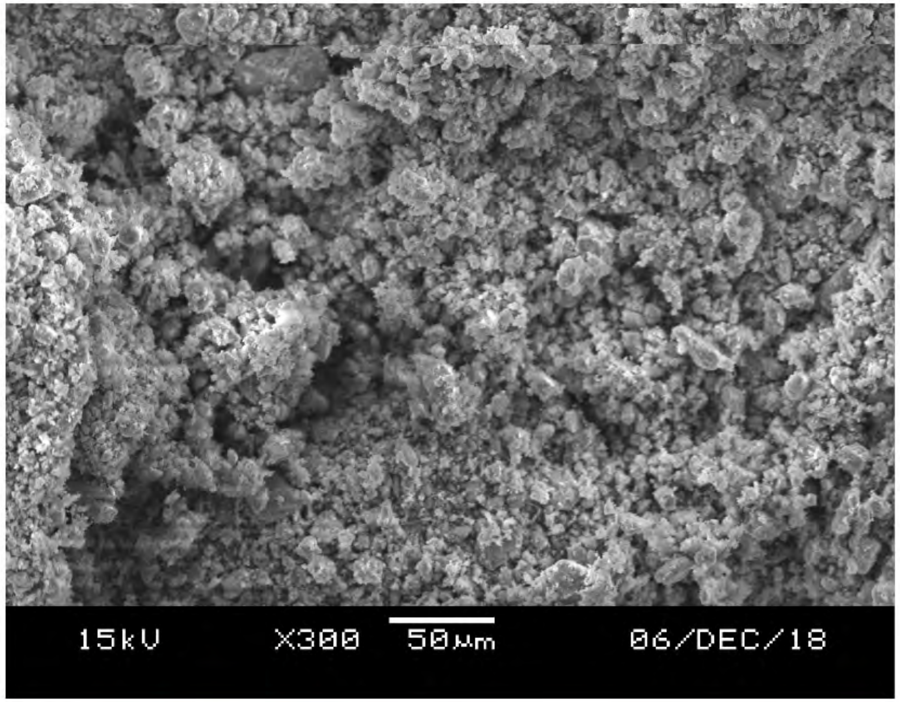
Tests



The Morphology

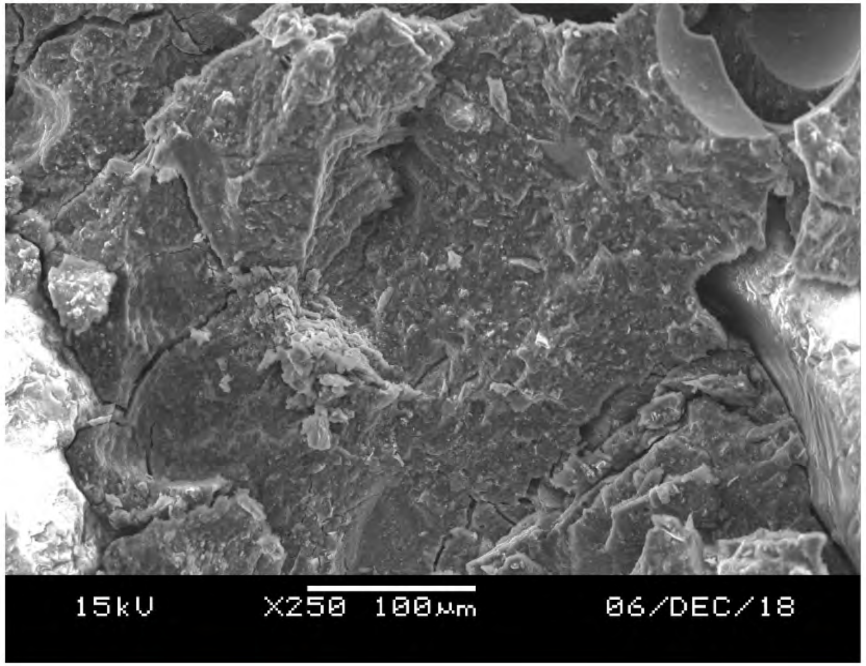


SEM micrograph for CC – 32SH mortar



SEM micrograph for CC – 44.1SSP mortar

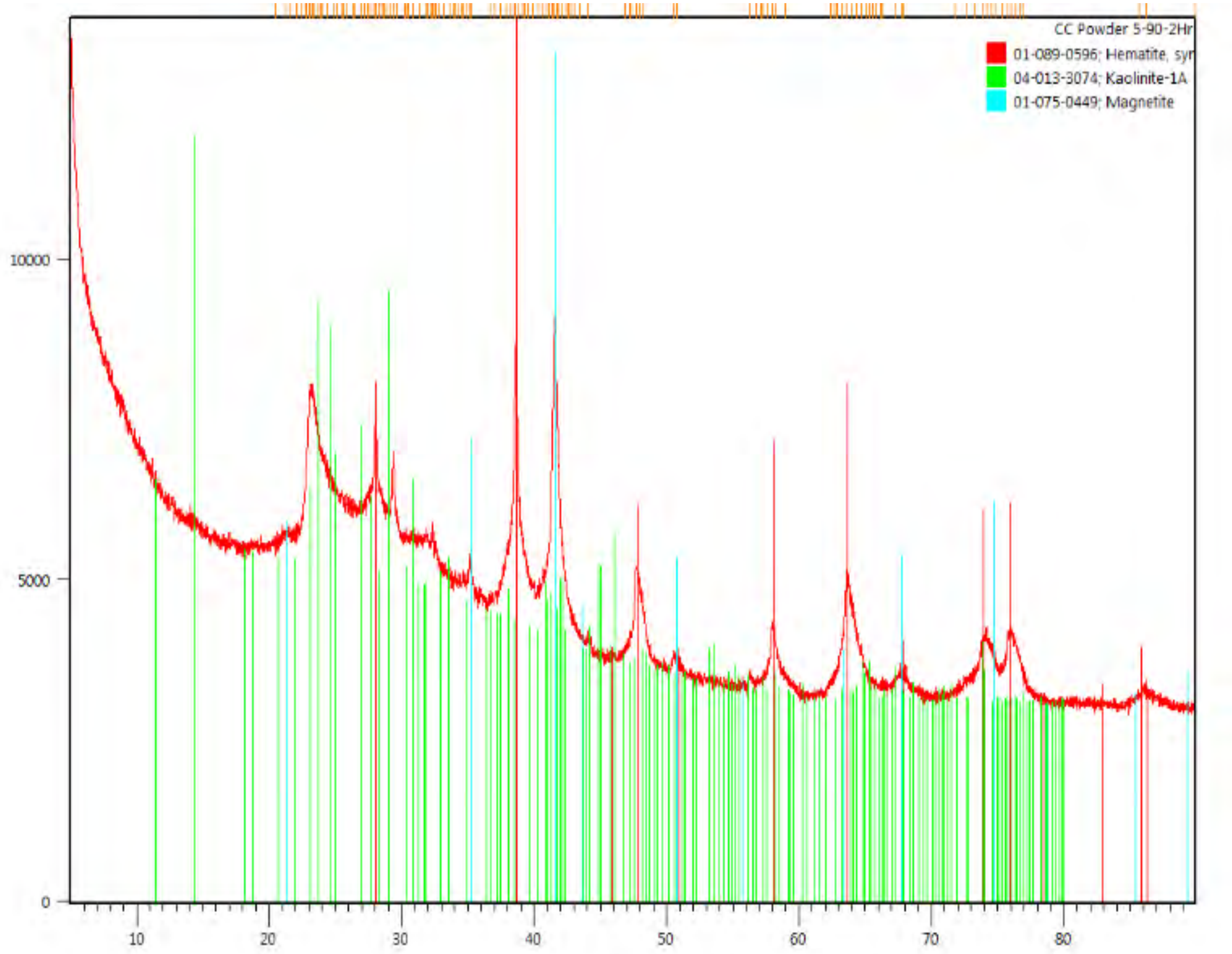
