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TECHNICAL MEMORANDUM

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SUBJECT:	Mount Peake Project Darwin Process Plant – Waste Characterisation

1. INTRODUCTION

This document provides the results of a test programme designed to characterise waste products supplied to EGi by TNG Ltd. These waste materials were created to simulate waste products that will be generated by TNG's proposed Darwin process plant. This process plant will provide a secondary processing facility for their Mount Peake magnetite operations.

The proposed Darwin process plant will receive magnetite concentrate containing vanadium and titanium minerals from the Mount Peake mine for further processing. Processing will be undertaken using the TIVAN process which will produce high purity titanium dioxide, vanadium pentoxide and ferric oxide products. The plant will also produce a number of waste streams that will be collected in closed containers and transported by rail to the Mount Peake mine site for co-disposal with rejects from the Mount Peake process plant in an integrated waste landform (IWL). The scope of this project covered characterisation of the waste materials that will be generated at the Darwin process plant and a preliminary assessment of key watershed environmental risk issues in handling and transport of these materials by rail to the IWL at Mount Peake.

This report does not represent a detailed ecological risk assessment of potential impacts to the rail corridor (i.e., source-pathway-toxicity at reception), nor does it cover investigations into potential environmental risks associated with disposal of the wastes within the IWL.

The Mount Peake Project Darwin Processing plant will produce a number of solid products that may not have commercial value. The products are summarized in the table below:

Table 1. Summary of waste products which will be generated from the Darwin processing plant

Waste Product	Area	Waste Description
1	200	Coke Ash
2	500	Sulphate digest leach residue (SDLR)
3	500	Acid waste neutralization gypsum
4	500	Acid waste neutralization metal hydroxides
5	770	Acid waste neutralization metal hydroxides
6	WRP	WRP salt residue
7	WRP	WRP clarifier solids
8	710	Scrubber gypsum

EGi was supplied with waste products 1, 2, 4, 5 and 6. Waste product 7 was not generated in these trials as the process could not be reliably conducted on a bench scale. The composition of this product is expected to be similar to waste products 3, 4 and 5. Waste product 8 was not generated in these trials as it is a standard industrial process and there is no reason to believe sample generated in this process will be any different. Waste product 3 was produced in the trials; however, it is a gypsum product which is expected to have minor impurities. The minor impurities will be similar in composition to waste product 4. The decision to exclude these samples from the detailed characterization tests was made in consultation with TNG. EGi were however provided with detailed elemental analysis results for product 3.

2. TEST PROGRAMME

The characterisation of the various waste materials can be split into two types, namely physical characterisation and dissolution properties. Physical characterisation provides compositional information for each of the waste products so that the nature of the materials is well defined. Examination of the dissolution properties of these wastes was conducted to understand what these materials may release to the watershed as a result of accidental spillage in transport and handling.

Physical characterisation testing included:

- Elemental analysis including quantitative assay of trace heavy metals/metalloids using multi-acid digestion and ICP-OES and ICP-MS analysis, analysis of chloride and sulphate content by KOH fusion followed by dissolution in water and analysis by ion chromatography and carbon speciation analysis, including total C by high temperature combustion, organic carbon by the same method after pre-treatment with acid to remove carbonates and inorganic carbon calculated as the difference between total and organic carbon. XRF analysis was also undertaken, principally to provide Si content, which is not quantitatively measured using acid digestion methods, and to provide confirmation of the acid digest results for major elements.
- Mineralogical analysis by quantitative XRD. This provides a (semi-) quantitative list of crystalline minerals present in the wastes. It was anticipated that due to the nature of the processing methods used to generate the waste products, a significant amount of some of these materials may be non- or poorly crystalline and therefore X-ray invisible. Therefore, the amorphous content of the wastes was quantified to provide more accurate mineralogical compositions. This was done by measuring a standard separately using identical data collection conditions as for the sample, and then using elemental compositions for the waste materials to calculate mass absorption coefficients and then work out the amount of amorphous content.
- To obtain information on the composition of amorphous or poorly crystalline phases in the wastes scanning electron microscopy (SEM) was used. Energy dispersive spectroscopy (EDS) was used in conjunction with SEM to determine the elemental composition of amorphous phases. Both point analysis and elemental mapping over a wider area were performed. SEM analysis also provided information on particle size to correlate with particle size measurements.
- Particle size distribution analysis (PSD) was undertaken for each of the waste materials. PSD analysis was conducted using laser diffraction methods. Because some wastes have significant water solubility, analyses were conducted using non-aqueous liquids (cyclohexane, acetone) for dispersion, in addition to water. Results included details on the amount of material that is of respirable size (<2.5 mm), which was used in conjunction with phase identification to estimate risks arising from the presence of respirable silica.

The dissolution behaviour of the waste materials was investigated using EPA Method 1316 batch extraction tests. This method is designed to provide the liquid-solid partitioning (LSP) of inorganic constituents (e.g., metals, anions) at the natural pH of the solid material as a function of liquid-to-solid ratio (LSR) under conditions that approach liquid-solid chemical equilibrium. Typically, the method uses LSR of between 0.5 and 10, with the lower end representing likely concentrations in pore water, while the higher end provides concentrations indicative of drainage. However, for some samples, especially the metal hydroxide samples, the low end of LSR could not be used, as sufficient leachate could not be recovered due to the high water-retention of these materials. In these cases, the range of LSR were extended to 25. Leachates were analysed for a range of parameters including pH, EC, acidity, alkalinity, major anions (Cl, SO4), and a broad range of metals and metalloids. Leachate water quality parameters were compared with ANZECC guidelines where applicable, after applying appropriate dilution factors to account for solid: liquid ratios used in the batch extraction experiments. It should be noted that ANZECC guidelines were used as a screening tool to identify parameters of potential concern and exceedances do not indicate significant risk to the environment but rather a requirement for further investigation and or qualification of risk.

It should also be noted that total trace heavy metal concentrations were not compared against any ecological-based guidelines as natural background concentrations along the rail corridor are currently unknown (both in terms of land areas and waterway beds).

Except for the coal ash, which was received dry, all other samples contained water varying from entrained moisture to saturated with the presence of supernatant liquor. As all test work, except PSD analysis and batch leach tests, required dry samples, a portion of each sample was dried to a constant mass at 60^{o}C. Waste product 4 (Metal Hydroxide Precipitation #1 Residue) was received at about 77% water content due to the low filterability of this material. It is anticipated that press filtration in the plant will achieve a moisture content of around 50 to 60%, so this sample was dried to 54 wt.% water content at 40^{o}C prior to conducting any test work. These temperatures were chosen to minimise conversion of amorphous metal hydroxides to crystalline phases, a process which can be accelerated by higher temperatures. Waste product 5 (Artificial Solution #2 Neutralisation #1 Residue) was received with excess water present. Decant water was first removed and the samples dried as above. Waste product 6 (Bulk Crystallisation Slurry) was expected to contain essentially only crystalline salts and so this sample was dried at 85^{o}C to a constant weight.

Table 2 below provides details of all the tests conducted on each sample.

Table 2. Summary of characterisation work conducted on waste products

EGi Sample Code	Solid Waste Product	Area	Description	Dry Weight	Bulk Assay[1]	XRF	XRD	SEM-EDS	PSD	Batch Leach Extraction
20276	1	200	Coal Ash	✗	✓	✓	✓	✓	✓	✓
20277	2	500	Sulphate digest leach residues	✓	✓	✓	✓	✗[2]	✓	✓
	3	500	SAS Neutralisation #1 Residue (Gypsum)	✗	✓[3]	✗	✗	✗	✗	✗
20278	4	500	Metal Hydroxide Precipitation #1 Residue	✓	✓	✓	✓	✓	✓	✓
20529	5	770	Artificial Solution #2 Neutralisation #1 Residue	✓	✓	✓	✓	✓	✓	✓
20530	6	WRP	Bulk Crystallisation Slurry	✓	✓	✓	✓	✓	✓	✓

1. Includes four-acid digest with ICP-MS/OES finish, KOH fusion with IC finish (Cl and SO4) and carbon forms (Total C, Organic C, Inorganic C).
2. SEM-EDS was not performed due to the acidity of the sample, which could result in damage to the instrument
3. This sample was not included in the test programme and results for bulk assay were provided by TNG

3. RESULTS

3.1. Characterisation

3.1.1. Coal Ash – Waste Product #1 (EGi Code 20276)

3.1.1.1. XRF Analysis

Results from XRF analysis of the coal ash sample (Waste Product 1) are provided in Table 3. The full set of results are provided in Appendix A.

Table 3. XRF results: Coal ash

ELEMENT	UNIT	CONCENTRATION
Major Elements		
Al	% as Al_2O_3	5.68
Ca	% as CaO	1.28
Cl	%	<0.05
Fe	% as Fe_2O_3	11.6
K	% as K_2O	0.122
Mg	% as MgO	0.75
Na	% as Na_2O	0.06
S	% as SO_3	0.02
Si	% as SiO_2	8.55
Ti	% as TiO_2	2.37
Heavy Metals		
Cr	ppm	904
Mn	ppm	676
Ni	ppm	1661
V	ppm	1598
Zn	ppm	911
LOI	%	68.9
Total	%	100.4

3.1.1.2. Bulk Assay

Results from bulk assay of the coal ash sample (Waste Product 1) are provided in Table 4. The full set of results are provided in Appendix B.

Table 4. Bulk assay results: Coal ash

ELEMENT	UNIT	CONCENTRATION
Major Elements		
Al	%	2.6
Ca	%	0.9
Cl	%	0.01
Fe	%	7.8
K	%	0.10
Mg	%	0.44
Na	%	0.09
S	%	0.3
Ti	%	1.5
Total C	%	68.0
Organic C	%	66.2
Inorganic C	%	1.8
Heavy Metals		
Cr	ppm	817
Mn	ppm	639
Ni	ppm	1880
V	ppm	1400
Zn	ppm	787
Total[1]	%	96.2

1. Calculated using SiO_2 content measured by XRF

3.1.1.3. QXRD Analysis

Results for QXRD analysis of the coal ash sample (Waste Product 1) are provided in Table 5. Diffraction results are provided in Appendix C.

Table 5. QXRD results: Coal ash

PHASE	FORMULA	AMOUNT (%)[1]
Anatase	TiO_2	0.1
Calcite	$CaCO_3$	0.5
Halite	NaCl	0.5
Hercynite	$FeAl_2O_4$	3
Ilmenite	$FeTiO_3$	3
Iron	Fe	3

Magnetite	Fe_3O_4	2
Mullite	$Al_{4+2x}Si_{2-2x}O_{10-x}$	2
Nickel	Ni	0.7
Quartz	SiO_2	1
Rutile	TiO_2	0.4
Taenite	γ-FeNi	0.1
Amorphous	-	86
SUM		100

1. Amounts shown in red (<1%) are unreliable and serve as a guide only as the high amorphous content makes quantitation difficult.

3.1.1.4. SEM-EDS Analysis

Typical images obtained during SEM analysis of the coal ash sample (Waste Product 1) are provided in Figures 1 - 3. Full SEM-EDS results are provided in Appendix D. The major results from SEM-EDS analysis are:

The major phase is carbon particles <200 μm in size (Figure 1)

Amorphous calcium silicate particles <200 μm in size are also relatively abundant (Figure 1)

The sample contains porous titanium oxide particles with metallic iron droplets deposited on the surface (Figure 3)

Amorphous/poorly crystalline Fe,Ti oxide (Ilmenite?) particles up to 1 mm in size are also relatively abundant (Figure 2)

Amorphous/poorly crystalline Al oxide particles can also be seen in this sample (Figure 2)

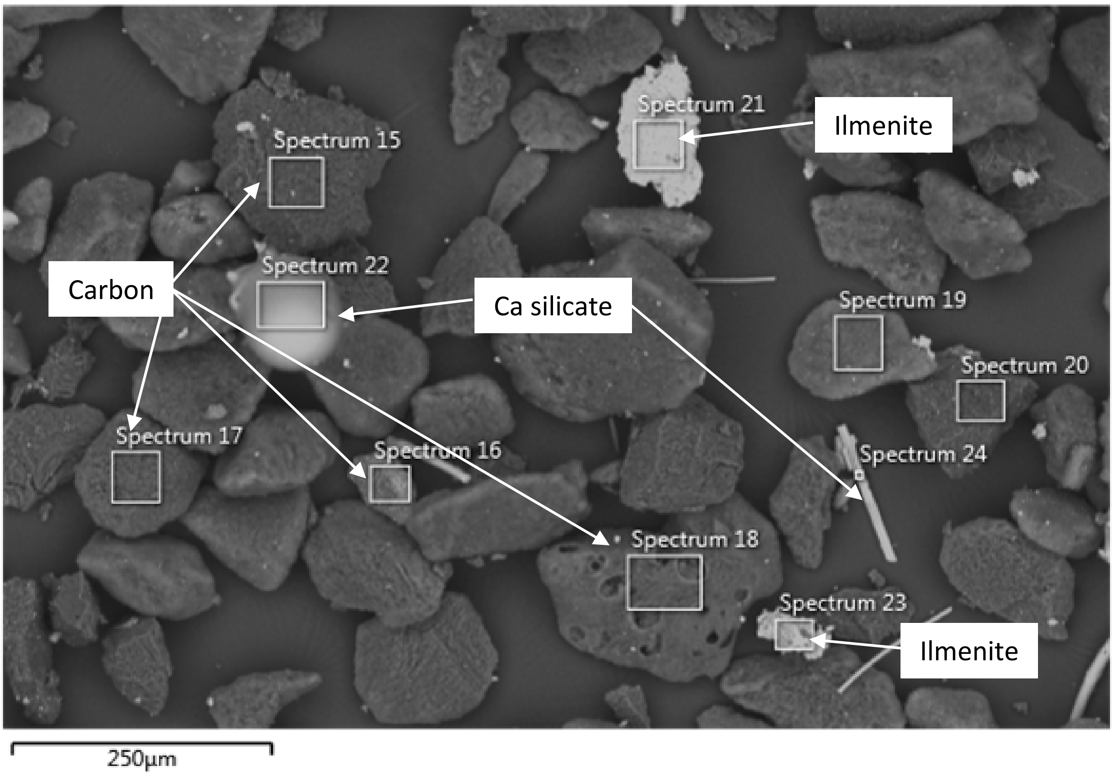

Figure 1. Back-scattered electron image of particles in the coal ash sample. Phase contrast is shown with heavier elements showing up as a lighter shade.

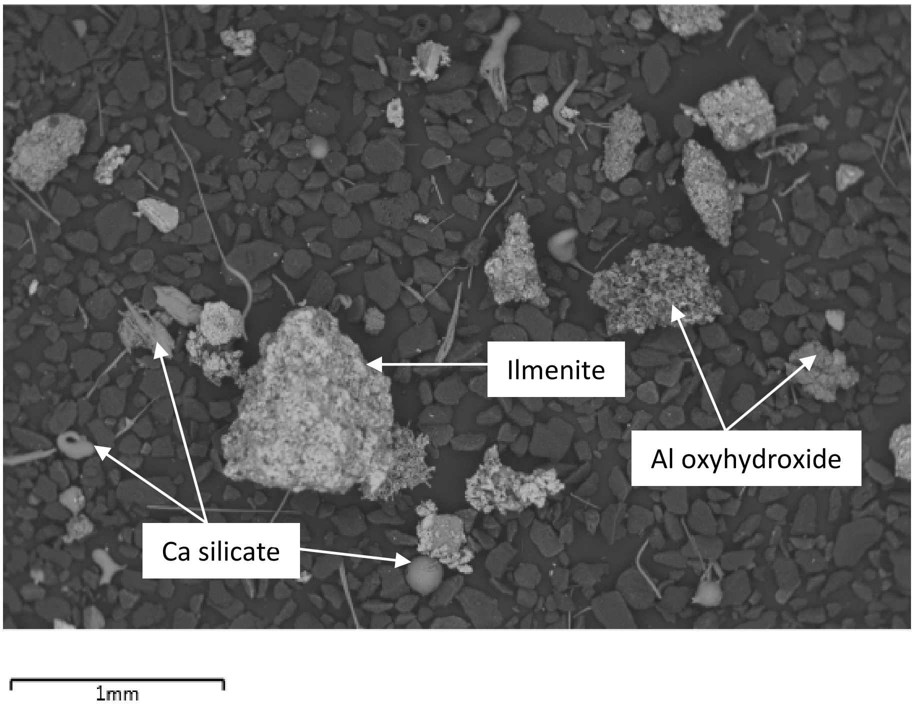

Figure 2. Back-scattered electron image of particles in the coal ash sample. Phase contrast is shown with heavier elements showing up as a lighter shade.

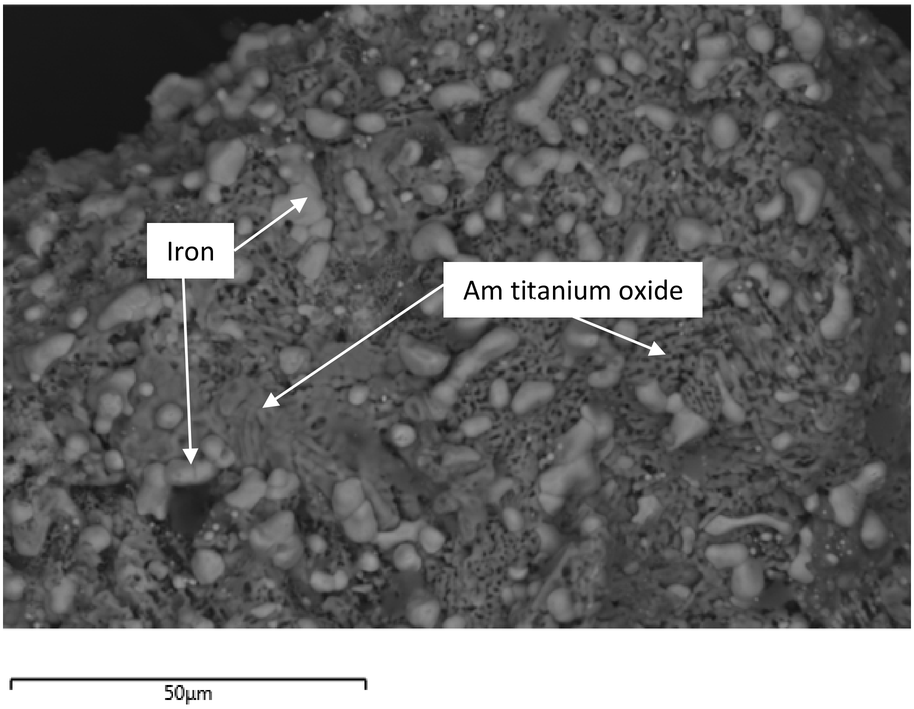

Figure 3. Back-scattered electron image of the coal ash sample showing a porous titanium oxide particle with metallic iron droplets deposited on the surface.

3.1.1.5. PSD Analysis

Table 6 provides summary statistics from PSD analysis of the coal ash sample (Waste Product 1) and Figure 4 shows the PSD for this material. Full PSD results are provided in Appendix E.

Comparison of PSD results with SEM results shows that the coal ash sample is dominated by carbon particles which appear to be in the size range of 20 to 200 µm. In addition to carbon, there are calcium silicate particles which also appear to be generally less than 200 µm in size. A few larger particle (0.5 - 1 mm) of Fe, Ti oxide and Al oxide/hydroxide are also present, but these appear not to be properly dispersed and do not appear in the PSD analysis. This material contains very little respirable quartz.

Table 6. PSD results: Coal ash. D(0.1), d(0.5) and d(0.9) represent the particle sizes under which 10, 50 and 90% of particles by volume respectively are found.

d(0.1) µm	**d(0.5)** µm	**d(0.9)** µm	**AMOUNT < 2.5 µm** Vol %
22.9	64.6	123.8	1.4

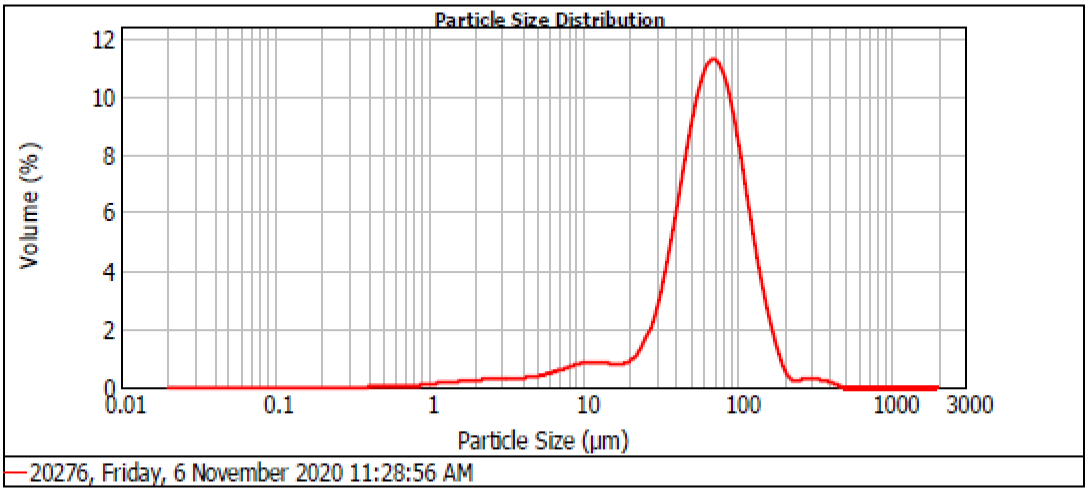

Figure 4. PSD of the coal ash sample dispersed in water.

3.1.1.6. Summary of Characterisation Results for the Coal Ash Sample

The coal ash sample is dominated by carbon particles 20 – 200 µm in size (66 wt.% organic C), possibly containing adsorbed sulphate. The remainder of the sample is comprised of relatively large (0.2 – 1 mm) metal oxides, including Fe, Al, and Ti oxides, together with calcium silicates and metallic iron. Most phases are amorphous or poorly crystalline, with very little material providing useful diffractions. Trace heavy metals of significance include, in decreasing concentration, Ni, V, Zn, Cr and Mn.

3.1.2. Sulphate digest leach residues – Waste Product #2 (EGi Code 20277)

3.1.2.1. XRF Analysis

Results from XRF analysis of the sulphate digest leach residues sample (Waste Product 2) are provided in Table 7. The full set of results are provided in Appendix A.

Table 7. XRF results: Sulphate digest leach residues

ELEMENT	UNIT	CONCENTRATION
Major Elements		
Al	% as Al_2O_3	4.63
Ca	% as CaO	4.68
Cl	%	<0.05
Fe	% as Fe_2O_3	2.46
K	% as K_2O	1.12
Mg	% as MgO	1.32
Na	% as Na_2O	0.40
S	% as SO_3	1.30
Si	% as SiO_2	40.90
Ti	% as TiO_2	23.70
Heavy Metals		
Cr	ppm	1890
Mn	ppm	741
Ni	ppm	63
V	ppm	1318
Zn	ppm	250
LOI	%	18.50
Total	%	99.8

3.1.2.2. Bulk Assay

Results from bulk assay of the sulphate digest leach residues sample (Waste Product 2) are provided in Table 8. The full set of results are provided in Appendix B.

Table 8. Bulk assay results: Sulphate digest leach residues

ELEMENT	UNIT	CONCENTRATION
Major Elements		
Al	%	2.26
Ca	%	3.29
Cl	%	0.30

Fe	%	1.82
K	%	0.91
Mg	%	0.73
Na	%	0.22
S	%	3.43
Ti	%	>10
Total C	%	1.6
Organic C	%	1.6
Inorganic C	%	<0.02
Heavy Metals		
Cr	ppm	1380
Mn	ppm	630
Ni	ppm	23.4
V	ppm	491
Zn	ppm	163
Total[1]	%	56.29

1. Calculated using SiO_2 and TiO_2 content measured by XRF

3.1.2.3. QXRD Analysis

Results for QXRD analysis of the sulphate digest leach residues sample (Waste Product 2) are provided in Table 9. Diffraction results are provided in Appendix C.

Table 9. QXRD results: Sulphate digest leach residues

PHASE	FORMULA	AMOUNT (%)[1]
Anhydrite	$CaSO_4$	3
Bassanite	$CaSO_4.0.5H_2O$	1
Diopside	$CaMgSi_2O_6$	7
Gypsum	$CaSO_4.2H_2O$	3
Quartz	SiO_2	0.3
Rutile	TiO_2	20
Amorphous	-	66
SUM		100

1. Amounts shown in red (<1%) are unreliable and serve as a guide only as the high amorphous content makes quantitation difficult.