Morphology of the Peak strength Mix

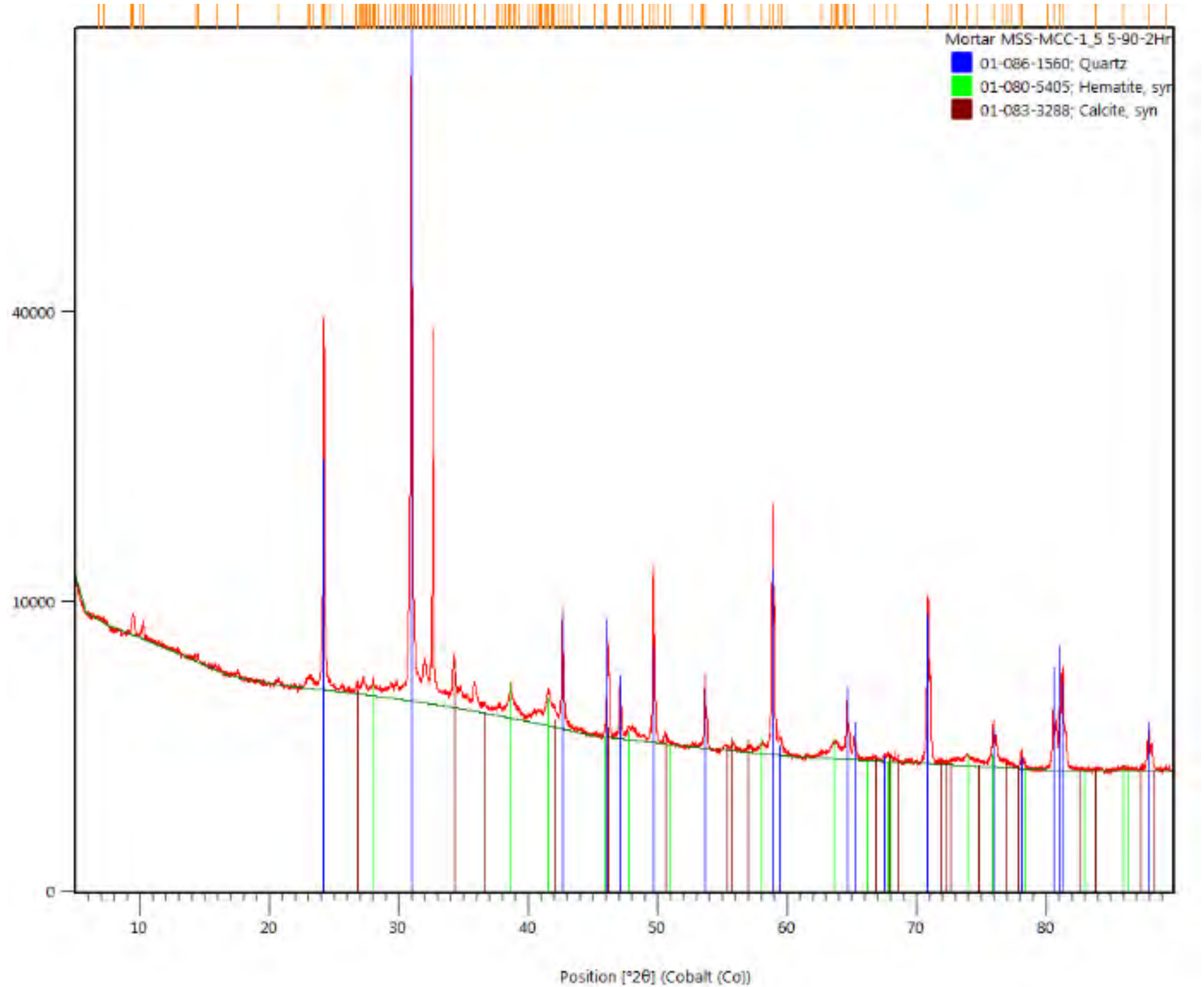


SEM image for CC - 54.5SS mortar

- The microstructure of the peak strength mix reveals a compact rock mass bulk geopolymer structure which resulted in the high strength mortar achieved by the mix.
- The SEM image also showed some dispersed cracks which is thought to be caused by the loading of the sample during compression test