3.1.2.4. SEM-EDS Analysis

SEM analysis was not conducted for this sample due to the potential for damage to the instrument as this material contained considerable acidity.

3.1.2.5. PSD Analysis

Table 10 provides summary statistics from PSD analysis of the sulphate digest leach residues sample (Waste Product 2) and Figure 5 shows the PSD for this material. Full PSD results are provided in Appendix E.

Results suggest that the vast majority of particles are under 100 µm in size. However, there is no SEM data to support this and the identification of rutile by XRD could indicate the presence of larger heavy particles which are not readily dispersed and do not appear in the PSD analysis, although this cannot be confirmed. This material contains very little respirable quartz.

Table 10. PSD results: Sulphate digest leach residues sample. D(0.1), d(0.5) and d(0.9) represent the particle sizes under which 10, 50 and 90% of particles by volume respectively are found.

d(0.1) µm	**d(0.5)** µm	**d(0.9)** µm	**AMOUNT < 2.5 µm** Vol %
3.1	18.5	54.6	8.3

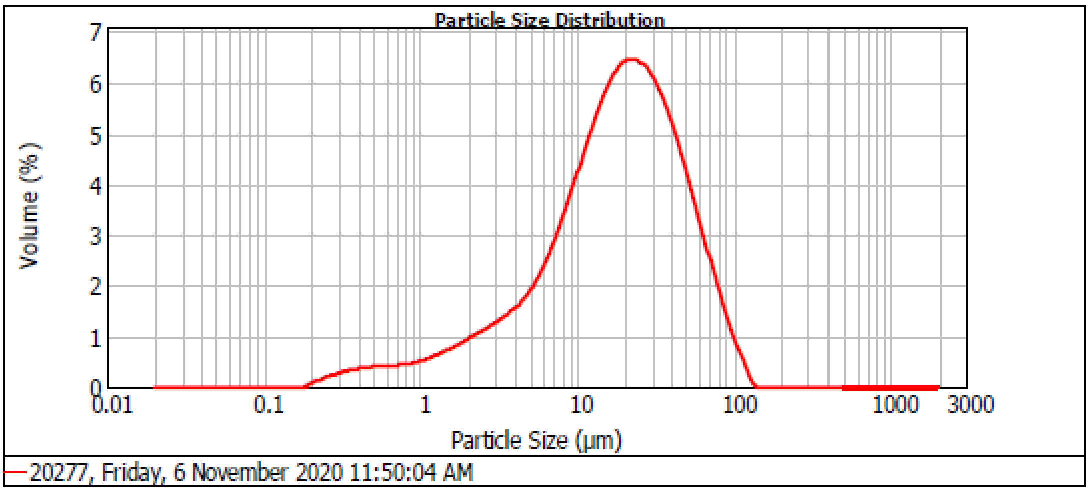

Figure 5. PSD of the sulphate digest leach residues sample dispersed in water.

3.1.2.6. Summary of Characterisation Results for the Sulphate Digest Leach Residues

The sulphate digest leach residue sample includes major crystalline phases rutile (20%), calcium sulphates (gypsum, bassanite and anhydrite (7%)) and silicates (7%), mainly as diopside (6.6%), with minor quartz. The remaining Al, Fe and Si is probably present as x-ray invisible silicates and metal oxides/sulphates. However, together these substances account for only 81% of the material. The total carbon content is only 2%, so a significant portion (17%) of the sample is not defined. Hydration water, especially in amorphous metal oxides, may account for some of the unaccounted material, as the sample was dried to a constant weight at only 60°C to avoid conversion of amorphous phases to crystalline phases. This is consistent with the measured LOI of 18.5%. Trace heavy metals of significance include, in decreasing concentration, Cr, Mn, V, Zn and Ni.

3.1.3. Metal Hydroxide Precipitation #1 Residue – Waste Product #4 (EGi Code 20278)

3.1.3.1. XRF Analysis

Results from XRF analysis of the metal hydroxide precipitation #1 residue sample (Waste Product 4) are provided in Table 11. The full set of results are provided in Appendix A.

Table 11. XRF results: Metal hydroxide precipitation #1 residue sample

ELEMENT	UNIT	CONCENTRATION
Major Elements		
Al	% as Al_2O_3	5.36
Ca	% as CaO	20.8
Cl	%	<0.05
Fe	% as Fe_2O_3	15.70
K	% as K_2O	0.01
Mg	% as MgO	2.80
Na	% as Na_2O	0.13
S	% as SO_3	16.08
Si	% as SiO_2	2.06
Ti	% as TiO_2	4.02
Heavy Metals		
Cr	ppm	1199
Mn	ppm	3237
Ni	ppm	39
V	ppm	2911
Zn	ppm	1637
LOI	%	29.80
Total	%	98.2

3.1.3.2. Bulk Assay

Results from bulk assay of the metal hydroxide precipitation #1 residue sample (Waste Product 4) are provided in Table 12. The full set of results are provided in Appendix B.

Table 12. Bulk assay results: Metal hydroxide precipitation #1 residue sample

ELEMENT	UNIT	CONCENTRATION
Major Elements		
Al	%	2.87
Ca	%	15.20
Cl	%	2.68
Fe	%	11.50
K	%	0.10
Mg	%	1.63
Na	%	0.30
S	%	6.93
Ti	%	2.38
Total C	%	1.1
Organic C	%	0.1
Inorganic C	%	1.0
Heavy Metals		
Cr	ppm	809
Mn	ppm	3060
Nb	99m	>500
Ni	ppm	6
V	ppm	2840
Zn	ppm	1830
Total[1]	%	75.55

1. Calculated using SiO_2 content measured by XRF

3.1.3.3. QXRD Analysis

Results for QXRD analysis of the metal hydroxide precipitation #1 residue sample (Waste Product 4) are provided in Table 13. Diffraction results are provided in Appendix C.

Table 13. QXRD results: Metal hydroxide precipitation #1 residue sample

PHASE	FORMULA	AMOUNT (%)
Bassanite	$CaSO_4.0.5H_2O$	2
Calcite	$CaCO_3$	5
Goethite	FeOOH	4
Gypsum	$CaSO_4.2H_2O$	26
Quartz	SiO_2	1

Amorphous	-	62
SUM		100

3.1.3.4. SEM-EDS Analysis

Typical images obtained during SEM analysis of the metal hydroxide precipitation #1 residue sample (Waste Product 4) are provided in Figures 6 and 7. Full SEM-EDS results are provided in Appendix D. The major results from SEM-EDS analysis are:

The sample contains gypsum and anhydrite (bassanite?) particles in the order of 50 to 100 μm in size (Figures 6 & 7)

Fe and Al oxide/hydroxide, with minor Mg and Ti particles up to 100 μm in size are also relatively abundant (Figure 7)

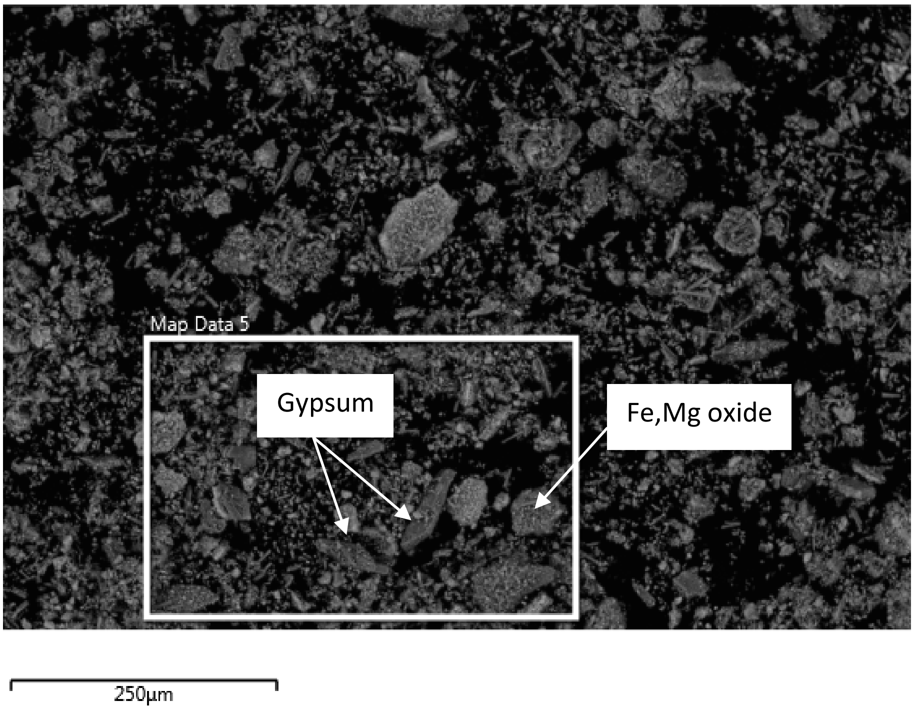

Figure 6. Back-scattered electron image of the metal hydroxide precipitation #1 residue sample showing gypsum and iron magnesium oxide particles.

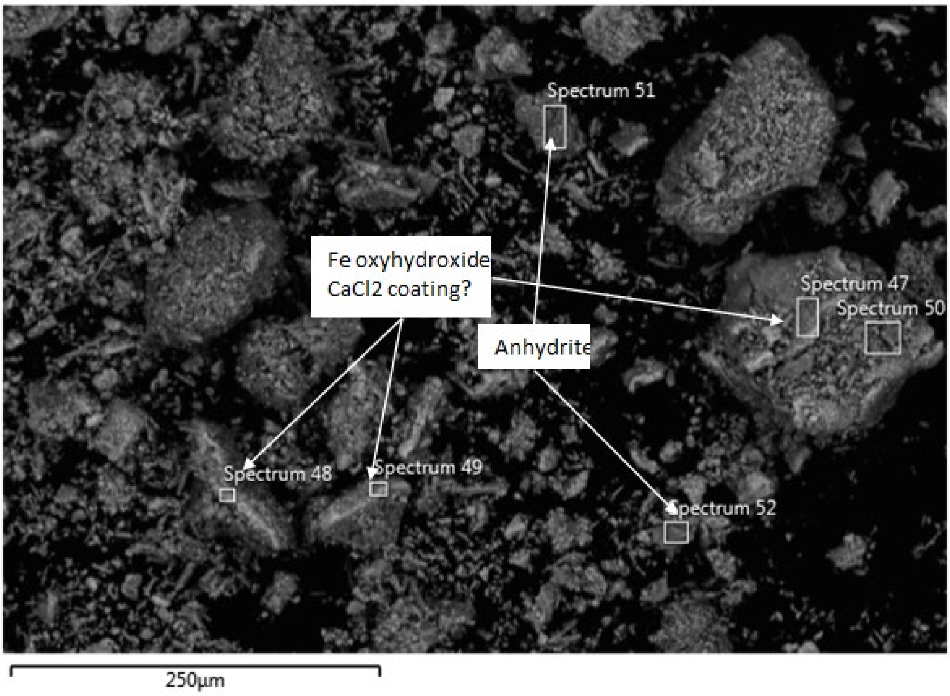

Figure 7. Back-scattered electron image of the metal hydroxide precipitation #1 residue sample showing anhydrite and iron oxide particles.

3.1.3.5. PSD Analysis

Table 14 provides summary statistics from PSD analysis of the Metal hydroxide precipitation #1 residue sample (Waste Product 4) and Figure 8 shows the PSD for this material. Full PSD results are provided in Appendix E.

Results show a bimodal distribution of particles centred around 3 µm and 30 - 40 µm. Comparison of PSD results with SEM results suggest the larger particles to be either calcium sulphate (gypsum and anhydrite) or Fe and/or Al oxide/hydroxide and possibly Fe,Mg oxide/hydroxides. The smaller particles probably include Fe and Al oxide/hydroxides and Na and Ca chlorides. Elemental mapping suggests Si containing particles are very limited and >2.5 µm in size.

Table 14. PSD results: Metal hydroxide precipitation #1 residue sample. D(0.1), d(0.5) and d(0.9) represent the particle sizes under which 10, 50 and 90% of particles by volume respectively are found.

d(0.1) µm	**d(0.5)** µm	**d(0.9)** µm	**AMOUNT < 2.5 µm** Vol %
1.0	6.3	18.1	27.8

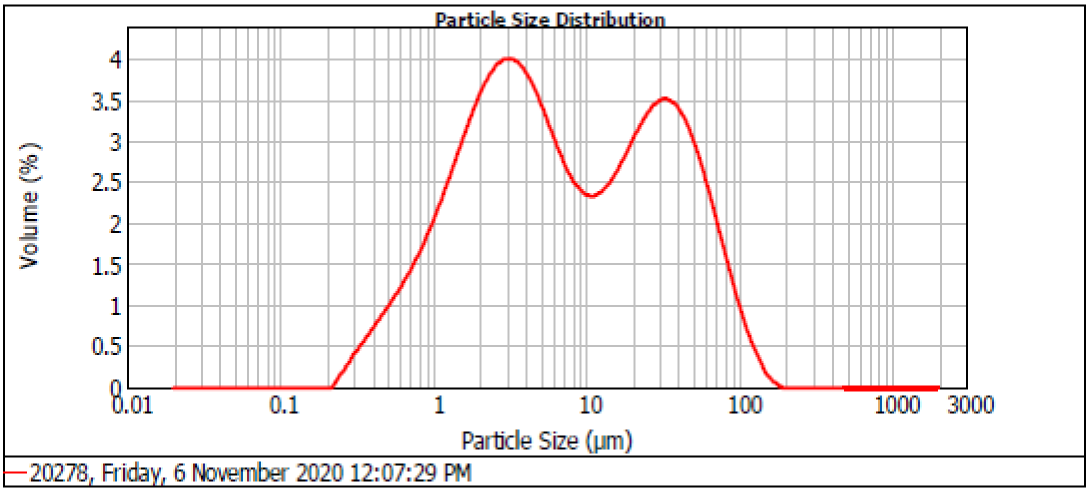

Figure 8. PSD of the metal hydroxide precipitation #1 residue sample dispersed in water.

3.1.3.6. Summary of Characterisation Results for the Metal Hydroxide Precipitation #1 Residue

Calcium sulphate as gypsum and anhydrite/bassanite, together with Fe,Al oxides, some crystalline some amorphous, were the major compounds identified in the metal hydroxide precipitation #1 residue sample. Assayed elements account for only 75% of the mass, suggesting the samples dried to a constant weight at 60°C may still contain hydration water. Results are consistent with the measured LOI of 29.8% (25.8% excluding organic and inorganic C). This material contains significant concentrations of a number of heavy metals including, in decreasing concentration, Mn, V, Zn and Cr. The sample also contains a significant amount of Nb. Approximately 25% of particles are <2.5 µm in size, meaning the respirable content of this material is potentially high, but most of this is likely Fe/Al oxyhydroxides with a low quartz content (≈1%).

3.1.4. Artificial Solution #2 Neutralisation #1 Residue – Waste Product #5 (EGi Code 20529)

3.1.4.1. XRF Analysis

Results from XRF analysis of the artificial solution #2 neutralisation #1 residue (Waste Product 5) are provided in Table 15. The full set of results are provided in Appendix A.

Table 15. XRF results: Artificial solution #2 neutralisation #1 residue sample

ELEMENT	UNIT	CONCENTRATION
Major Elements		
Al	% as Al_2O_3	6.43
Ca	% as CaO	12.40
Cl	%	16.10
Fe	% as Fe_2O_3	38.59
K	% as K_2O	0.08

Mg	% as MgO	5.51
Na	% as Na_2O	0.30
S	% as SO_3	0.17
Si	% as SiO_2	2.49
Ti	% as TiO_2	1.00
Heavy Metals		
Cr	ppm	164
Mn	ppm	1748
Ni	ppm	94
V	ppm	4201
Zn	ppm	<50
LOI	%	27.90
Total	%	108.4

3.1.4.2. Bulk Assay

Results from bulk assay of the artificial solution #2 neutralisation #1 residue (Waste Product 5) are provided in Table 16. The full set of results are provided in Appendix B.

Table 16. Bulk assay results: Artificial solution #2 neutralisation #1 residue sample

ELEMENT	UNIT	CONCENTRATION
Major Elements		
Al	%	2.80
Ca	%	6.10
Cl	%	14.28
Fe	%	21.30
K	%	0.06
Mg	%	2.60
Na	%	0.18
S	%	0.05
Ti	%	0.47
Total C	%	0.5
Organic C	%	0.1
Inorganic C	%	0.4
Heavy Metals		
Cr	ppm	148
Mn	ppm	1280
Nb	ppm	4

Ni	ppm	94
V	ppm	3080
Zn	ppm	1080
Total[1]	%	65.37

1. Calculated using SiO_2 content measured by XRF

3.1.4.3. QXRD Analysis

Results for QXRD analysis of the artificial solution #2 neutralisation #1 residue (Waste Product 5) are provided in Table 17. Diffraction results are provided in Appendix C.

Table 17. QXRD results: Artificial solution #2 neutralisation #1 residue sample

PHASE	FORMULA	AMOUNT (%)[1]
Akaganeite	β-FeOOH,Cl	13.1
Calcite	$CaCO_3$	2.4
Halite	NaCl	0.3
Quartz	SiO_2	0.5
Amorphous	-	83.7
SUM		100

1. The Rietveld refinement of the XRD pattern was not successful due to the inadequacy of available crystal structures for porous, poorly crystalline akaganeite with its highly variable chemical composition (illustrated by its broad diffraction peaks). Consequently, amounts are shown in red and serve as a guide only.

3.1.4.4. SEM-EDS Analysis

Typical images obtained during SEM analysis of the artificial solution #2 neutralisation #1 residue (Waste Product 5) are provided in Figures 9 and 10. Full SEM-EDS results are provided in Appendix D. The major results from SEM-EDS analysis are:

The sample is dominated by large (≈1 mm) particles of iron/aluminium oxide/hydroxide (with minor titanium oxide), all conformably coated with calcium/magnesium chloride.

There are a lesser number of smaller (≈75 µm) aluminosilicate particles which are adhering to the larger oxide particles.

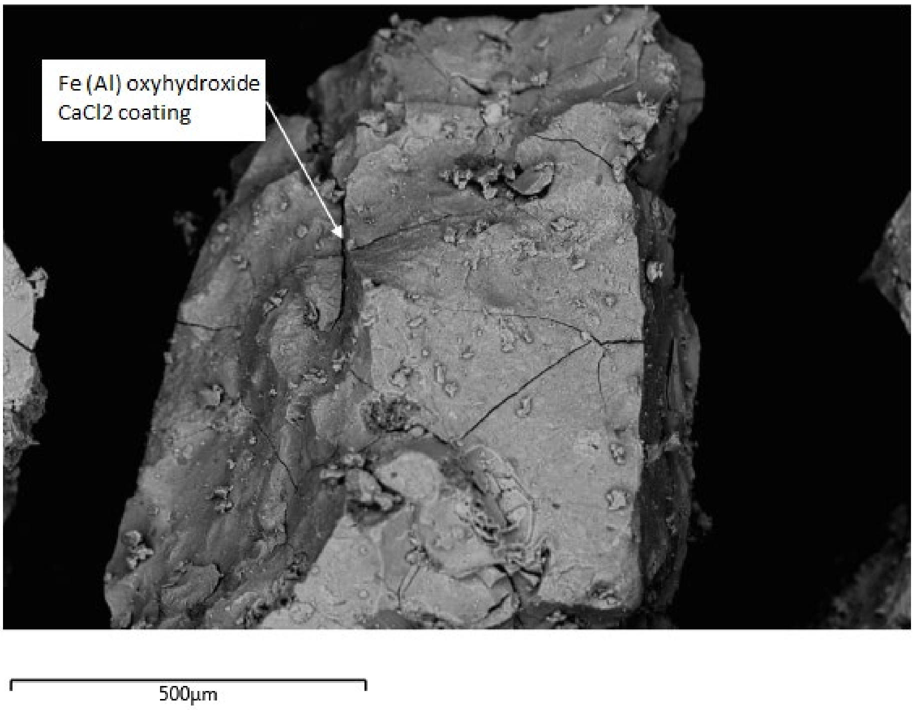

Figure 9. Back-scattered electron image of the artificial solution #2 neutralisation #1 residue sample showing iron/aluminium oxide particle with a coating of amorphous calcium chloride.

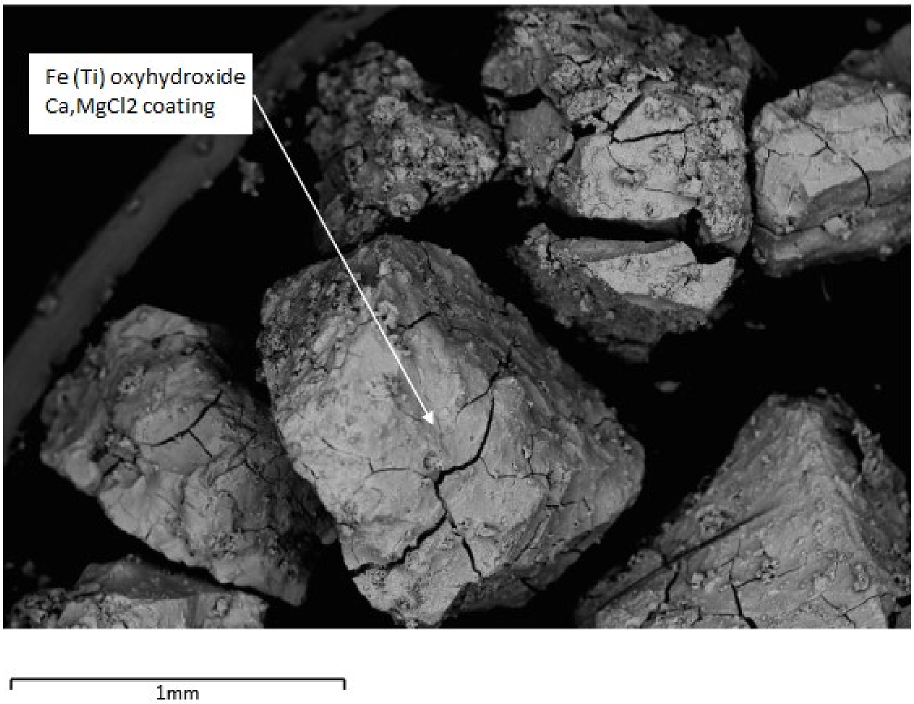

Figure 10. Back-scattered electron image of the artificial solution #2 neutralisation #1 residue sample showing iron/titanium oxide particles with a coating of amorphous calcium/magnesium chloride.

3.1.4.5. PSD Analysis

Table 18 provides summary statistics from PSD analysis of the artificial solution #2 neutralisation #1 residue (Waste Product 5) and Figure 11 shows the PSD for this material. Full PSD results are provided in Appendix E.

Results show a distribution of particles principally between 1 and 70 µm. SEM results show the sample contains very large particles (>0.5 mm) of Fe,Al,Ti oxide/hydroxide which are probably not dispersed during PSD analysis and so do not appear in the analysis. Small fragments, probably of metal oxide/hydroxides and aluminosilicates disperse and appear to indicate particle sizes are much smaller than in reality. There are very few particles <2.5 µm in size, which together with the limited quartz content (2%), suggests the sample is likely to contain little respirable quartz.

Table 18. PSD results: Artificial solution #2 neutralisation #1 residue sample. D(0.1), d(0.5) and d(0.9) represent the particle sizes under which 10, 50 and 90% of particles by volume respectively are found.

d(0.1) µm	**d(0.5)** µm	**d(0.9)** µm	**AMOUNT < 2.5 µm** Vol %
2.8	10.5	33.7	1.6

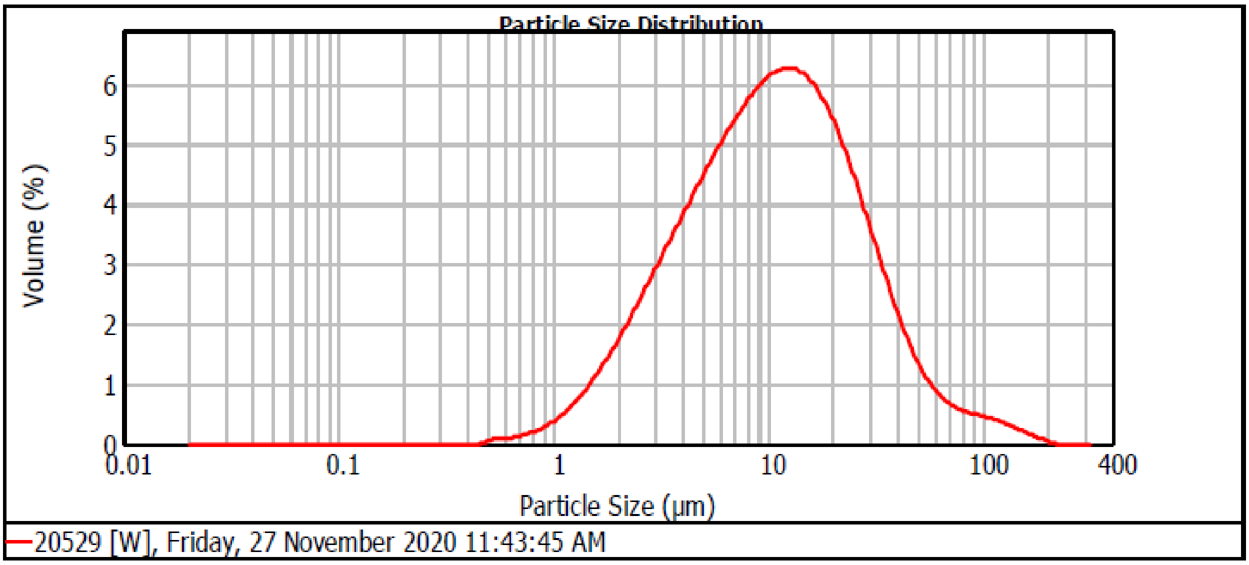

Figure 11. PSD of the artificial solution #2 neutralisation #1 residue sample dispersed in water.

3.1.4.6. Summary of Characterisation Results for the Artificial Solution #2 Neutralisation #1 Residue

The residue precipitated from the artificial solution #2 on neutralisation consists principally of iron oxide/hydroxides, together with lesser amounts aluminium oxide/hydroxide and minor titanium oxide. The particles of metal oxide/hydroxide are coated in calcium/magnesium chloride, possibly as a result of drying the sample prior to analysis which was received as a slurry containing 86% water. In the plant, it is likely that considerably more neutralisation liquor would be removed during press filtering. Consequently, the plant waste neutralisation residue would potentially contain less calcium chloride[1]. The sample also contains a number of heavy metals including in decreasing concentration V, Mn, Zn, Cr, Ni, Nb.

3.1.5. Bulk Crystallisation Slurry Residue – Waste Product #6 (EGi Code 20530)

3.1.5.1. XRF Analysis

Results from XRF analysis of the bulk crystallisation slurry residue (Waste Product 6) are provided in Table 19. The full set of results are provided in Appendix A.

Table 19. XRF results: Bulk crystallisation slurry residue sample

ELEMENT	UNIT	CONCENTRATION
Major Elements		
Al	% as Al_2O_3	0.17
Ca	% as CaO	0.21
Cl	%	46.30
Fe	% as Fe_2O_3	0.09
K	% as K_2O	0.12
Mg	% as MgO	0.17
Na	% as Na_2O	50.72
S	% as SO_3	15.88
Si	% as SiO_2	0.01
Ti	% as TiO_2	0.01
Heavy Metals		
Cr	ppm	<50
Mn	ppm	<50
Ni	ppm	<50
V	ppm	804
Zn	ppm	<50
LOI	%	-
Total	%	103.4

[1] It is noted that mass balance calculations during generation of this waste sample, showed that the majority (82%) of calcium reported to the solution phase after precipitation with lime solution. Results for chloride were not reported, but chloride can be expected to remain in solution. Reported in "Waste Characterisation Testwork Summary" TNG Ltd, 11 December 2020.

3.1.5.2. Bulk Assay

Results from bulk assay of the bulk crystallisation slurry residue (Waste Product 6) are provided in Table 20. The full set of results are provided in Appendix B.

Table 20. Bulk assay results: Bulk crystallisation slurry residue sample

ELEMENT	UNIT	CONCENTRATION
Major Elements		
Al	%	0.04
Ca	%	0.04
Cl	%	44.45
Fe	%	0.06
K	%	0.09
Mg	%	0.02
Na	%	>10
S	%	5.25
Ti	%	<0.00005
Total C	%	0.02
Organic C	%	<0.02
Inorganic C	%	0.02
Heavy Metals		
Cr	ppm	2
Mn	ppm	15
Nb	ppm	0.4
Ni	ppm	0.7
V	ppm	650
Zn	ppm	4
Total	%	72.75

1. Calculated using SiO_2 content measured by XRF

3.1.5.3. QXRD Analysis

Results for QXRD analysis of the bulk crystallisation slurry residue (Waste Product 6) are provided in Table 21. Diffraction results are provided in Appendix C.

Table 21. QXRD results: Bulk crystallisation slurry residue sample

PHASE	FORMULA	AMOUNT (%)
Halite	NaCl	69
Quartz	SiO_2	<1
Thenardite	Na_2SO_4	25
Amorphous	-	6
SUM		100

3.1.5.4. SEM-EDS Analysis

Typical images obtained during SEM analysis of the bulk crystallisation slurry residue (Waste Product 6) are provided in Figure 12. Full SEM-EDS results are provided in Appendix D. The major results from SEM-EDS analysis are:

The sample consists of large (>1 mm) particles of NaCl, coated with smaller (2 – 20 μm) particles of Na_2SO_4.

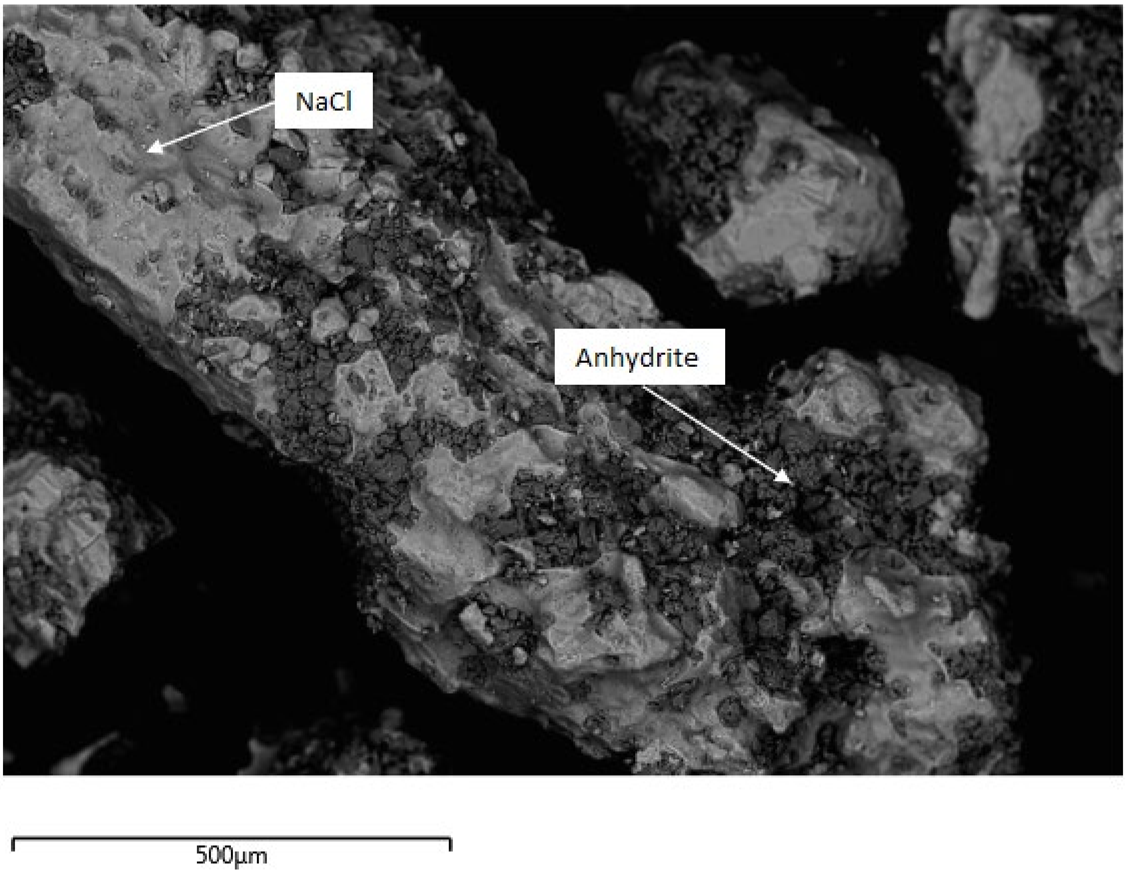

Figure 12. Back-scattered electron image of the bulk crystallisation slurry residue sample showing large sodium chloride particles with a coating of much smaller particles of sodium sulphate.

3.1.5.5. PSD Analysis

Table 22 provides summary statistics from PSD analysis of the bulk crystallisation slurry residue (Waste Product 6) and Figure 13 shows the PSD for this material. Full PSD results are provided in Appendix E.

Results show a distribution of particles principally between 0.2 and 20 µm. SEM results show the sample contains very large particles (>1 mm) of NaCl which are may have rapidly dissolved when dispersed in water during PSD analysis. Consequently, these particles do not appear in the PSD analysis. Small particles, probably of sodium sulphate disperse and appear to indicate particle sizes are much smaller than in reality. Dispersion of this sample in cyclohexane gave a similar result to dispersion in water, with no particles measured above 20 µm, suggesting the larger sodium chloride particles seen in SEM analysis are not well dispersed in cyclohexane. There are very few particles <2.5 µm in size, which together with very low quartz content (<1%), suggests the sample is likely to contain no respirable quartz.

Table 22. PSD results: Bulk crystallisation slurry residue sample. D(0.1), d(0.5) and d(0.9) represent the particle sizes under which 10, 50 and 90% of particles by volume respectively are found.

d(0.1) µm	**d(0.5)** µm	**d(0.9)** µm	**AMOUNT < 2.5 µm** Vol %
0.6	2.7	9.7	3.7

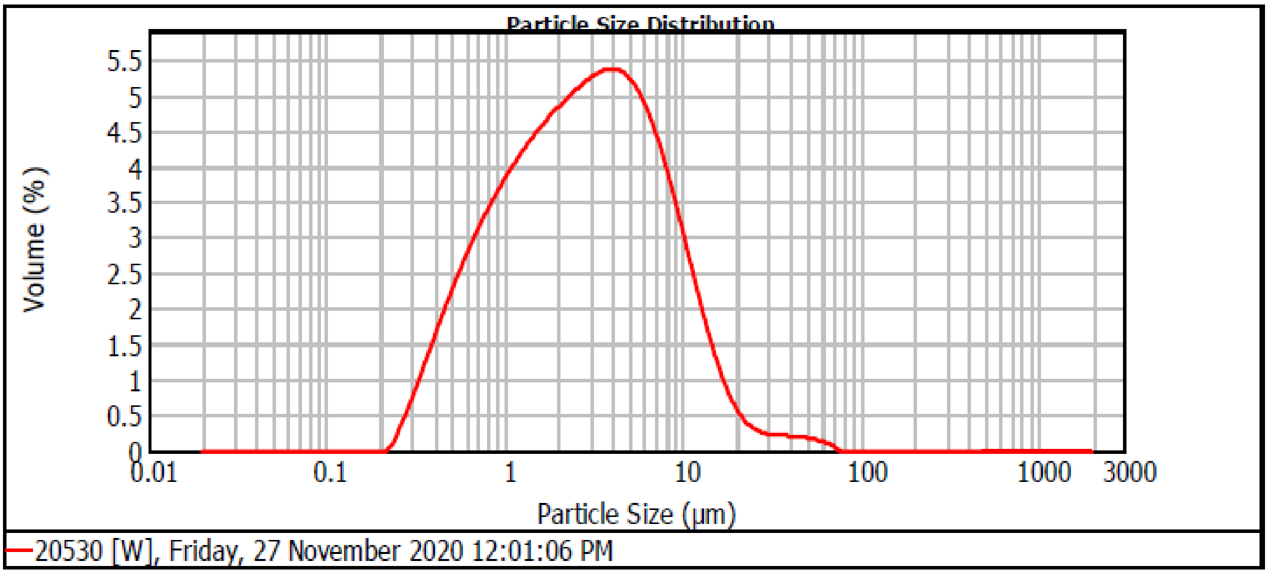

Figure 13. PSD of the bulk crystallisation slurry residue sample dispersed in water.

3.1.5.6. Summary of Characterisation Results for the Bulk Crystallisation Slurry Residue

The bulk crystallisation slurry residue assays for 76% NaCl and 23% Na_2SO_4 (XRF) or 73% NaCl and 27% Na_2SO_4 (ICP/IC). These results agree with results from QXRD analysis which gave 69% NaCl and 25% Na_2SO_4. The sample also contains minor calcium and potassium salts. The only heavy metal at significant levels is vanadium (≈0.1%).

3.1.6. SAS Neutralisation Residue #1 (Gypsum) – Waste Product #3

3.1.6.1. Bulk Assay

Results from bulk assay of the SAS Neutralisation Residue #1 (Gypsum) (Waste Product 3) are provided in Table 23. The full set of results are provided in Appendix B.

Table 23. Bulk assay results: SAS neutralisation residue #1 (Gypsum) sample

ELEMENT	UNIT	CONCENTRATION
Major Elements		
Al	%	2.0
Ca	%	24.3
Fe	%	2.4
K	%	0.09
Mg	%	0.15
Na	%	0.05
S	%	14.1
Ti	%	1.07
Heavy Metals		
Cr	ppm	535
Mn	ppm	390
Nb	ppm	1955
Ni	ppm	<5
V	ppm	1750
Zn	ppm	930
Total	%	81.4

3.1.6.2. Summary of Characterisation Results for the SAS Neutralisation Residue #1 (Gypsum) Sample

The SAS neutralisation residue #1 assays for about 76% gypsum assuming all measure S is present as gypsum (Table 24). All assayed elements account for just over 80% by mass of the sample (not including water of crystallisation in gypsum). Major impurities include iron and aluminium (≈3.5% each as the oxides), titanium (1.8% as the oxide) and silicon (2.6% as the oxide). Major heavy metal impurities include, in decreasing order of abundance, Nb, V, Zn, Cr and Mn (≈0.6% total).

Table 24. Comparison of measured Ca and S concentrations for gypsum and those measured in the SAS neutralisation residue #1 (Gypsum) sample

ELEMENT	REQUIRED FOR GYPSUM (%)	MEASURED (%)
Ca	23.3	24.3
S	18.6	14.1

3.2. Batch Leach Tests

3.2.1. Coal Ash – Waste Product #1 (EGi Code 20276)

The results of the batch leach tests are shown in Figure 14. The full leachate analysis results are provided in Appendix F. The coal ash sample was leached with deionised water covering liquid to solid ratios (LSR) between 2 and 25. Leachates had neutral to slightly acidic pH with very low salinity, principally from Fe, Cl and SO4. Metal concentrations were generally below ANZECC default guideline values (DGV) at all but the lowest LSR, with the exception of Ni which was present at slightly elevated concentrations (up to 10 times the DGV).

Key watershed environmental issues that may be associated with that spillage of the coal ash during handling and transport include: slightly elevated leachable nickel concentrations (the magnitude of which would reduce to negligible concentration upon moderate mixing with surface water flows); and an increased suspended solids load as a result of fine carbon particle fraction. Overall, the watershed risk profile of the material appears to be low to moderate in terms of the readily soluble fraction of this material.

3.2.2. Sulphate Digest Leach Residue – Waste Product #2 (EGi Code 20277)

The sulphate digest leach residue sample was leached with deionised water covering LSR between 2 and 25. Leachates had low pH (1.2 – 1.9), significant acidity (1400 – 9200 mg $CaCO_3$/L) and high salinity principally from calcium and sulphate which appear to be at equilibrium with gypsum at all LSR (Figure 14).

Leachate also contained metals including Al, B, Ba, Cd, Co, Cr, Ni, U, V and Zn at concentrations up to 4 orders of magnitude above the DGVs and across all LSR.

Key watershed environmental issues that maybe associated with that spillage of the sulphate digest leachate residue during handling and transport include: significant loadings of acidity, salinity and the forementioned metals and metalloids. Overall, the watershed risk profile of the material appears to be high in terms of the readily soluble fraction of this material.

The sample received for testing had been treated with "a small amount of limestone added to neutralise residual acid". Clearly insufficient limestone was added, and additional limestone or lime would be required to reduce the risk associated with potential spillage of this waste material during handling and transport.

3.2.3. Metal Hydroxide Precipitation #1 Residue – Waste Product #4 (EGi Code 20278)

The metal hydroxide precipitation #1 residue sample was leached with deionised water covering LSR between 5 and 25. Batch leach tests at lower LSR afforded insufficient leachate for analysis. Leachates were slightly alkaline (pH around 9) and had high salinity (5 – 13.5 g/L) principally from calcium, chloride, and sulphate. Calcium and sulphate may be at equilibrium with gypsum at all LSR (Figure 14).

Metal concentrations in leachates were generally very low, presumable due to low solubility at higher pH. However, both Al and Cr concentrations in leachate were one to two orders of magnitude above DGVs, particularly at higher LSR. This suggests solubility control on these metals, with soluble aluminium possibly present as an anionic species (e.g., $Al(OH)_4^-$).

Key watershed environmental issues that maybe associated with that spillage of the metal hydroxide precipitation #1 residue during handling and transport include: significant loadings of salinity; elevated leachable aluminium and chromium (although moderate mixing with surface water flows likely to reduce concentrations and lowering of the pH which may result in precipitation). Overall, the watershed risk profile of the material appears to be low to moderate in terms of the readily soluble fraction of this material.

It should be noted that this sample was received at approximately 77% moisture content which was reduced through drying to 54% before using for batch leach testing. This will have resulted in increased salinity, and leachates from metal hydroxide precipitation #1 residue produced in the plant would likely have lower salinity at the same LSR.

3.2.4. Artificial Solution #2 Neutralisation #1 Residue – Waste Product #5 (EGi Code 20529)

The artificial solution #2 neutralisation #1 residue sample was leached with deionised water covering LSR between 6 and 25. Batch leach tests at lower LSR afforded insufficient leachate for analysis. Leachates had neutral pH and high salinity (9 – 27 g/L) principally from calcium and chloride (Figure 14). The sample was received containing significant excess water and, after decanting supernatant, the moisture content was measured at 84%. This sample was then used for batch leach testing. The high salinity of leachates from the artificial solution #2 neutralisation #1 residue sample as a result of high Ca and Cl concentrations, is potentially derived from pore water rather than the solid phase. In the plant it is likely that considerably more neutralisation liquor would be removed during press filtering. Consequently, the amount of calcium and chloride in the plant waste neutralisation residue pore water could be less than in the laboratory sample and consequently leachates may have lower salinity.

Key watershed environmental issues that maybe associated with that spillage of the artificial solution #2 neutralisation #1 residue during handling and transport include: elevated leachable concentrations of a number of metals including Ba and Sr [higher concentrations of these metals are potentially as a result of the high concentration of calcium chloride in leachates. Barite ($BaSO_4$) and celestite ($SrSO_4$) solubility increases as calcium chloride concentration increases[2]] as well as Al, Cu and Zn (the magnitude of which would reduce to negligible concentration upon moderate mixing with surface water flows). Overall, the watershed risk profile of the material appears to be low to moderate in terms of the readily soluble fraction of this material.

3.2.5. Bulk Crystallisation Slurry Residue – Waste Product #6 (EGi Code 20530)

The bulk crystallisation slurry residue sample was leached with deionised water covering LSR between 0.5 and 10. Because of the expected high solubility of this waste material, lower LSR were used than for the other waste products. Essentially all of the material dissolved at LSR above 5 (Figure 15), producing highly saline (80 – 170 g/L), moderately low pH (3 – 4.5) leachates. Leachates also contained Al, Cu, V and Zn concentrations two to three orders of magnitude above DGVs.

Key watershed environmental issues that maybe associated with that spillage of the bulk crystallisation slurry residue during handling and transport include: significant loadings of acidity, salinity and the forementioned metals and

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[2] C. Monnin and C. Galinier, 1988. The solubility of celestite and barite in electrolyte solutions and natural waters at 25°C. A thermodynamic study, *Chemical Geology*, **71**, 283-296.

metalloids. Overall, the watershed risk profile of the material appears to be moderate to high in terms of the readily soluble fraction of this material.

3.2.6. SAS Neutralisation Residue #1 (Gypsum) – Waste Product #3

Batch leaching tests were not conducted on the gypsum sample. However, assay results (Section 3.1.6.1) show the major phase as gypsum, so dissolution would be expected to result in solutions containing calcium and sulphate in equilibrium with undissolved gypsum. This would limit leachate salinity. This product was precipitated at a solution pH around 5.4 and, as a consequence, it could be expected that leachates would have neutral to slightly acidic pH. The dissolution behaviour of major impurities in the gypsum including V, Nb, Zn, Cr and Mn are difficult to predict as mineral associations for these elements have not been established.

Batch extraction test work is required to assess the watershed risk profile of the material (i.e., in terms of the readily soluble fraction of this material).

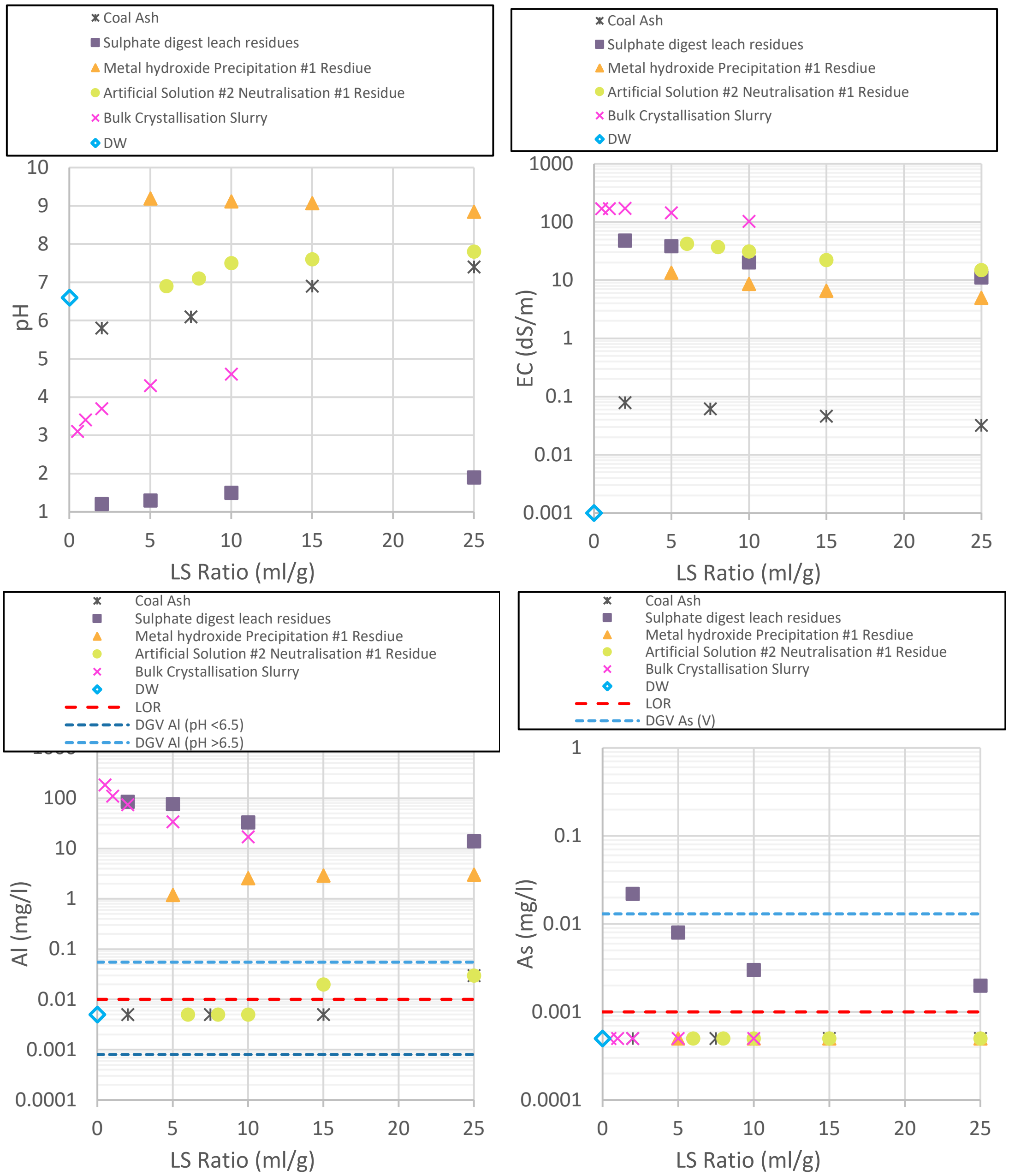

Figure 14. Results for batch leach tests. The open blue diamond represents a blank extraction. The broken red line indicates the analyte limit of reporting. Results reported as <LOR are plotted as LOR/2. The broken blue line represents the ANZECC default guideline value for 95% species protection in a freshwater system.