XRD Results - CC Powder



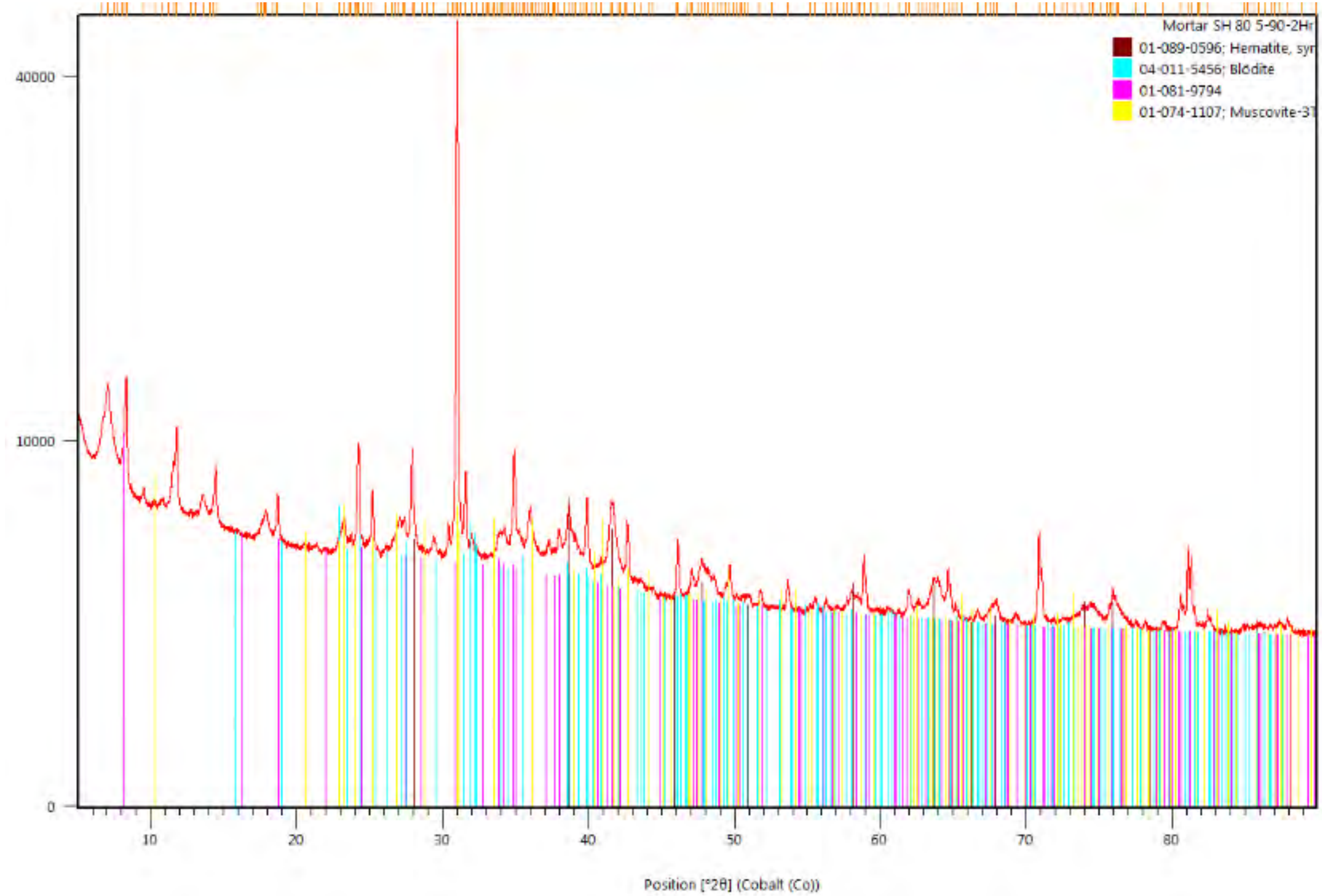

Mortar MSS-MCC-1_5 5-90-2Hr
01-086-1560; Quartz
01-080-5405; Hematite, syn
01-083-3288; Calcite, syn
40000
10000
0
10
20
30
40
50
60
70
80
Position [°2θ] (Cobalt (Co))

Pattern List

Quantification | Anchor Scan Data | Pattern List | Peak List | Refinement Control | Scan List | Structure Plot