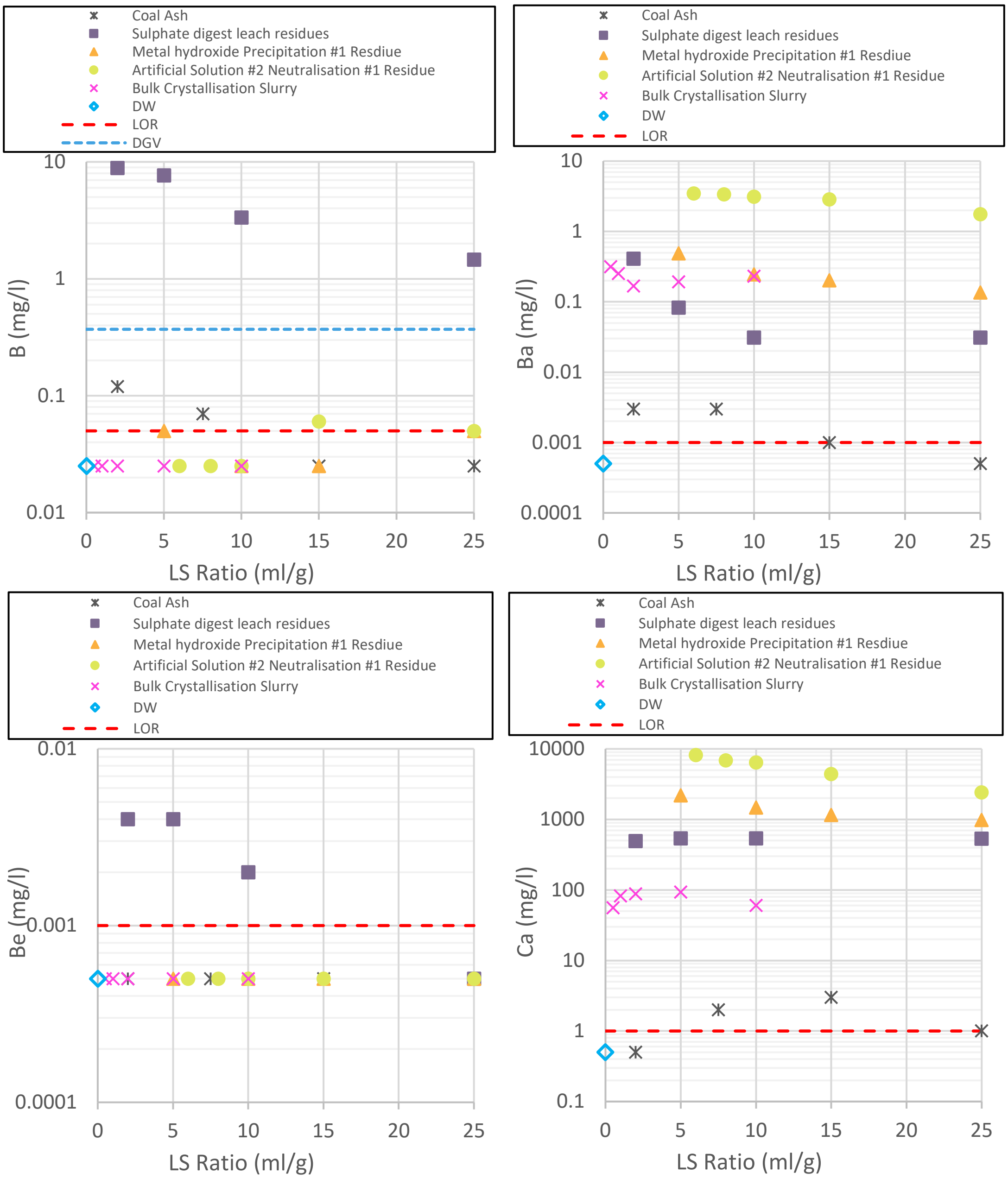

Figure 14 Cont. Results for batch leach tests. The open blue diamond represents a blank extraction. The broken red line indicates the analyte limit of reporting. Results reported as <LOR are plotted as LOR/2. The broken blue line represents the ANZECC default guideline value for 95% species protection in a freshwater system.

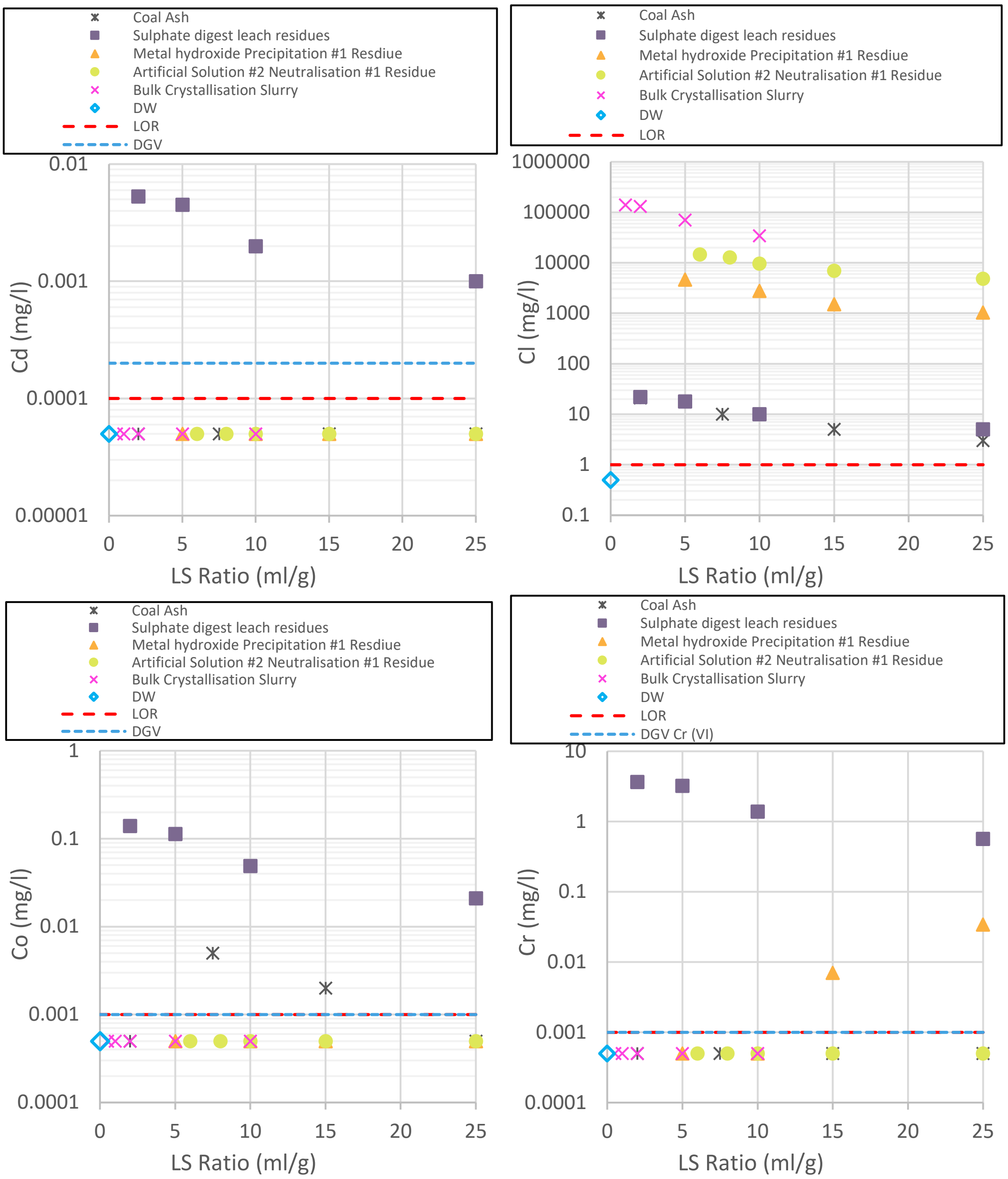

Figure 14 Cont. Results for batch leach tests. The open blue diamond represents a blank extraction. The broken red line indicates the analyte limit of reporting. Results reported as <LOR are plotted as LOR/2. The broken blue line represents the ANZECC default guideline value for 95% species protection in a freshwater system.

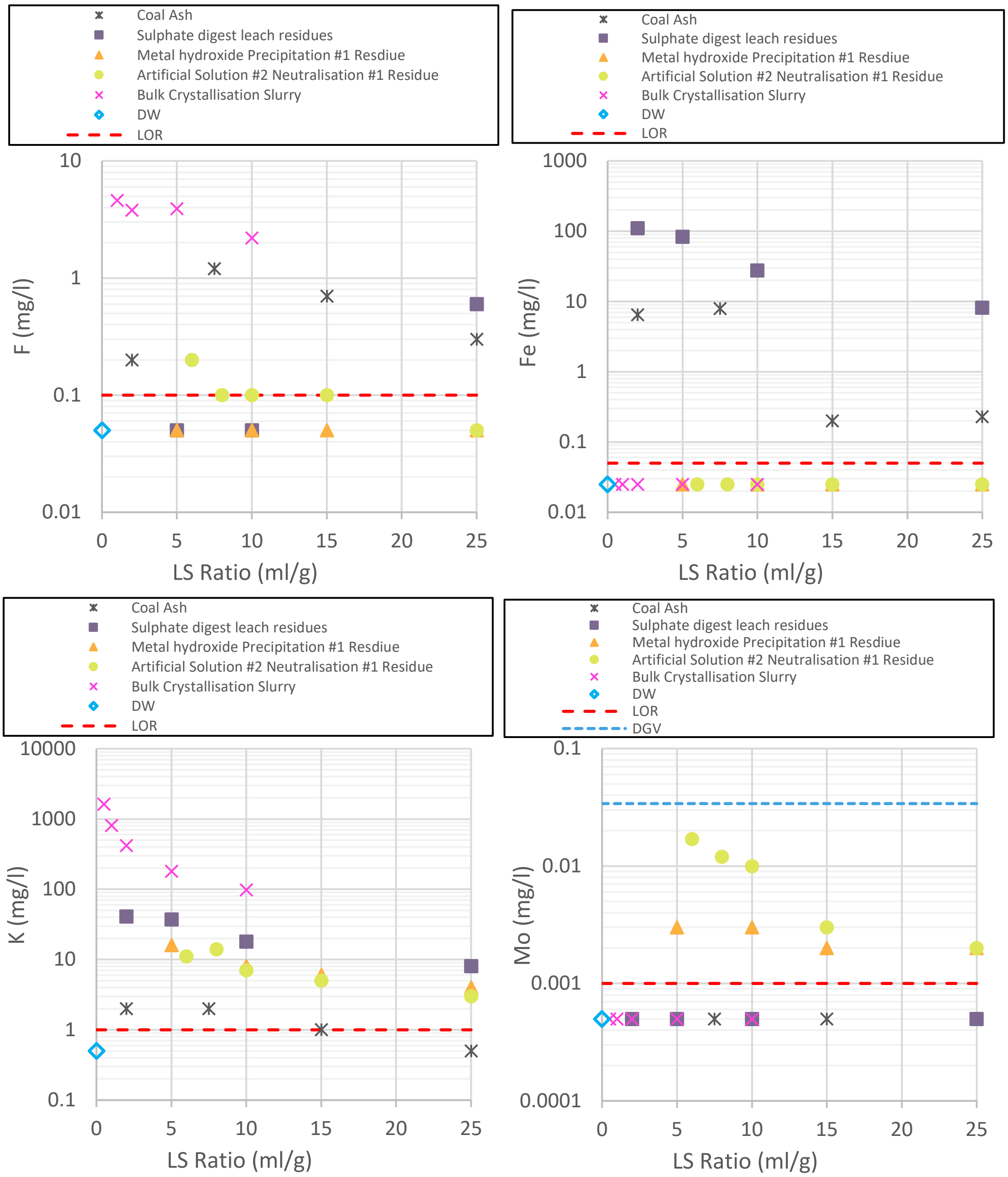

Figure 14 Cont. Results for batch leach tests. The open blue diamond represents a blank extraction. The broken red line indicates the analyte limit of reporting. Results reported as <LOR are plotted as LOR/2. The broken blue line represents the ANZECC default guideline value for 95% species protection in a freshwater system.

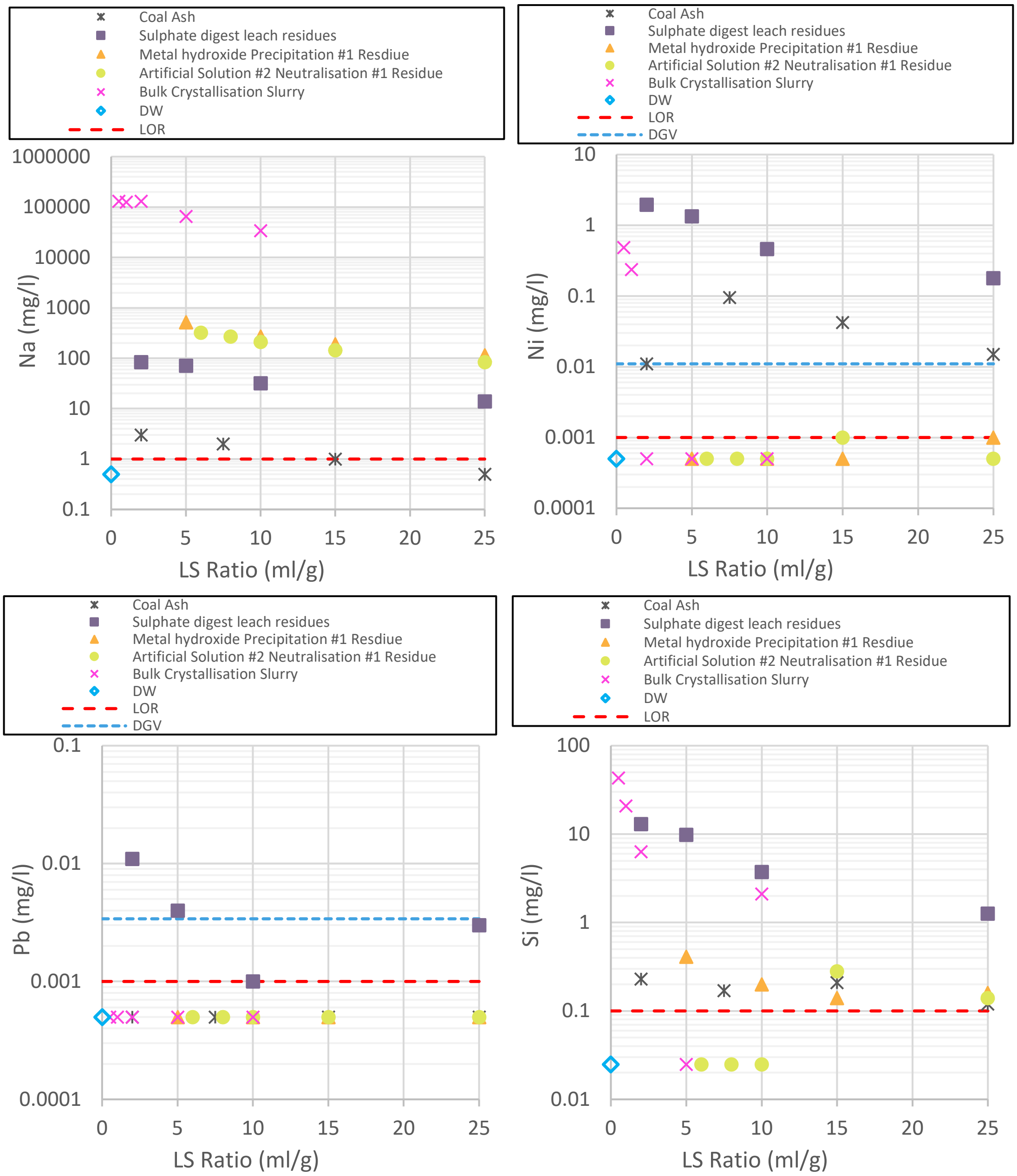

Figure 14 Cont. Results for batch leach tests. The open blue diamond represents a blank extraction. The broken red line indicates the analyte limit of reporting. Results reported as <LOR are plotted as LOR/2. The broken blue line represents the ANZECC default guideline value for 95% species protection in a freshwater system.

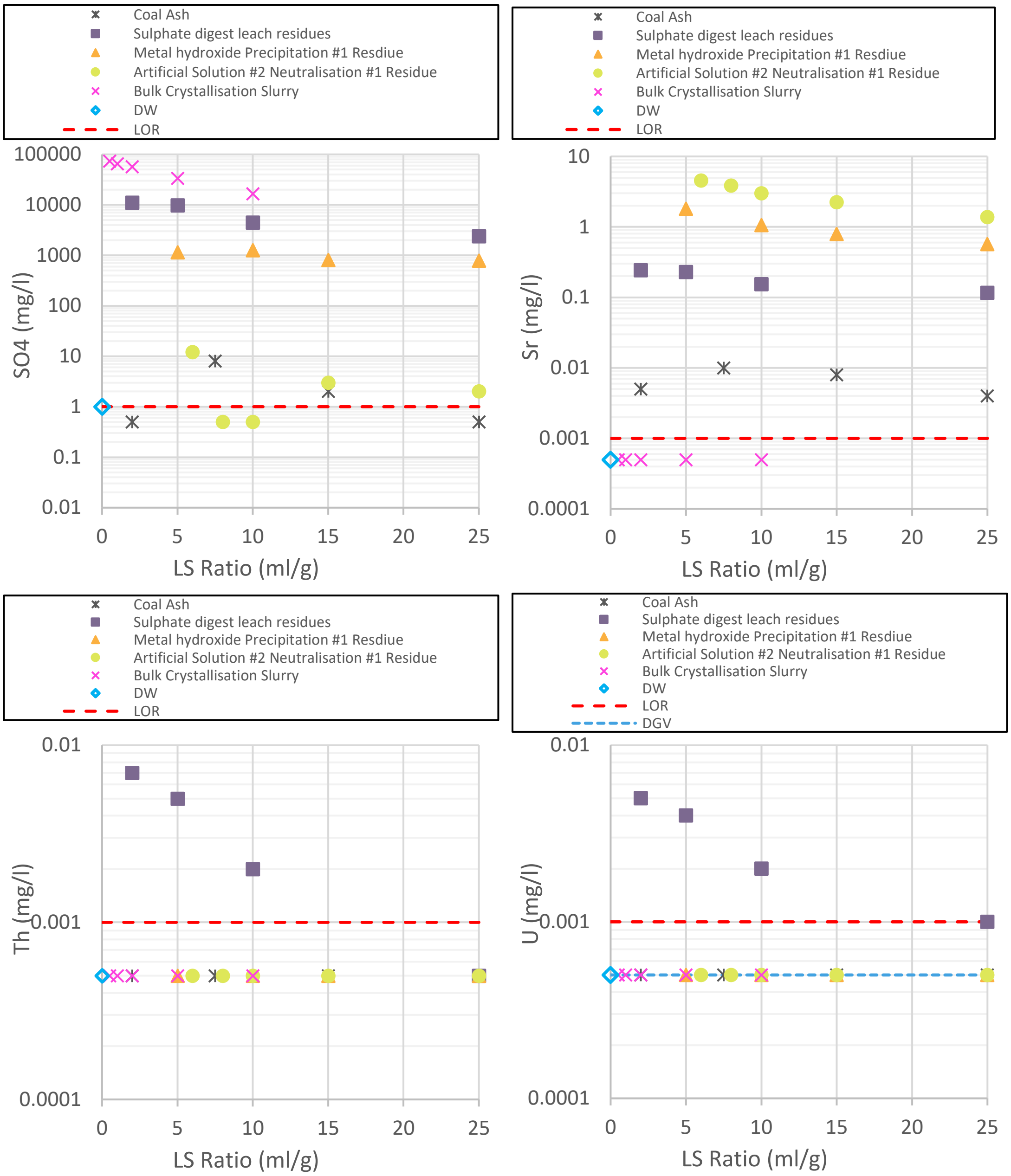

Figure 14 Cont. Results for batch leach tests. The open blue diamond represents a blank extraction. The broken red line indicates the analyte limit of reporting. Results reported as <LOR are plotted as LOR/2. The broken blue line represents the ANZECC default guideline value for 95% species protection in a freshwater system.

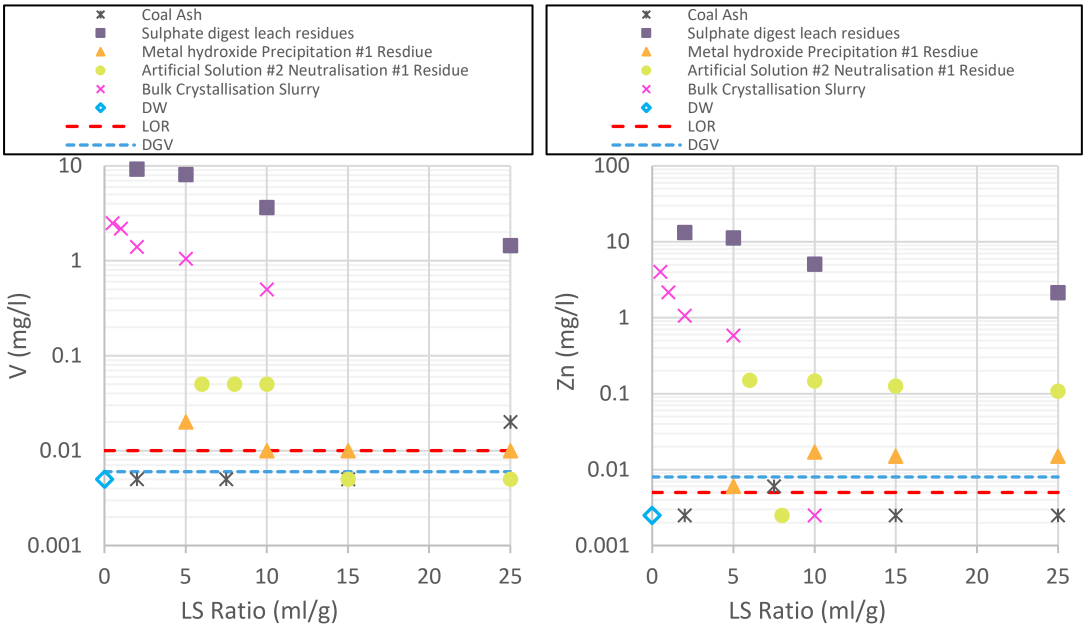

Figure 14 Cont. Results for batch leach tests. The open blue diamond represents a blank extraction. The broken red line indicates the analyte limit of reporting. Results reported as <LOR are plotted as LOR/2. The broken blue line represents the ANZECC default guideline value for 95% species protection in a freshwater system. Note the DGV for vanadium is a low reliability trigger value for freshwater systems and should be considered as an indicative value only.

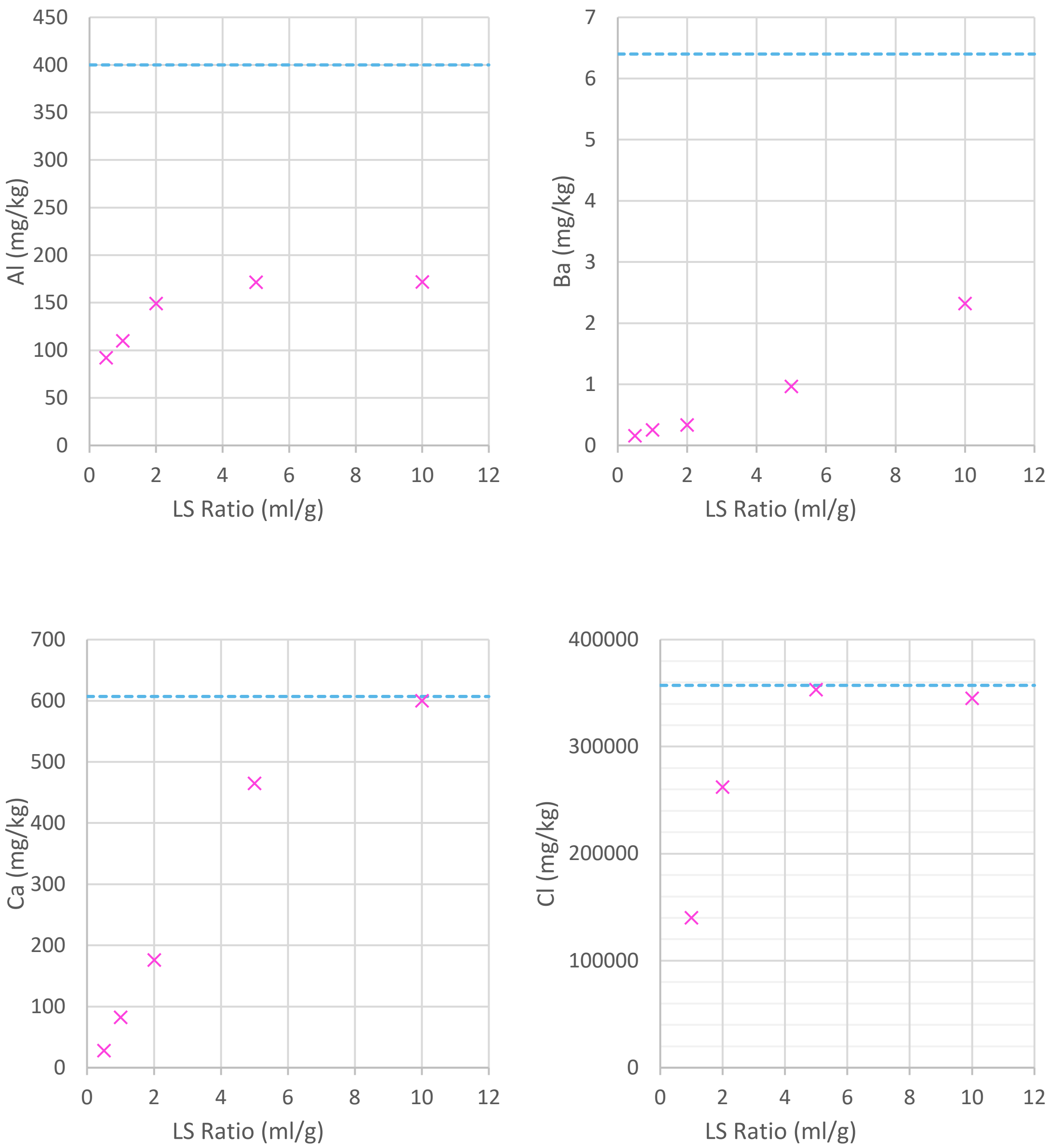

Figure 15. Results for batch leach tests for the bulk crystallisation slurry residue. The broken blue line indicates the maximum amount of each element that could be leached from the sample.

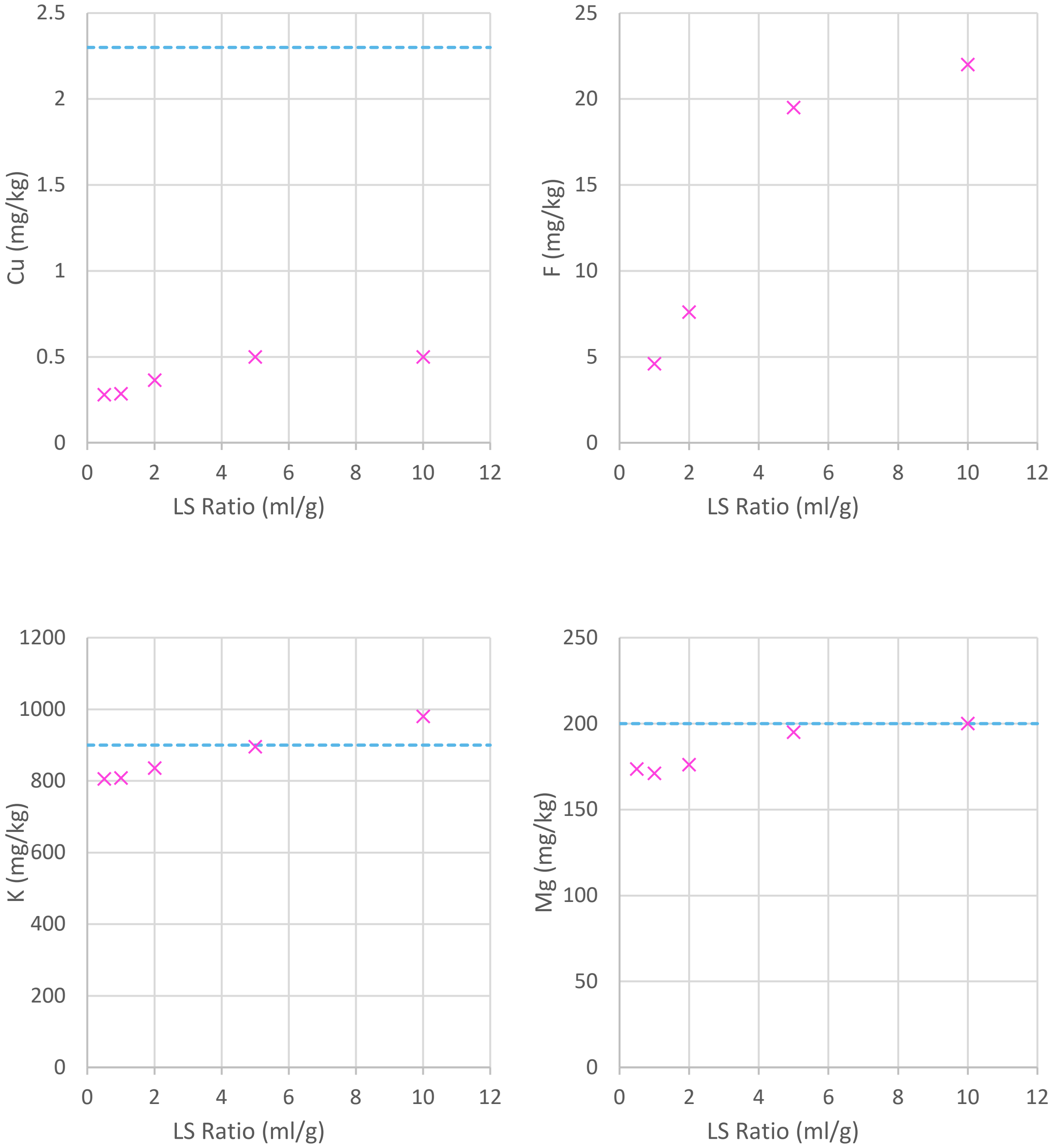

Figure 16 Cont. Results for batch leach tests for the bulk crystallisation slurry residue. The broken blue line indicates the maximum amount of each element that could be leached from the sample.

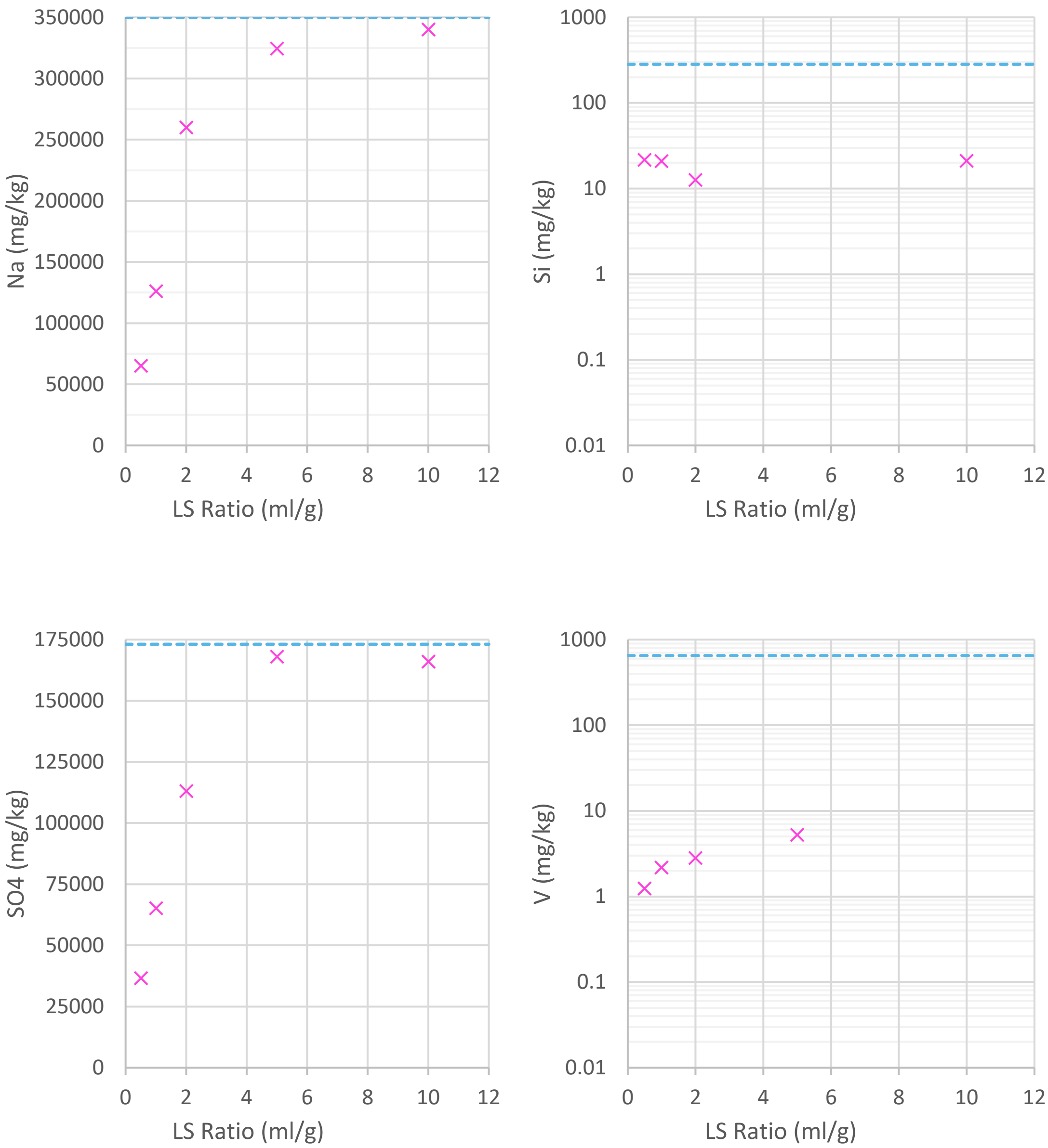

Figure 17 Cont. Results for batch leach tests for the bulk crystallisation slurry residue. The broken blue line indicates the maximum amount of each element that could be leached from the sample.

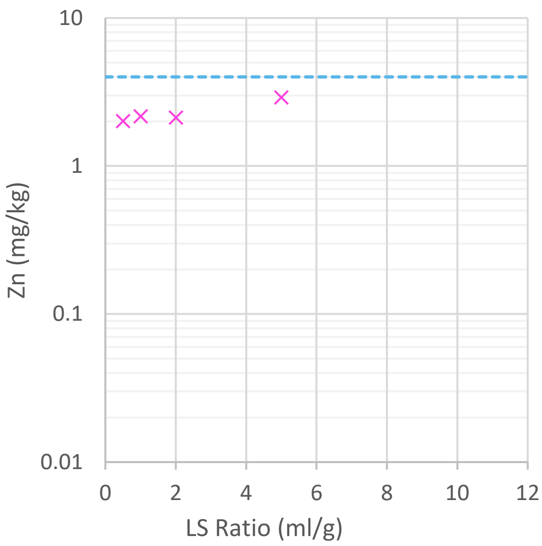

Figure 18 Cont. Results for batch leach tests for the bulk crystallisation slurry residue. The broken blue line indicates the maximum amount of each element that could be leached from the sample.

4. CONCLUSIONS

The Mount Peake Project Darwin Processing Plant will produce several solid waste products, many of which will be shipped by rail back to Mount Peake for disposal in an integrated waste landform. Six of these waste materials were created to simulate waste products that will be generated by TNG's proposed Darwin process plant. Wastes are described in the Table below:

Table 25. Details of samples examined in this work

WASTE PRODUCT #	DESCRIPTION	ORIGIN
1	Coal Ash	TIVAN Process
5	Artificial Solution #2 Neutralisation #1 Residue	TIVAN Process
2	Sulphate digest leach residues	Titanium Pigment Plant
3[1]	SAS Neutralisation #1 Residue (Gypsum)	Titanium Pigment Plant
4	Metal Hydroxide Precipitation #1 Residue	Titanium Pigment Plant
6	Bulk Crystallisation Slurry	Wastewater Treatment Plant

1. This sample was not tested by EGi. Elemental assay results were received from TNG.

It should be noted that these conclusions are primarily in relation to the watershed risk profile of these materials and if a detailed ecological risk assessment of the transport and handling of these materials is required, it would require a baseline sampling and analysis programme (i.e., to determine pre-existing condition of surface soils, waterway beds and water quality along the transportation corridor).

4.1. TIVAN Process Wastes

Wastes from the TIVAN process (#1 and #5) contain iron, aluminium and titanium oxides/hydroxides as major components together with a number of heavy metals (Cr, Mn, Ni, Nb, V and Zn) as minor elements. The coal ash is predominantly carbon, together with calcium silicates and metallic iron which are not present in the neutralisation residue. The latter waste contains significant calcium chloride, not present in the coal ash.

Water leachates from the neutralisation residue had high salinity due to the presence of calcium chloride. Both wastes gave slightly elevated metals (Ni, Al, Cu, and Zn) in water leachates, but the watershed risk profile for transport and handling of these materials is considered low to moderate in terms of the readily soluble fraction.

4.2. Titanium Pigment Plant Wastes

Wastes from the Titanium Pigment Plant (#2 - #4) contain iron, aluminium, titanium and silicon oxides/hydroxides as major components together with a number of heavy metals (Cr, Mn, Ni, Nb, V and Zn) as minor elements. The other major compounds common to all wastes are varying amounts of calcium sulphate ($CaSO_4.nH_2O$, n = 0, 0.5, 2).

Leachates from waste #4 had elevated salinity due to the presence of calcium sulphates and slightly elevated Al and Cr. Similar leachates can be expected from the gypsum precipitate (waste #3), but this needs testing to confirm. Wastes from the Darwin Process Plant represented by these materials are unlikely to present a significant risk during handling and transport.

The sulphate digest leach residue sample which was produced by neutralisation of the sulphate digest residue with limestone, produced water leachates with low pH, high acidity and elevated heavy metals at concentrations significantly above environmental guidelines.

Overall, the watershed risk profile for transport and handling of materials represented by the sulphate digest residue sample is considered high. Addition of more neutralising material, preferably as lime rather than limestone, is recommended to reduce the risks associated with potential spillage of this waste during handling and transport.

4.3. Wastewater Treatment Plant Waste

The Bulk Crystallisation Slurry sample produced by concentration of a synthetic solution is composed of sodium chloride (≈75%) and sodium sulphate (≈25%). The sample also contains minor calcium and potassium salts. The only heavy metal at significant levels is vanadium (≈0.1%).

Water leachates from the bulk crystallisation slurry residue were highly saline and slightly acidic. Leachates also contained elevated concentrations of a number of metals (Al, Cu, V and Zn). The watershed risk profile for transport and handling of these materials was considered moderate to high in terms of the readily soluble fraction.

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APPENDIX A
XRF Results

Appendix A: XRF Results

EGi Code		20276	20277	20278	20529	20530
Solid Waste Product		1	2	4	5	6
Description		Coal Ash	Sulphate digest leach residues	Metal Hydroxide Precipitation #1 Residue	Artificial Solution #2 Neutralisation #1 Residue	Bulk Crystallisation Slurry
Al2O3	(%)	5.68	4.63	5.36	6.43	0.167
CaO	(%)	1.28	4.68	20.8	12.4	0.21
CoO	(%)	0.007	0.060	0.019	0.000	0.003
CuO	(%)	0.246	0.006	<0.005	<0.005	<0.005
Cr2O3	(%)	0.132	0.276	0.175	0.024	0.000
Fe2O3	(%)	11.6	2.46	15.7	38.6	0.087
K2O	(%)	0.122	1.12	0.01	0.077	0.123
MgO	(%)	0.75	1.32	2.8	5.51	0.17
Mn3O4	(%)	0.094	0.103	0.45	0.243	<0.005
Na2O	(%)	0.06	0.4	0.13	0.302	50.72
NiO	(%)	0.211	0.008	0.005	0.012	<0.005
P2O5	(%)	0.020	0.059	0.067	0.033	<0.005
PbO	(%)	<0.005	<0.005	<0.005	<0.005	<0.005
SO3	(%)	0.02	1.30	16.1	0.17	15.9
SiO2	(%)	8.55	40.9	2.06	2.49	0.01
TiO2	(%)	2.370	23.70	4.020	0.999	0.000
V2O5	(%)	0.285	0.235	0.520	0.750	0.144
ZnO	(%)	0.113	0.031	0.204	<0.005	<0.005
ZrO2	(%)	0.009	0.031	<0.005	<0.005	<0.005
Cl	(%)	<0.05	<0.05	<0.05	16.1	46.3
LOI	(%)	68.9	18.5	29.8	27.9	
Total	(%)	100.4	99.8	98.2	108.4	103.4

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APPENDIX B

Bulk Assay Results

Appendix B: Bulk Assay Results

EGi Code	20276	20277	-	20278	20529	20530
Solid Waste Product	1	2	3	4	5	6
Description	Coal Ash	Sulphate digest leach residues	SAS Neutralisation #1 Residue (Gypsum)	Metal Hydroxide Precipitation #1 Residue	Artificial Solution #2 Neutralisation #1 Residue	Bulk Crystallisation Slurry
Ag	0.09	0.11	<1	1.34	0.03	<0.01
Al	2.63%	2.26%	1.98%	2.87%	2.80%	0.04%
As	2.9	16.3	<50	1.6	5.7	2.2
Ba	20	250	15	20	20	<10
Be	0.24	0.87	<0.5	0.22	0.23	<0.05
Bi	0.06	0.01	<0.1	0.07	0.88	0.38
Ca	0.92%	3.29%	24.30%	15.20%	6.10%	0.04%
Cd	0.09	0.03	<0.5	0.14	0.06	<0.02
Ce	12.55	17.85	4.5	5.74	6.04	0.27
Co	38.5	4.8	<5	3.2	9.7	0.2
Cr	817	1380	535	809	148	2
Cs	0.81	4.96	<0.5	0.44	0.13	0.07
Cu	58.5	22.4	20	4	51.2	2.3
Fe	7.79%	1.82%	2.35%	11.50%	21.30%	0.06%
Ga	29.3	10.8	<5	4.37	2.36	0.19
Ge	0.14	0.2	<5	0.19	0.16	0.14
Hf	1.1	7.8	<0.5	0.5	0.2	<0.1
Hg	0.01	0.006	<0.01	0.008	0.094	0.068
In	0.174	0.011	<0.1	0.007	0.033	<0.005
K	0.10%	0.91%	0.09%	0.10%	0.06%	0.09%
La	9.2	9.4	3.0	3.3	3.3	<0.5
Li	17.7	7.4	<1	9.2	1.8	0.4
Mg	0.44%	0.73%	0.15%	1.63%	2.60%	0.02%
Mn	639	630	390	3060	1280	15
Mo	30.4	47.7	<1	0.4	13.75	0.08
Na	0.09%	0.22%	0.05%	0.30%	0.18%	>10%
Nb	6.7	25.9	1955	>500	4	0.4
Ni	1880	23.4	<5	6	94	0.7
P	100	240	370	300	120	10
Pb	7.8	2	<10	14.2	11.9	1.3
Rb	4.4	40.1	3.0	4.6	2.8	0.4
Re	0.031	0.008	<0.05	0.002	0.004	<0.002

EGi Code	20276	20277	-	20278	20529	20530
Solid Waste Product	1	2	3	4	5	6
Description	Coal Ash	Sulphate digest leach residues	SAS Neutralisation #1 Residue (Gypsum)	Metal Hydroxide Precipitation #1 Residue	Artificial Solution #2 Neutralisation #1 Residue	Bulk Crystallisation Slurry
S	0.31%	3.43%	14.14%	6.93%	0.05%	5.25%
Sb	0.21	1.68	<0.5	0.17	0.39	<0.05
Sc	7	16.6	1	1.1	1.1	<0.1
Se	<1	11	<5	2	<1	<1
Si			1.20%			
Sn	1.9	1.6	0.5	2.4	3.4	0.2
Sr	17.1	53.9	45	74.1	48.8	0.7
Ta	6.11	2.53	2.0	3.23	0.14	<0.05
Te	<0.05	<0.05	<0.1	0.75	<0.05	<0.05
Th	7.25	1.82	0.3	0.91	1.8	0.03
Ti	1.46%	>10%	1.07%	2.38%	0.47%	<0.00005
Tl	0.28	<0.02	<0.05	0.04	0.04	<0.05
U	0.5	0.6	0.3	0.5	0.3	<0.1
V	1400	491	1750	2840	3080	650
W	1.7	5.4	<1	0.8	206	105
Y	4	9.6	3.5	3.1	3.4	0.1
Zn	787	163	930	1830	1080	4
Zr	36.4	227	5	13.5	9.1	1
Cl	0.01%	0.30%	-	2.68%	14.28%	44.45%
SO4	0.07%	0.18%	-	0.31%	0.41%	17.31%
Total C	68.0%	1.6%	-	1.1%	0.5%	0.02%
Organic C	66.2%	1.6%	-	0.1%	0.1%	<0.02%
Inorganic C	1.8%	<0.02%	-	1.0%	0.4%	0.02%

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APPENDIX C
XRD Results

Appendix C: XRD Results – Coal Ash

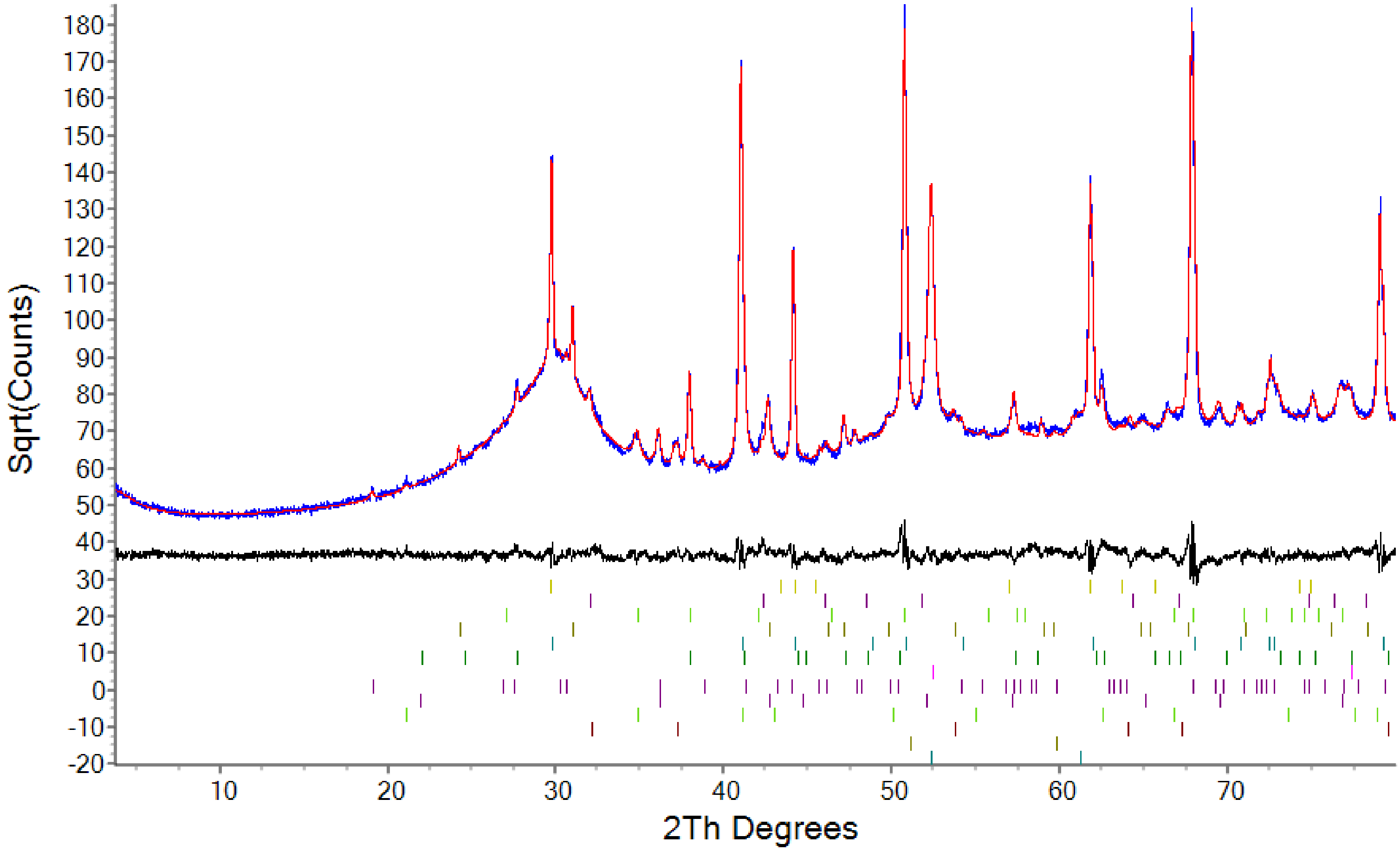

Figure C19 Output from Rietveld analysis of the Coal Ash sample (20276) where the blue line is the observed data, the red is the calculated pattern and the black the difference. The vertical lines are the Bragg peak markers for (top to bottom) anatase, rutile, calcite, quartz, corundum (added as internal standard), ilmenite, iron, mullite, hercynite, magnetite, halite, taenite, and nickel.