Accepted Ref. Pattern: 04-012-1905

No.	Visible	Ref. Code	Compound Name	Chemical Formula	Score	Scale Fac...	Display Color	Quality	Crystal System	SemiQuant [%]
1	☑	ICDD 01-086-1560	Silicon Oxide	Si O2	46	1.048	Blue	S;ALT	Hexagonal	34
2	☑	ICDD 01-080-5405	Iron Oxide	Fe2 O3	43	0.020	Lime	S;ALT	Rhombohedral	1
3	☑	ICDD 01-083-3288	Calcium Carbonate	Ca (C O3)	29	0.027	Maroon	S;ALT	Rhombohedral	1
4	☑	ICDD 00-009-0466	Sodium Aluminum Silicate	Na Al Si3 O8	31	0.026	Aqua	S	Anorthic	1
5	☑	ICDD 01-080-2107	Potassium Aluminum Silicate	K (Al Si3 O8)	25	0.020	Fuchsia	S;ALT	Monoclinic	3
6	☑	ICDD 04-012-1905	Potassium Sodium Iron Aluminum Silicon Oxide Hydroxide	K0.8 Na0.2 Fe0.05 Al2.95 Si3.1 O10 (O H)2	24	0.023	$000080FF	S	Monoclinic	6
7	☑	ICDD 04-011-6768	Potassium Sodium Aluminum Silicate	K0.22 Na0.78 Al Si3 O8	30	0.026	Yellow	I	Anorthic	4
8	☑	ICDD 04-011-7479	Potassium Aluminum Iron Silicon Oxide	K0.98 Fe0.51 Al0.48 Si3.03 O8	24	0.076	Red	S	Monoclinic	9
9	☑	ICDD 01-070-3754	Potassium Aluminum Silicate Hydroxide	K (Al4 Si2 O9 (O H)3)	20	0.200	Navy	I	Monoclinic	43



Mortar SH 80 5-90-2Hr
01-089-0596; Hematite, syn
04-011-5456; Blödite
01-081-9794
01-074-1107; Muscovite-3T
40000
10000
0
10
20
30
40
50
60
70
80
Position [°2θ] (Cobalt (Co))

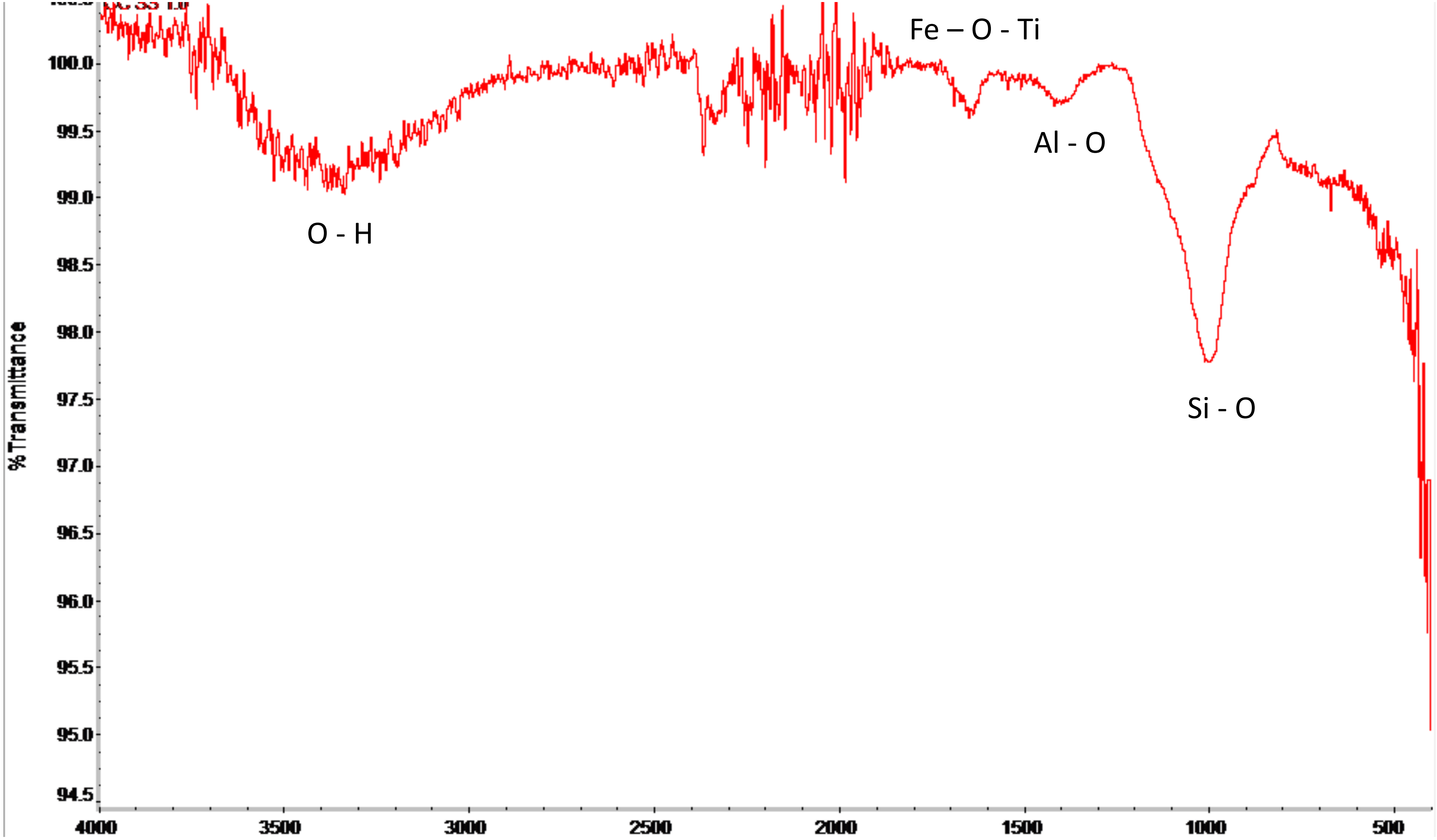
Pattern List

Quantification | Anchor Scan Data | Pattern List X | Peak List | Refinement Control | Scan List | Structure Plot