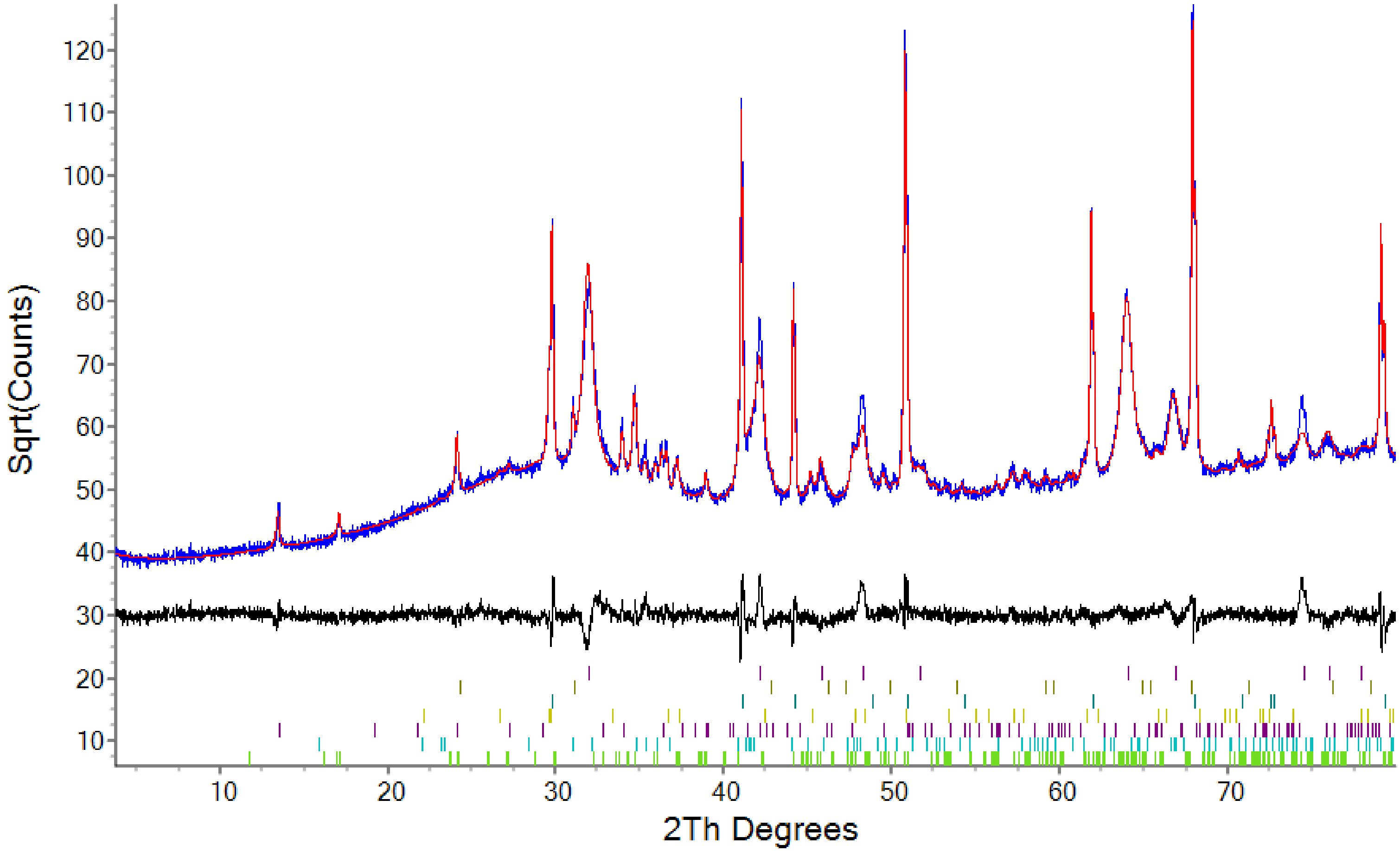

Figure C20 Output from Rietveld analysis of the Sulphate Digest Leachate Residue sample (20277) where the blue line is the observed data, the red is the calculated pattern and the black the difference. The vertical lines are the Bragg peak markers for (top to bottom) rutile, quartz, corundum (added as internal standard), anhydrite, gypsum, diopside, and bassanite.

Appendix C: XRD Results – Metal Hydroxide Precipitation #1 Residue

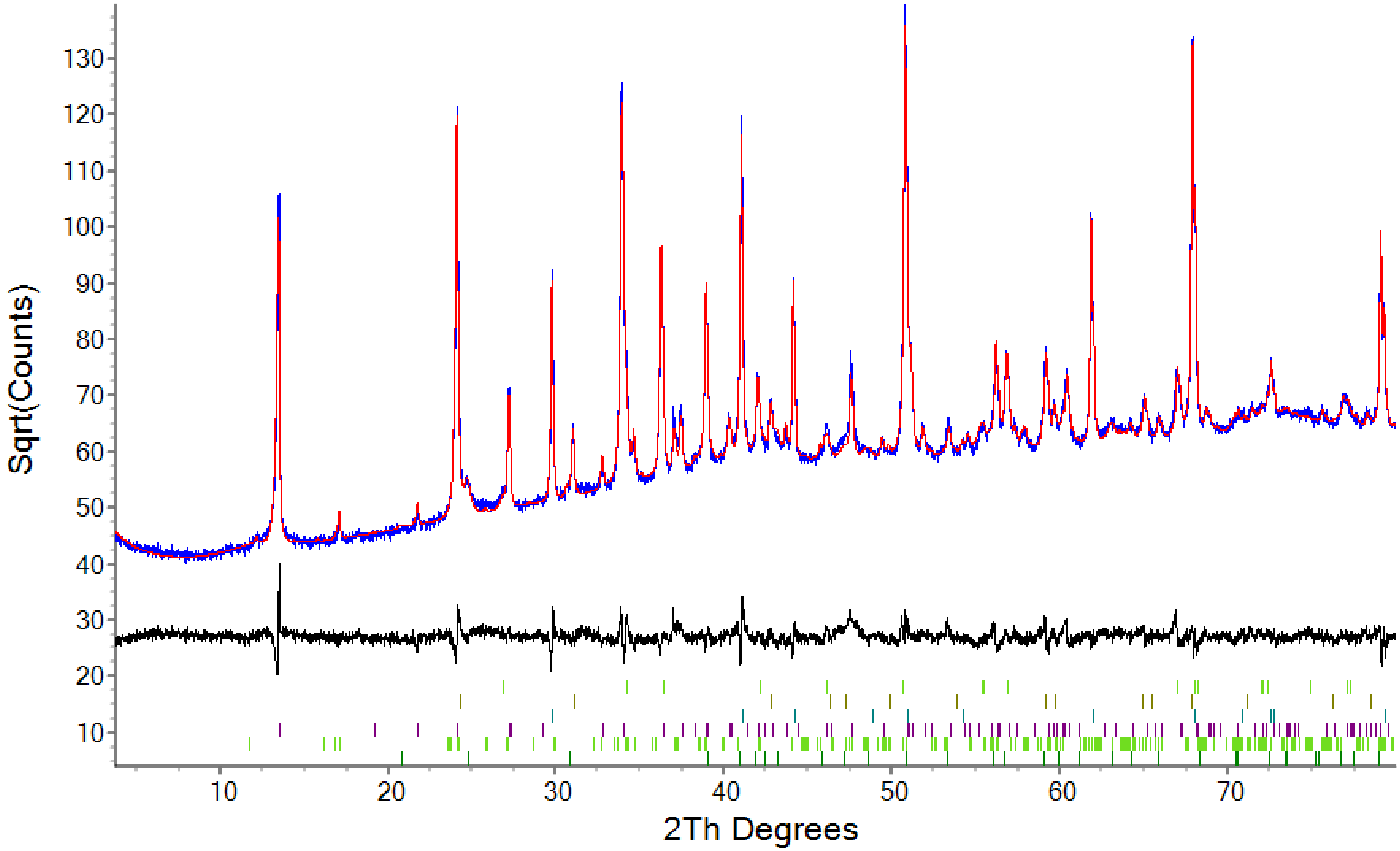

Figure C21 Output from Rietveld analysis of the Metal Hydroxide Precipitation #1 Residue sample (20278) where the blue line is the observed data, the red is the calculated pattern and the black the difference. The vertical lines are the Bragg peak markers for (top to bottom) calcite, quartz, corundum (added as internal standard), gypsum, bassanite, and goethite.

Appendix C: XRD Results – Artificial Solution #2 Neutralisation #1 Residue

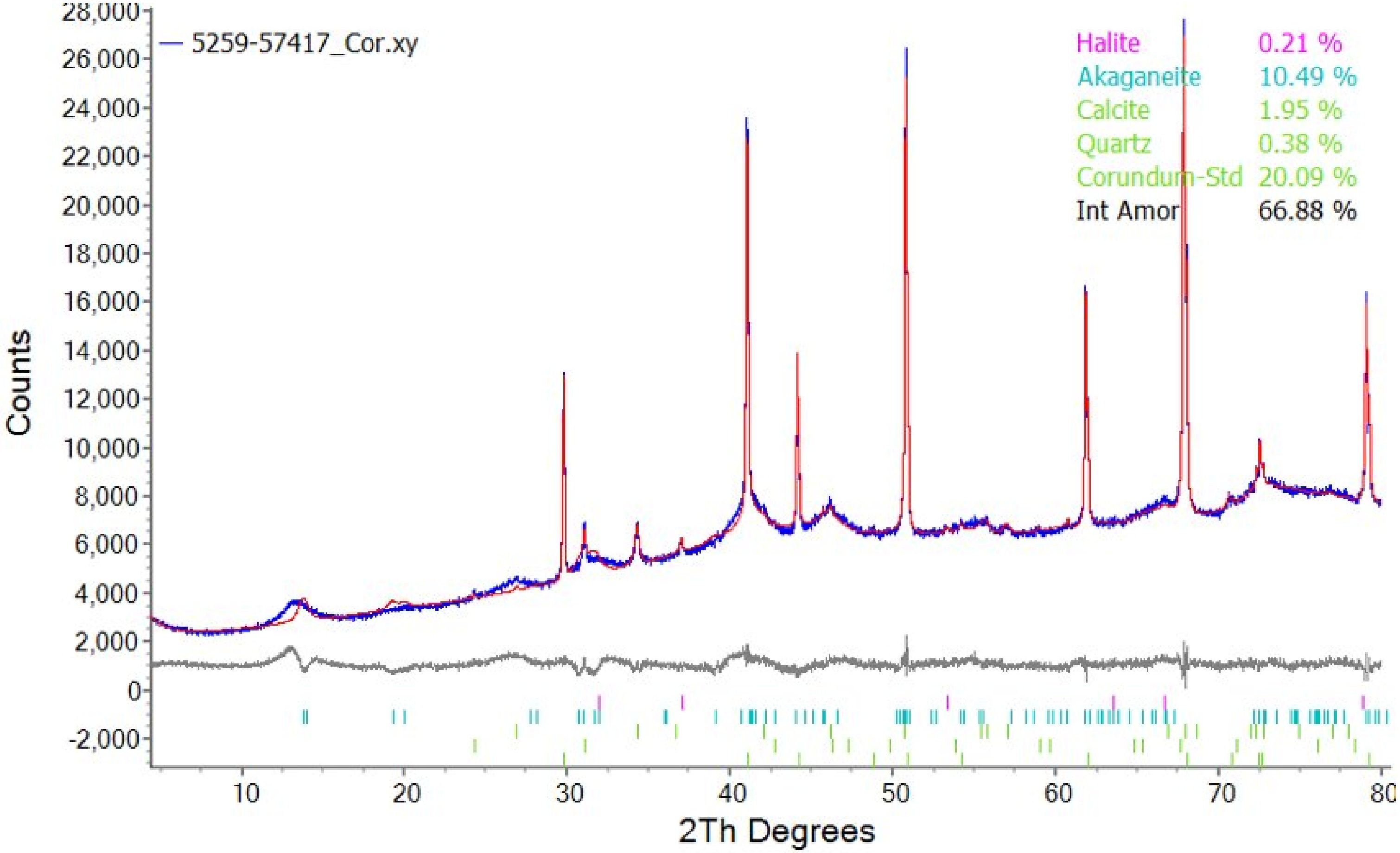

Figure C4 Output from Rietveld analysis of the Artificial Solution #2 Neutralisation #1 Residue sample (20529) where the blue line is the observed data, the red is the calculated pattern and the black the difference. The vertical lines are the Bragg peak markers for (top to bottom) halite, akaganeite, calcite, quartz and corundum (added as internal standard).

Appendix C: XRD Results – Bulk Crystallisation Slurry

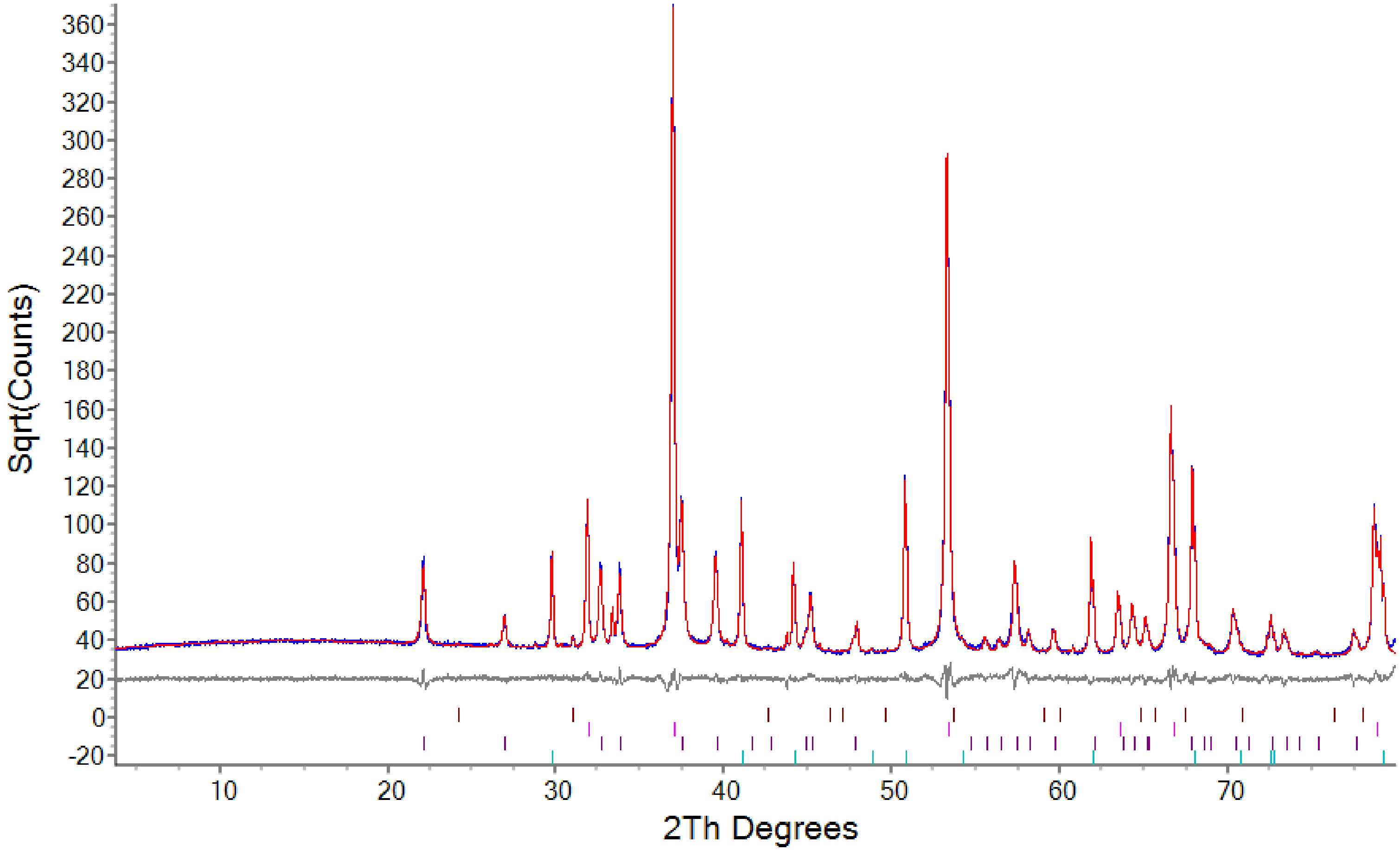

Figure C5 Output from Rietveld analysis of the Bulk Crystallisation Slurry sample (20530) where the blue line is the observed data, the red is the calculated pattern and the black the difference. The vertical lines are the Bragg peak markers for (top to bottom) quartz, halite, thenardite, and corundum (added as internal standard).

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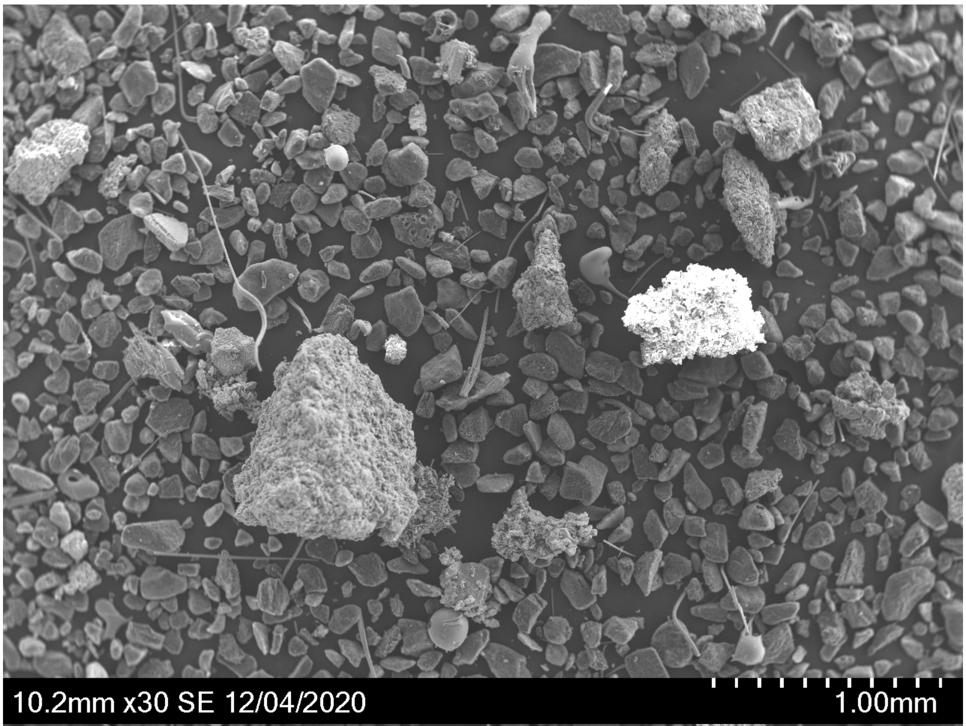
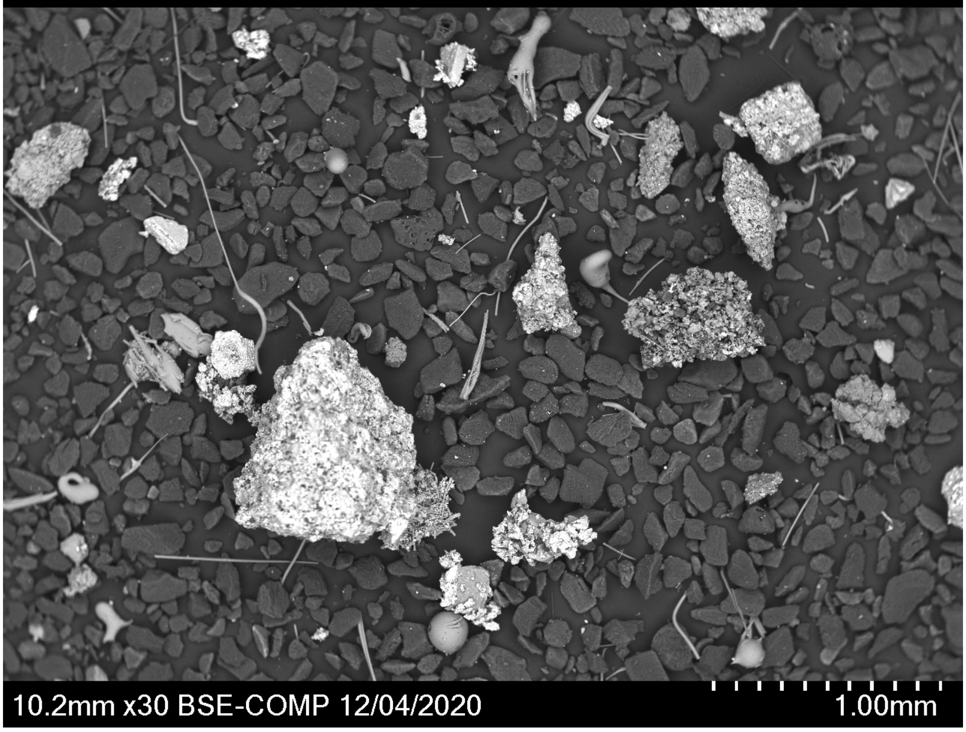
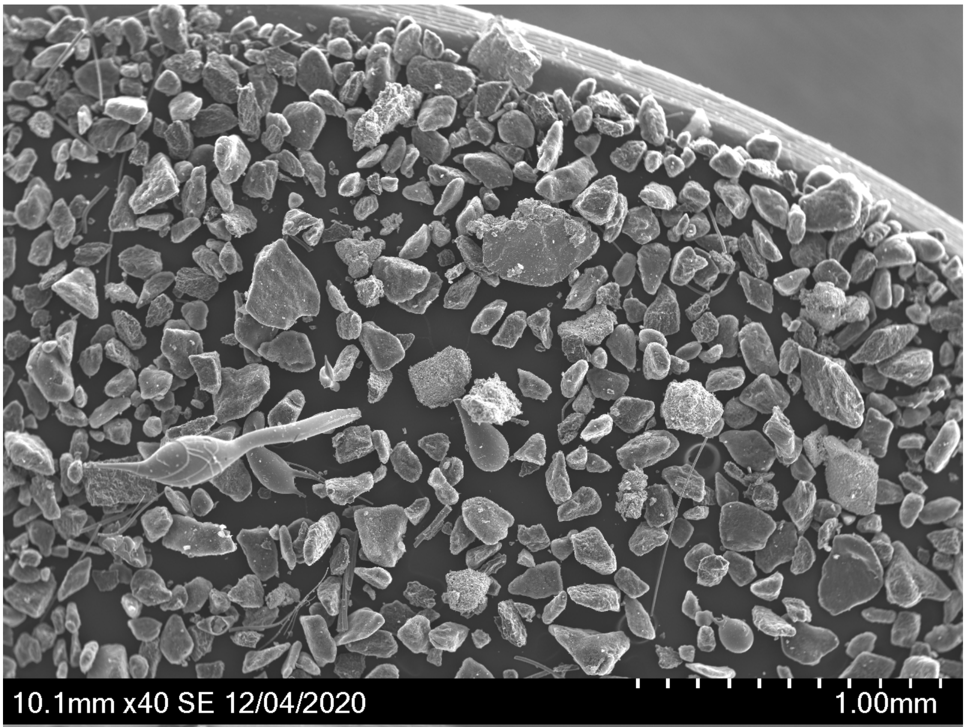
APPENDIX D