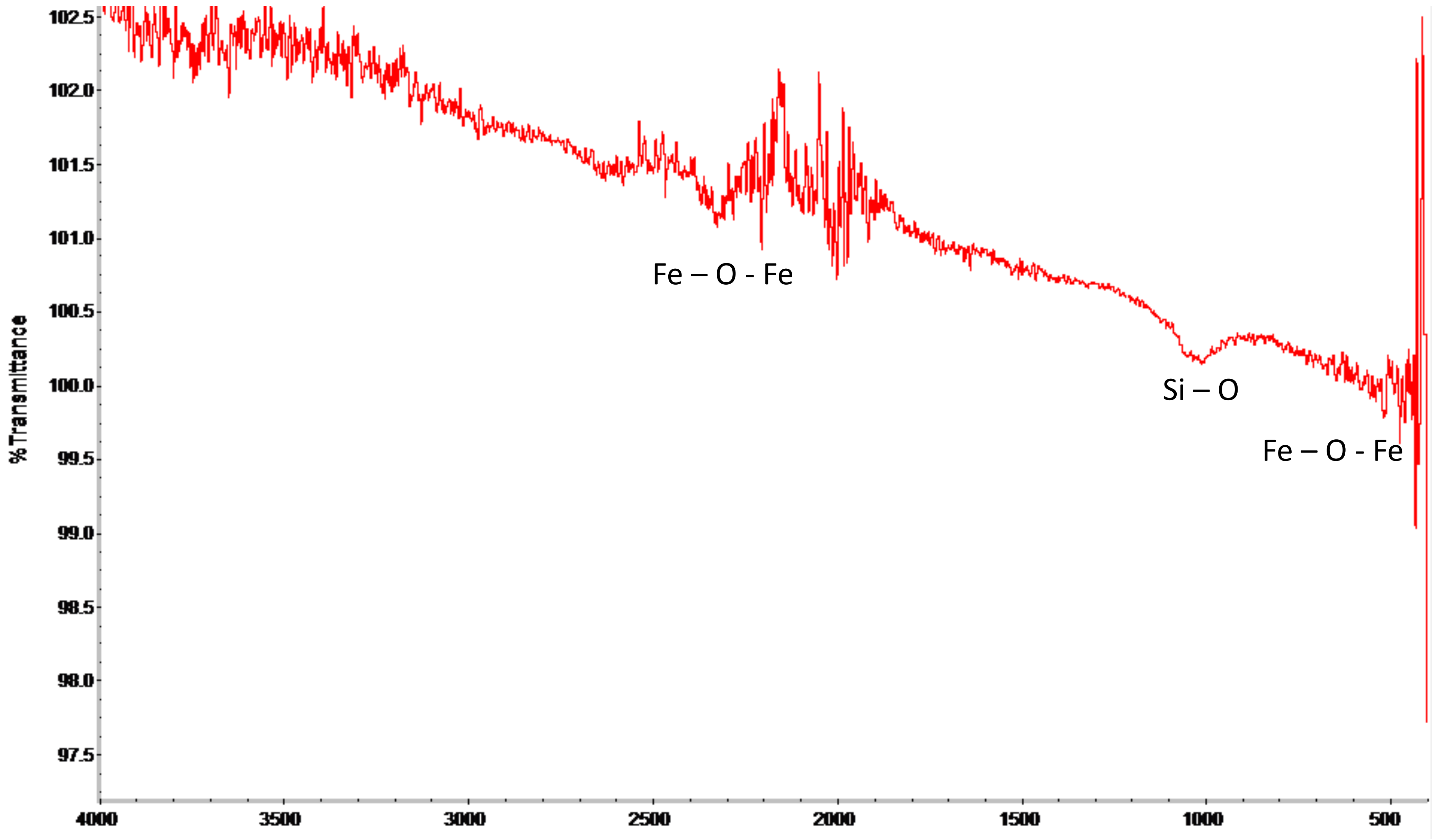
Accepted Ref. Pattern: 01-074-1107

No.	Visible	Ref. Code	Compound Name	Chemical Formula	Score	Scale F...	Display Color	Quality	Crystal System	SemiQuant [%]
1		ICDD 04-016-9920	Sodium Aluminum Silicate Hydrate	Na24 Al24 Si24 O96 (H2 O)64.8	42	0.110	Blue	B;ALT	Cubic	10
2		ICDD 01-086-1560	Silicon Oxide	Si O2	45	0.703	Lime	S;ALT	Hexagonal	30
3		ICDD 01-074-2534	Sodium Aluminum Silicate Hydrate	Na96 (Al96 Si96 O384) (H2 O)384.3	43	0.247	Purple	B	Cubic	13
4		ICDD 01-089-0596	Iron Oxide	Fe2 O3	39	0.072	Maroon	S;ALT	Rhombohedral	3
5		ICDD 04-011-5456	Sodium Magnesium Sulfate Hydrate	Na2 Mg (S O4)2 (H2 O)4	18	0.040	Aqua	I;ALT	Monoclinic	7
6		ICDD 01-081-9794	Calcium Silicate Hydroxide Hydrate	Ca3 (Si2 O6 (O H)2) (H2 O)	24	0.058	Fuchsia	I	Orthorhombic	1
7		ICDD 01-074-1107	Potassium Aluminum Iron Silicate Hydroxide	K (Al1.91 Fe0.09) (Si3 Al) O10 (O H)2	29	0.056	Yellow	S	Hexagonal	12
8	✓	ICDD 04-017-3641	Potassium Sodium Magnesium Aluminum Iron Silicon Titanium Oxide Hydroxide	K0.90 Na0.05 Mg0.32 Ti0.03 Fe0.18 Al2.11 Si3.40 O10 (O H)2	30	0.019	Red	I;ALT	Hexagonal	4
9	✓	ICDD 01-073-9859	Potassium Sodium Aluminum Iron Magnesium Silicon Oxide Hydroxide Fluoride	K0.92 Na0.08 Al1.88 Fe0.12 Mg0.04 (Al1.08 Si2.92 O10) (O H)1.89 F0.11	25	0.055	Navy	S	Monoclinic	19
10	✓	ICDD 04-011-2101	Silicon Oxide	Si O2	16	0.048	S004080...	S	Monoclinic	1