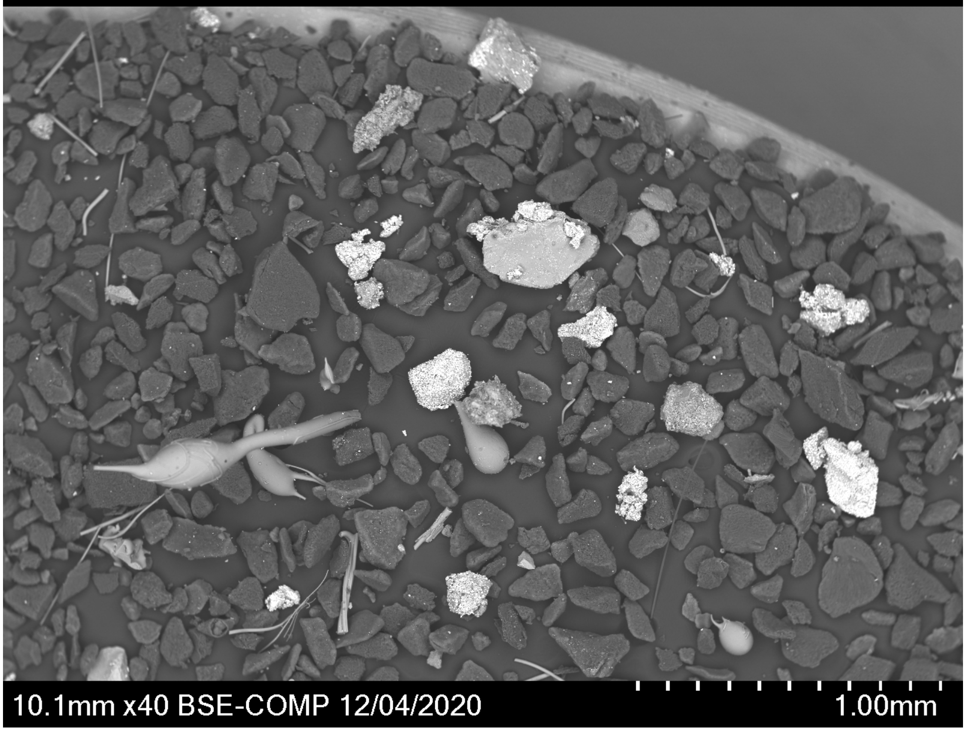
SEM-EDS Results

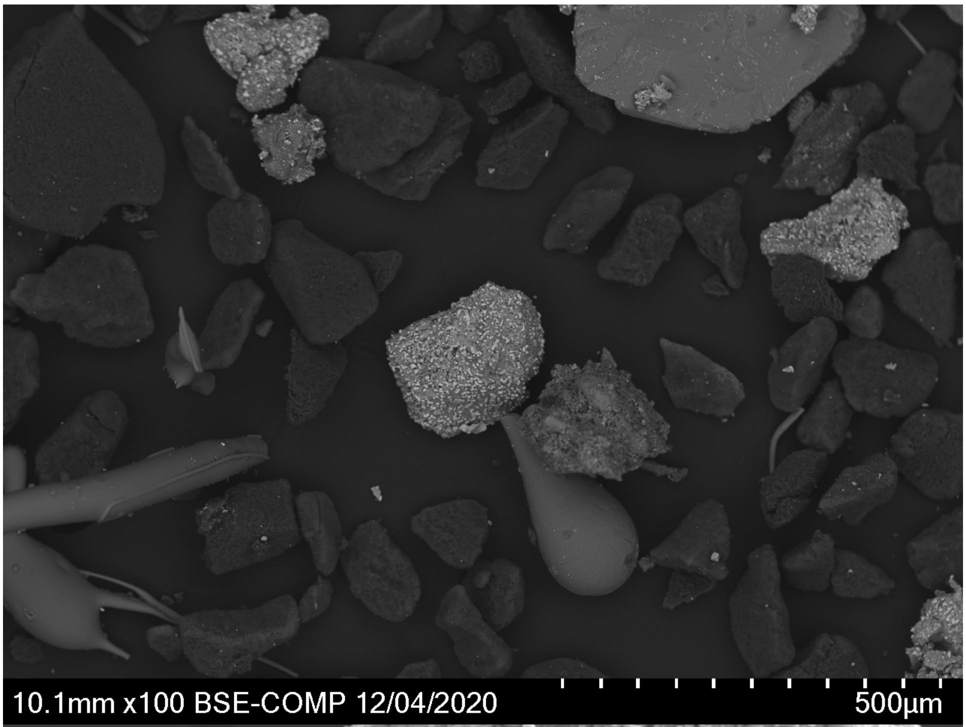
Appendix D: SEM Images – Coal Ash

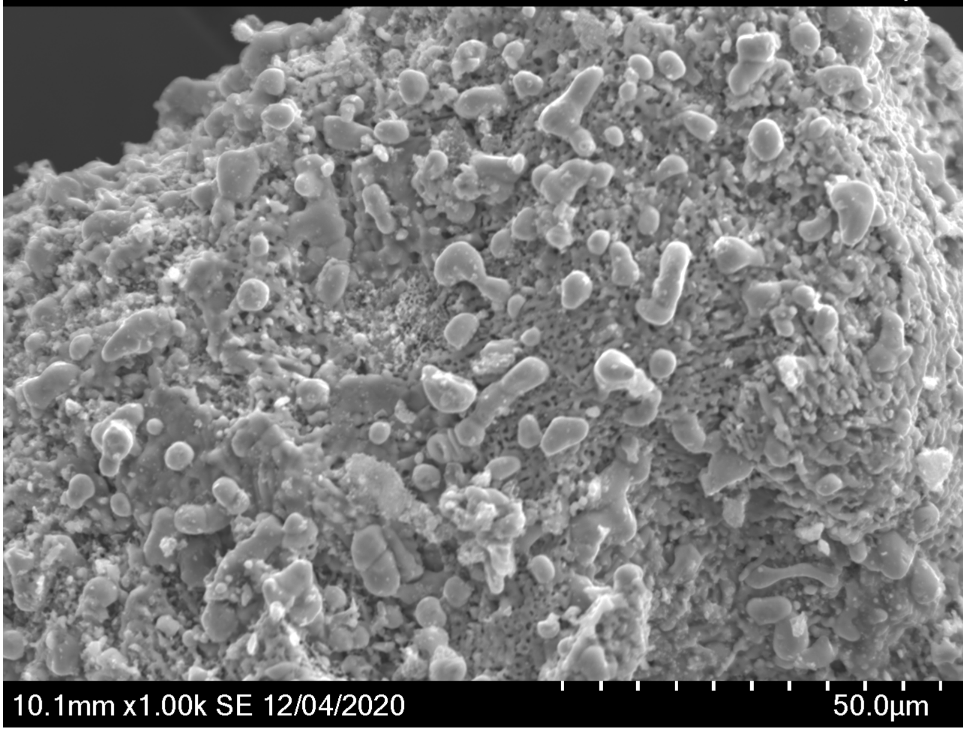


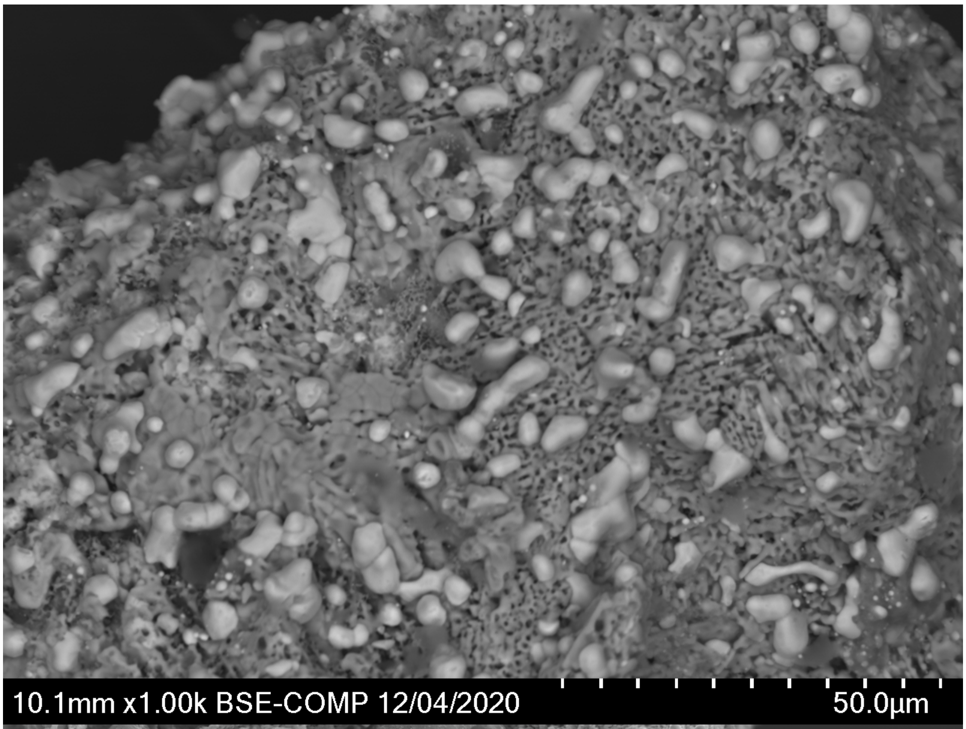


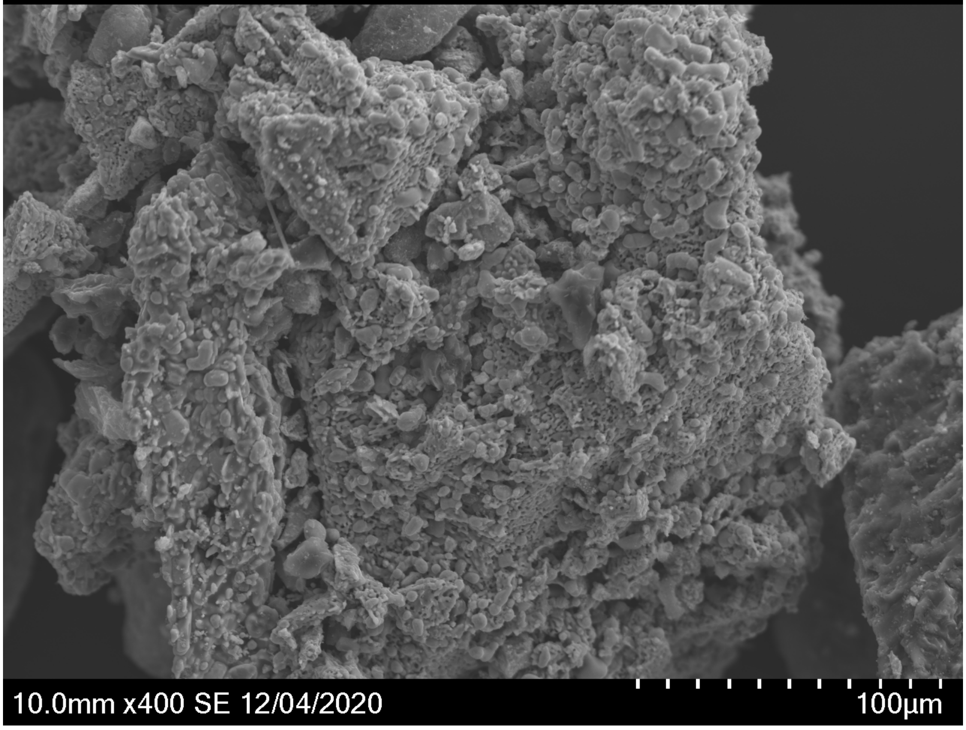

10.1mm x40 SE 12/04/2020
1.00mm

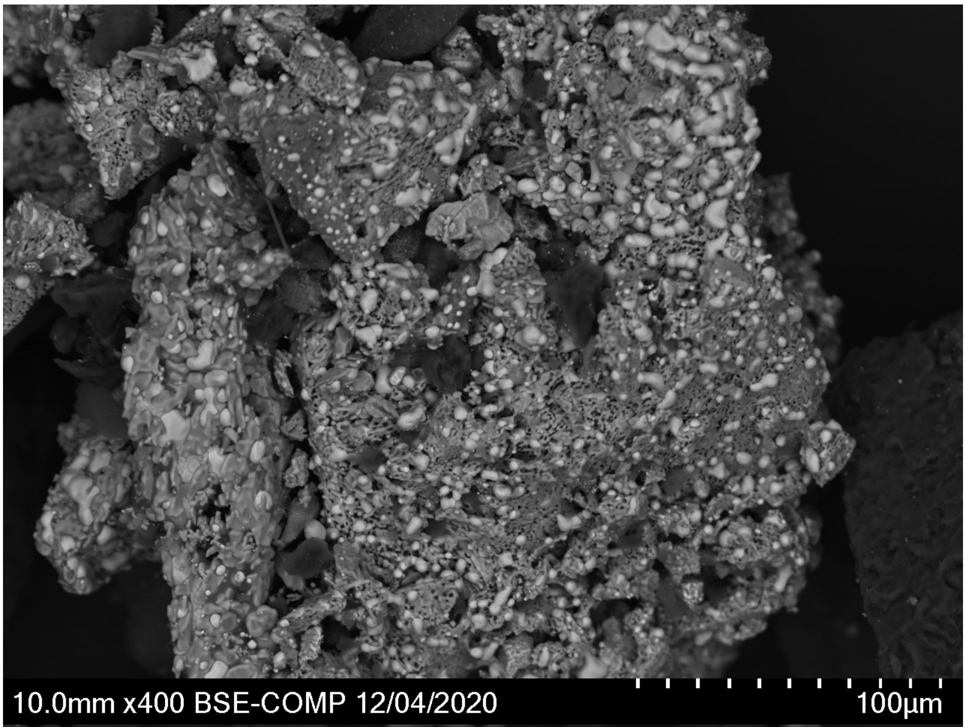

10.1mm x40 BSE-COMP 12/04/2020
1.00mm

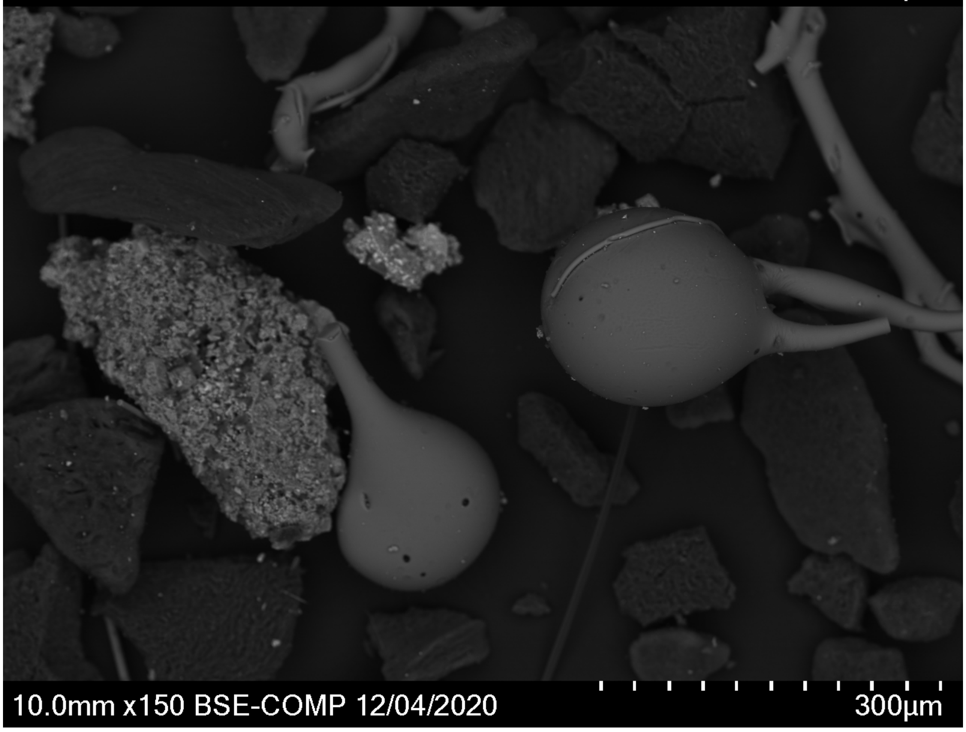

10.1mm x100 BSE-COMP 12/04/2020
500µm


10.1mm x1.00k SE 12/04/2020
50.0µm

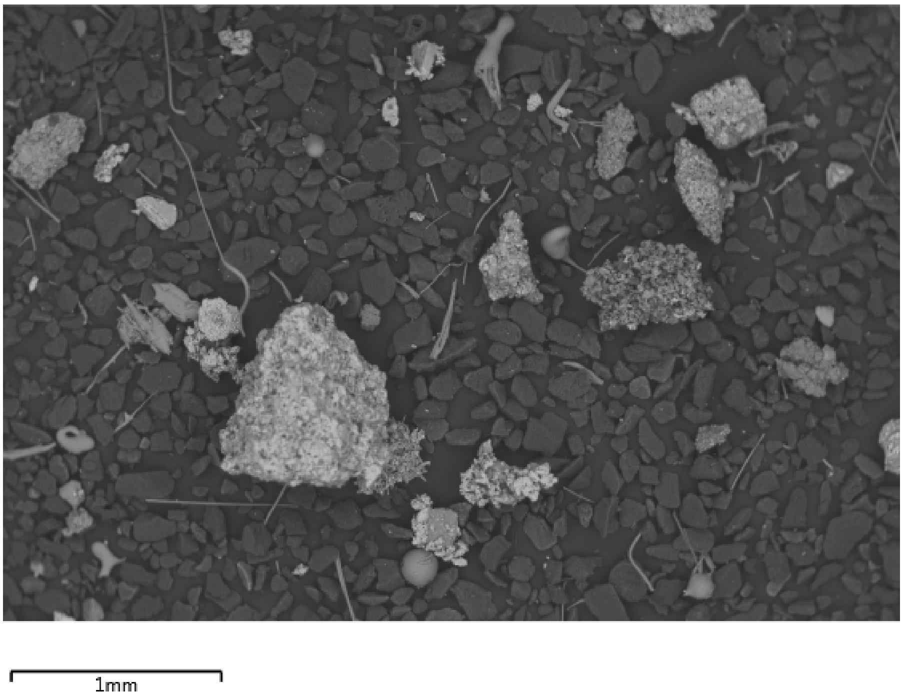

10.1mm x1.00k BSE-COMP 12/04/2020
50.0µm

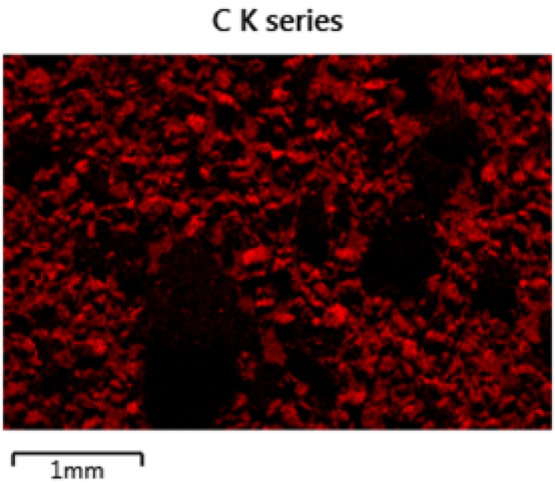

10.0mm x400 SE 12/04/2020
100µm

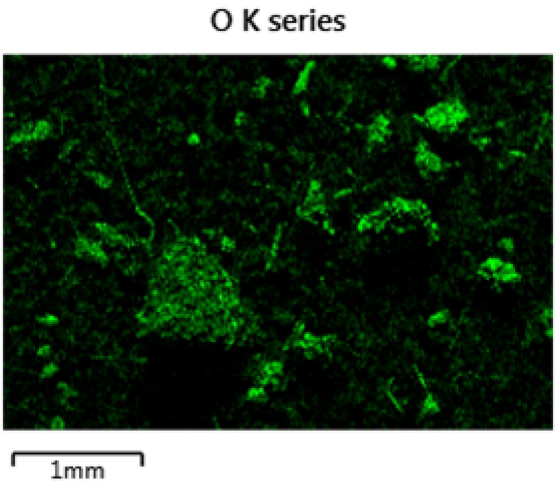

10.0mm x400 BSE-COMP 12/04/2020
100µm

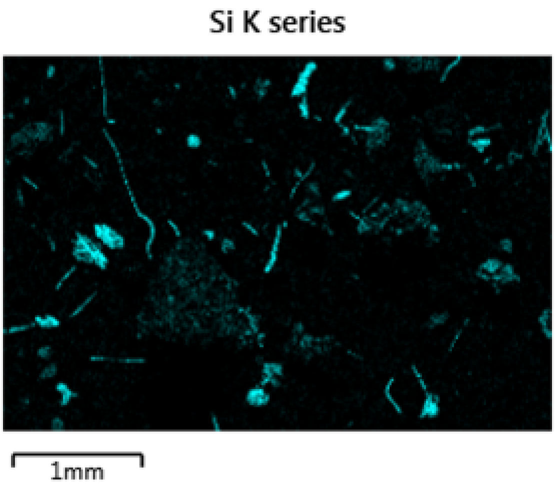

10.0mm x150 BSE-COMP 12/04/2020
300µm

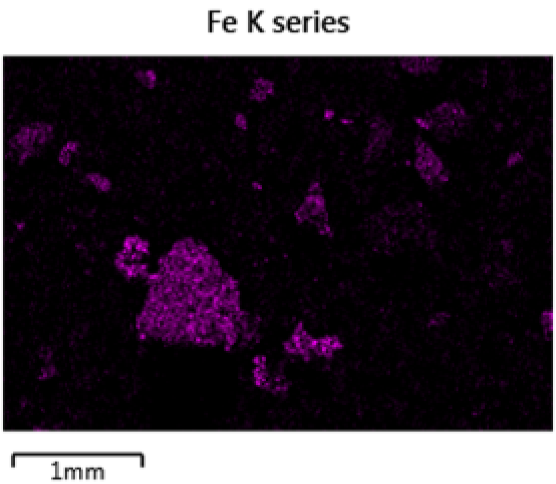
Appendix D: Elemental map – Coal Ash

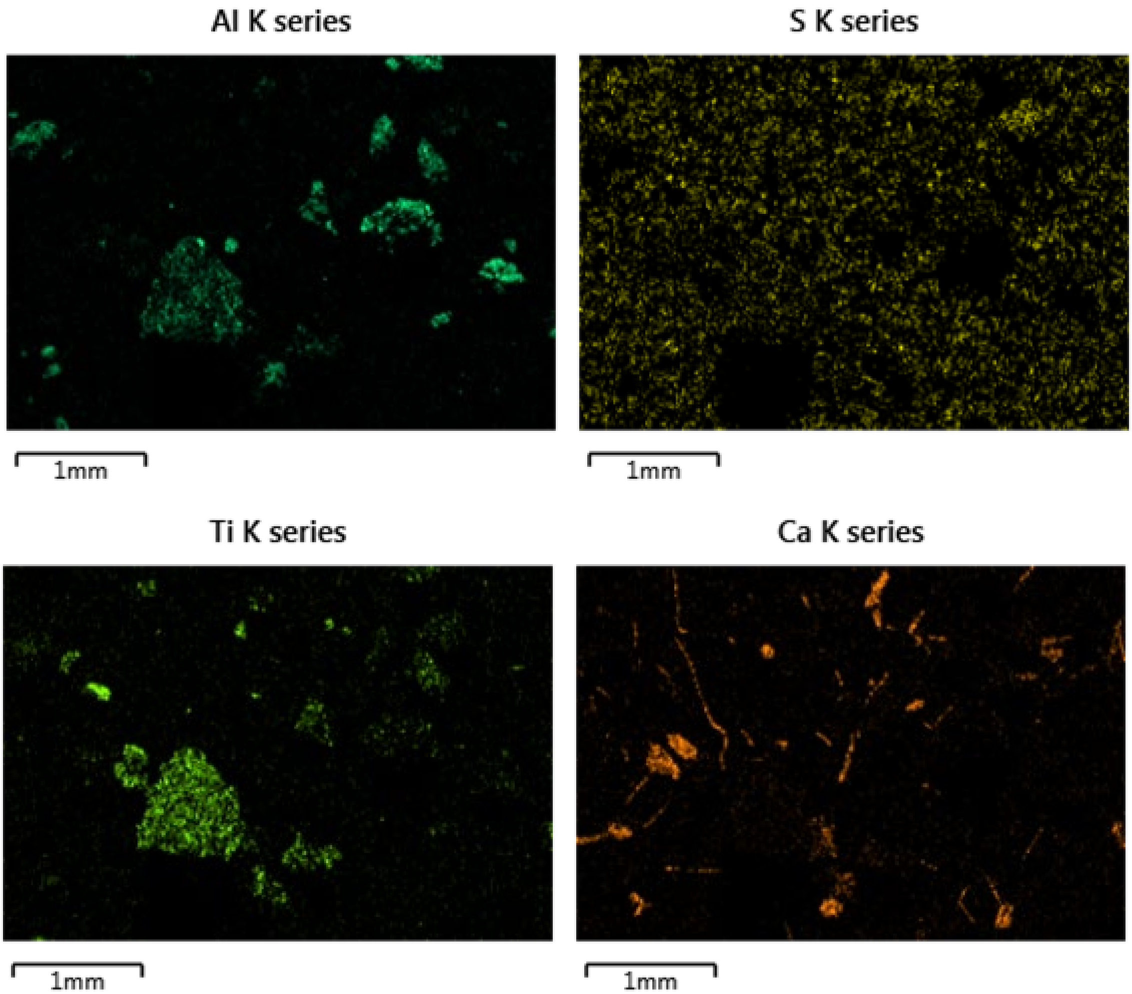




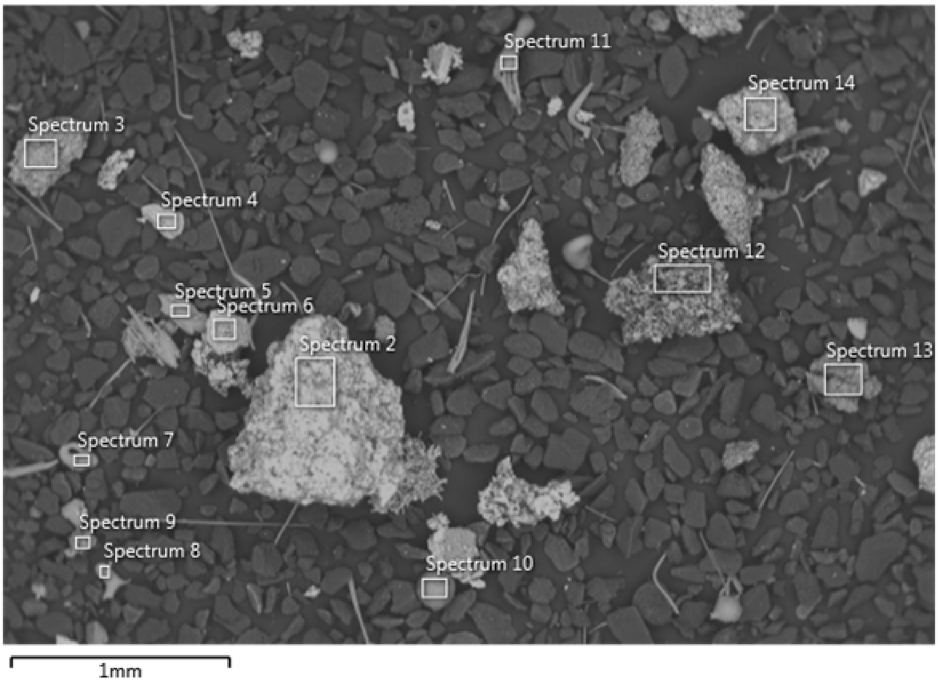


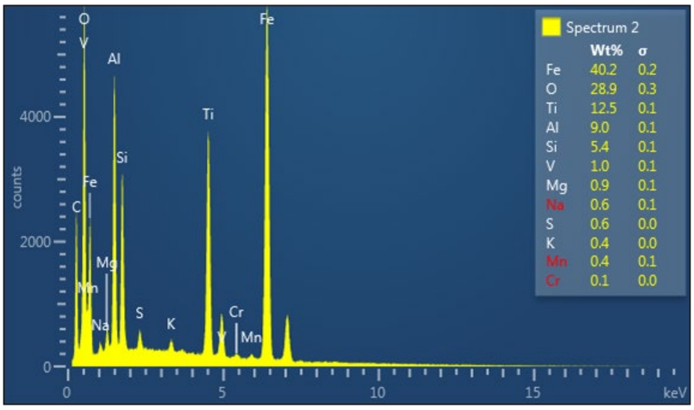


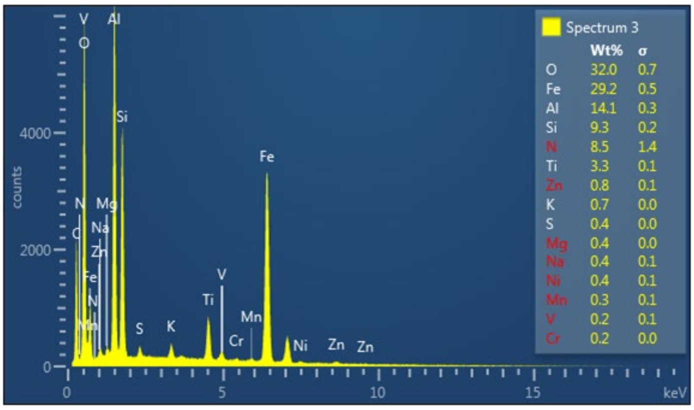


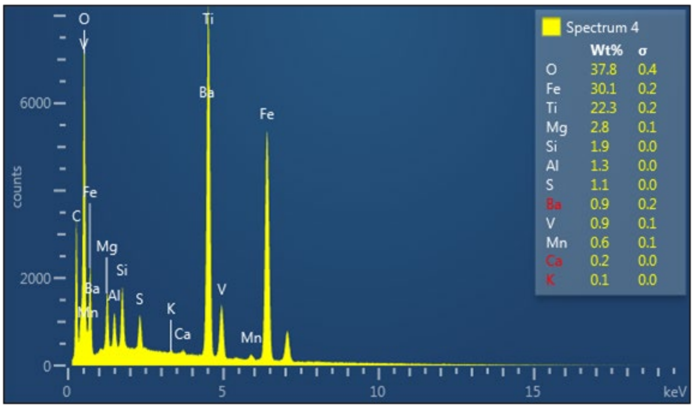

Al K series
S K series
1mm
1mm
Ti K series
Ca K series
1mm
1mm

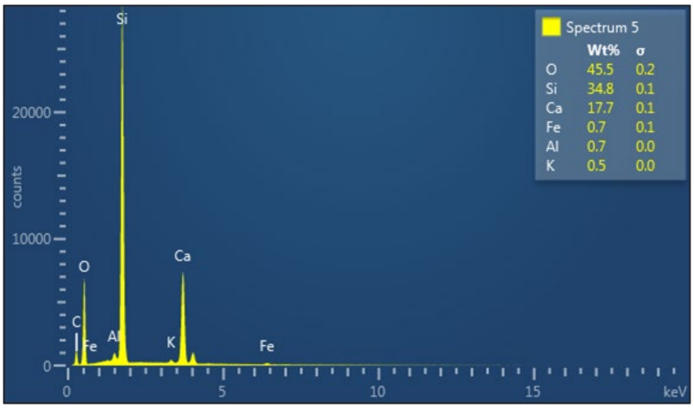
Appendix D: EDS spectra – Coal Ash

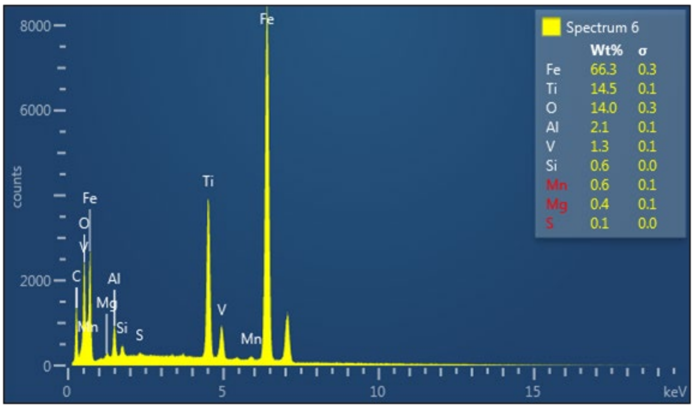