FTIR Results for the Peak strength

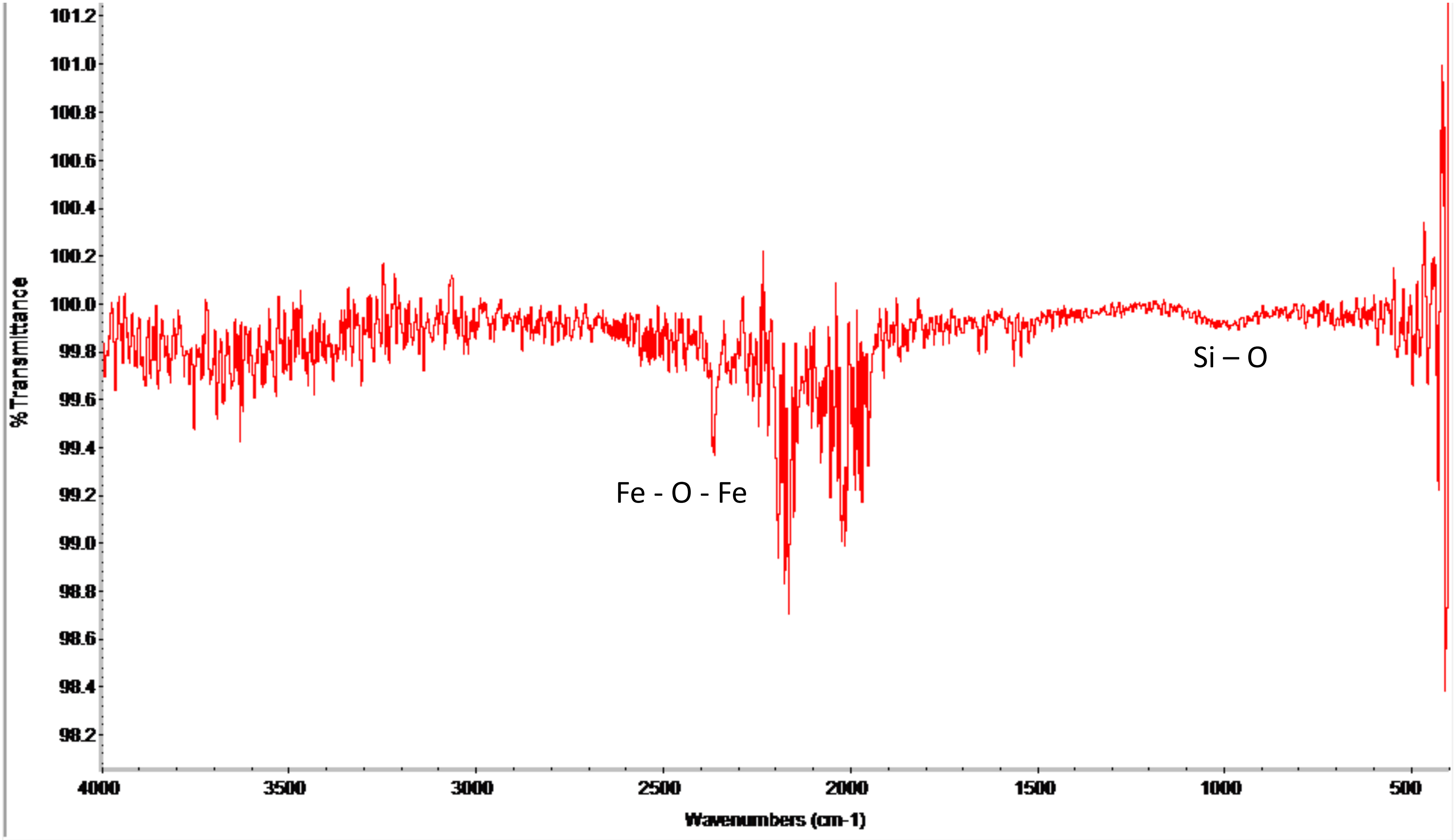


CC – SSP80



102.5
102.0
101.5
101.0
100.5
100.0
99.5
99.0
98.5
98.0
97.5
%Transmittance
4000
3500
3000
2500
2000
1500
1000
500
Fe – O - Fe
Si – O
Fe – O - Fe

FTIR – SH80


101.2
101.0
100.8
100.6
100.4
100.2
100.0
99.8
99.6
99.4
99.2
99.0
98.8
98.6
98.4
98.2
%Transmittance
Fe - O - Fe
Si – O
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)

Conclusion

- Geopolymer synthesis of the calcined clay mortar showed sensitivity to the type of chemical activator used in the system;

- utilizing hydrous $Na_2SiO_3.5H_2O$ as activator precipitate low strength non-geopolymer matrix that has unreactive residual calcined clay which disrupt complete development of geopolymer network;

- The peak strength mix shows a rock mass microstructure

- The amount of hematite in the CC powder decrease due to alkalination in the mortar

- The 8 M NaOH solution precipitated zeolites as one of its reaction product.


Thanks
For Your Time