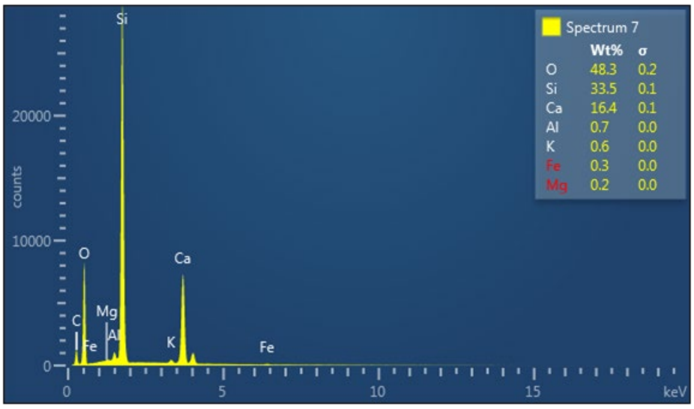


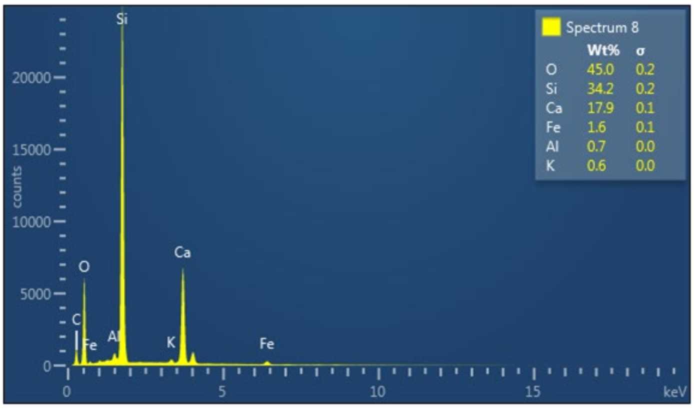


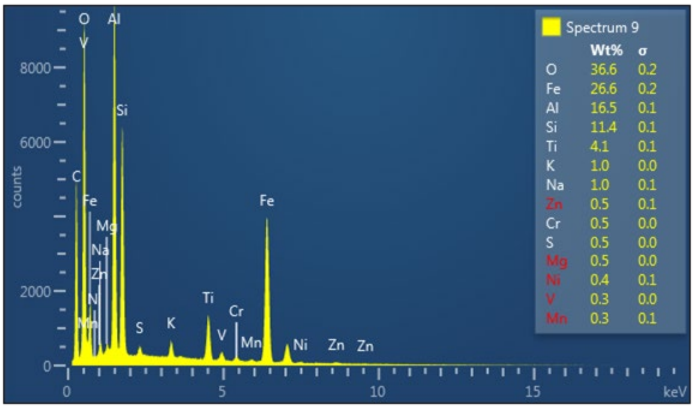


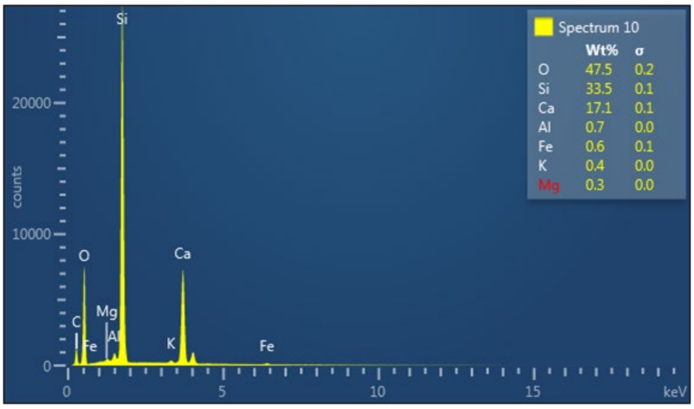


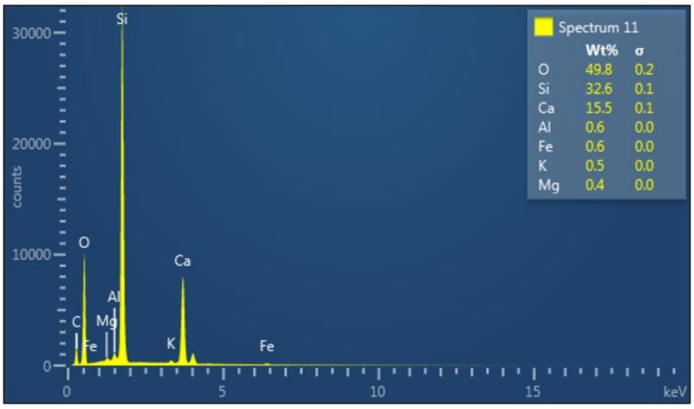

Spectrum 6
Wt% σ
Fe 66.3 0.3
Ti 14.5 0.1
O 14.0 0.3
Al 2.1 0.1
V 1.3 0.1
Si 0.6 0.0
Mn 0.6 0.1
Mg 0.4 0.1
S 0.1 0.0
counts
keV

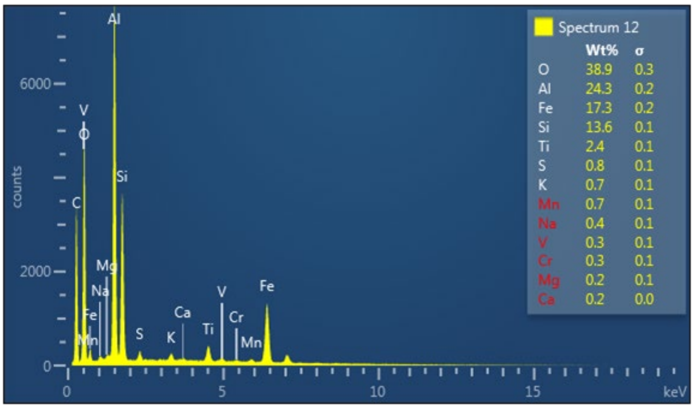

Spectrum 7
Wt% σ
O 48.3 0.2
Si 33.5 0.1
Ca 16.4 0.1
Al 0.7 0.0
K 0.6 0.0
Fe 0.3 0.0
Mg 0.2 0.0
counts
keV

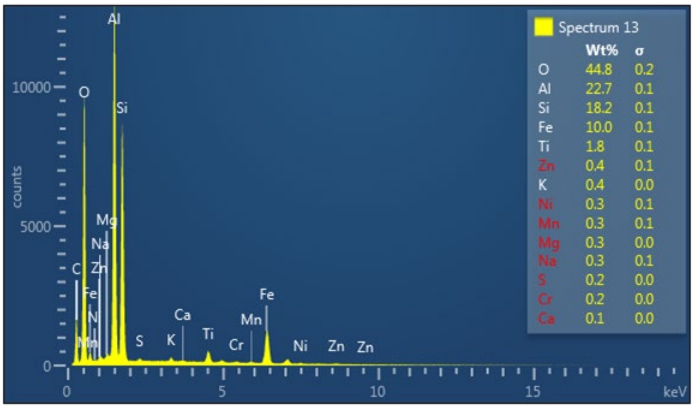

Spectrum 8
Wt% σ
O 45.0 0.2
Si 34.2 0.2
Ca 17.9 0.1
Fe 1.6 0.1
Al 0.7 0.0
K 0.6 0.0
counts
keV

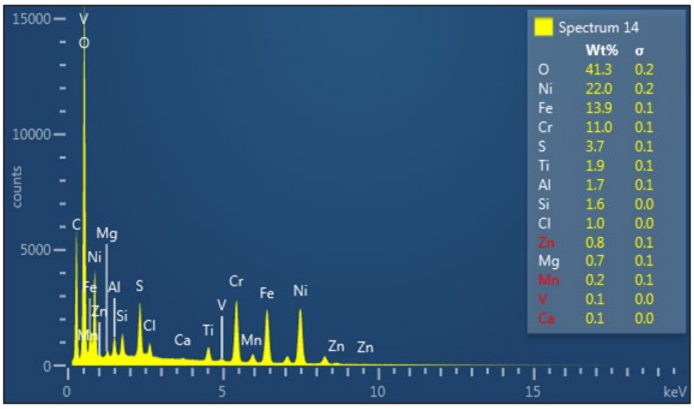

Spectrum 9
Wt% σ
O 36.6 0.2
Fe 26.6 0.2
Al 16.5 0.1
Si 11.4 0.1
Ti 4.1 0.1
K 1.0 0.0
Na 1.0 0.1
Zn 0.5 0.1
Cr 0.5 0.0
S 0.5 0.0
Mg 0.5 0.0
Ni 0.4 0.1
V 0.3 0.0
Mn 0.3 0.1
counts
keV


Spectrum 10
Wt% σ
O 47.5 0.2
Si 33.5 0.1
Ca 17.1 0.1
Al 0.7 0.0
Fe 0.6 0.1
K 0.4 0.0
Mg 0.3 0.0
counts
keV

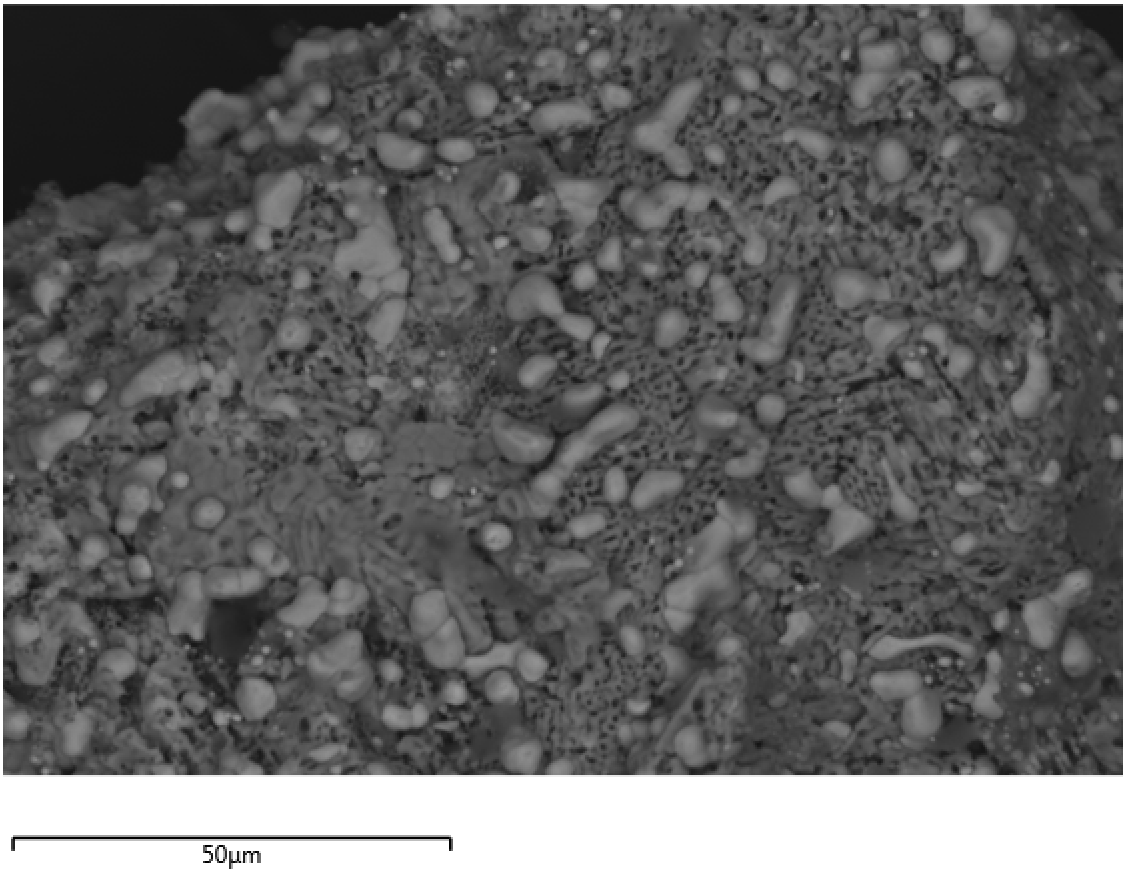

Spectrum 11
Wt% σ
O 49.8 0.2
Si 32.6 0.1
Ca 15.5 0.1
Al 0.6 0.0
Fe 0.6 0.0
K 0.5 0.0
Mg 0.4 0.0
counts
keV

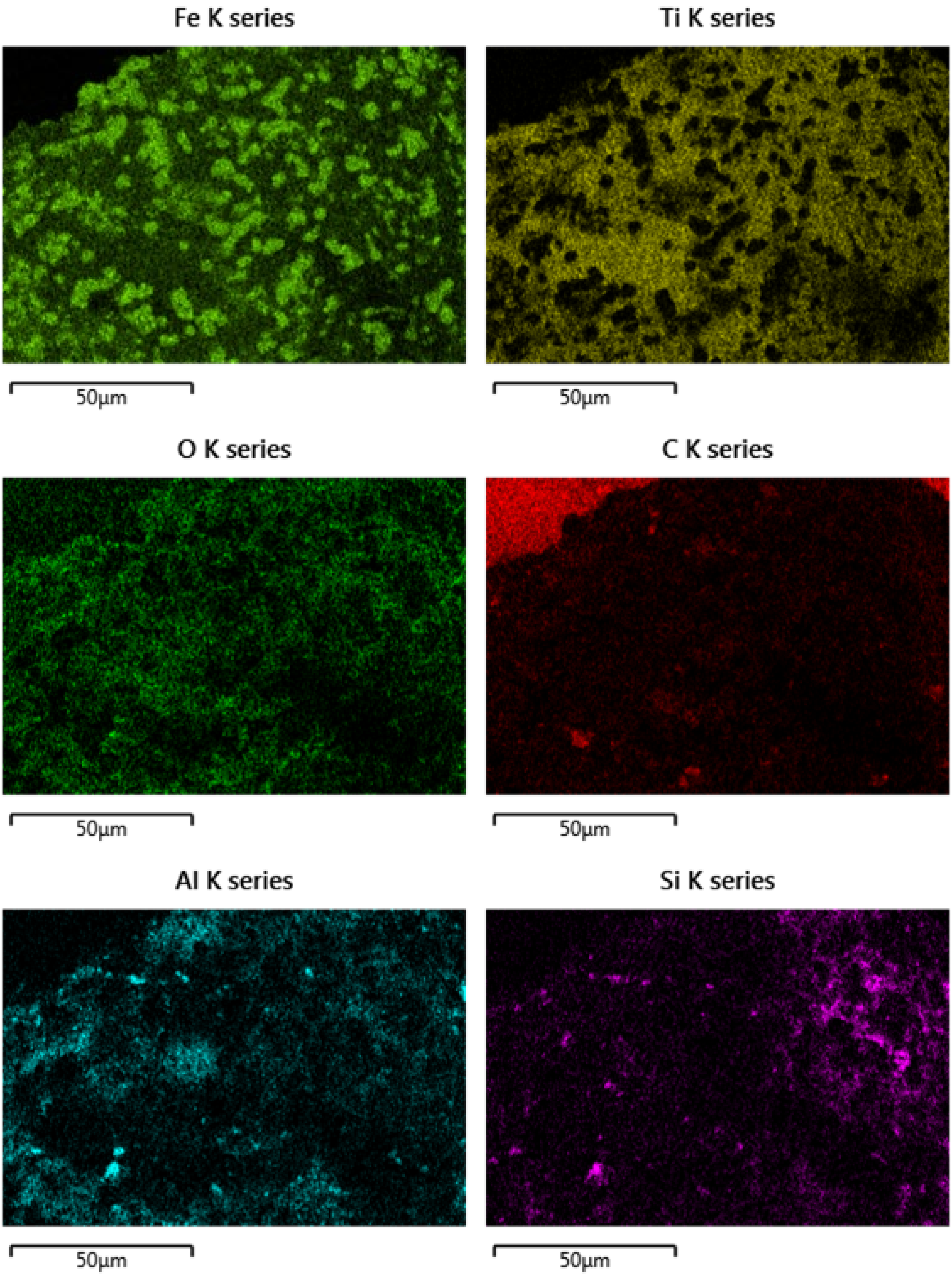

Spectrum 12
Wt% σ
O 38.9 0.3
Al 24.3 0.2
Fe 17.3 0.2
Si 13.6 0.1
Ti 2.4 0.1
S 0.8 0.1
K 0.7 0.1
Mn 0.7 0.1
Na 0.4 0.1
V 0.3 0.1
Cr 0.3 0.1
Mg 0.2 0.1
Ca 0.2 0.0
counts
keV

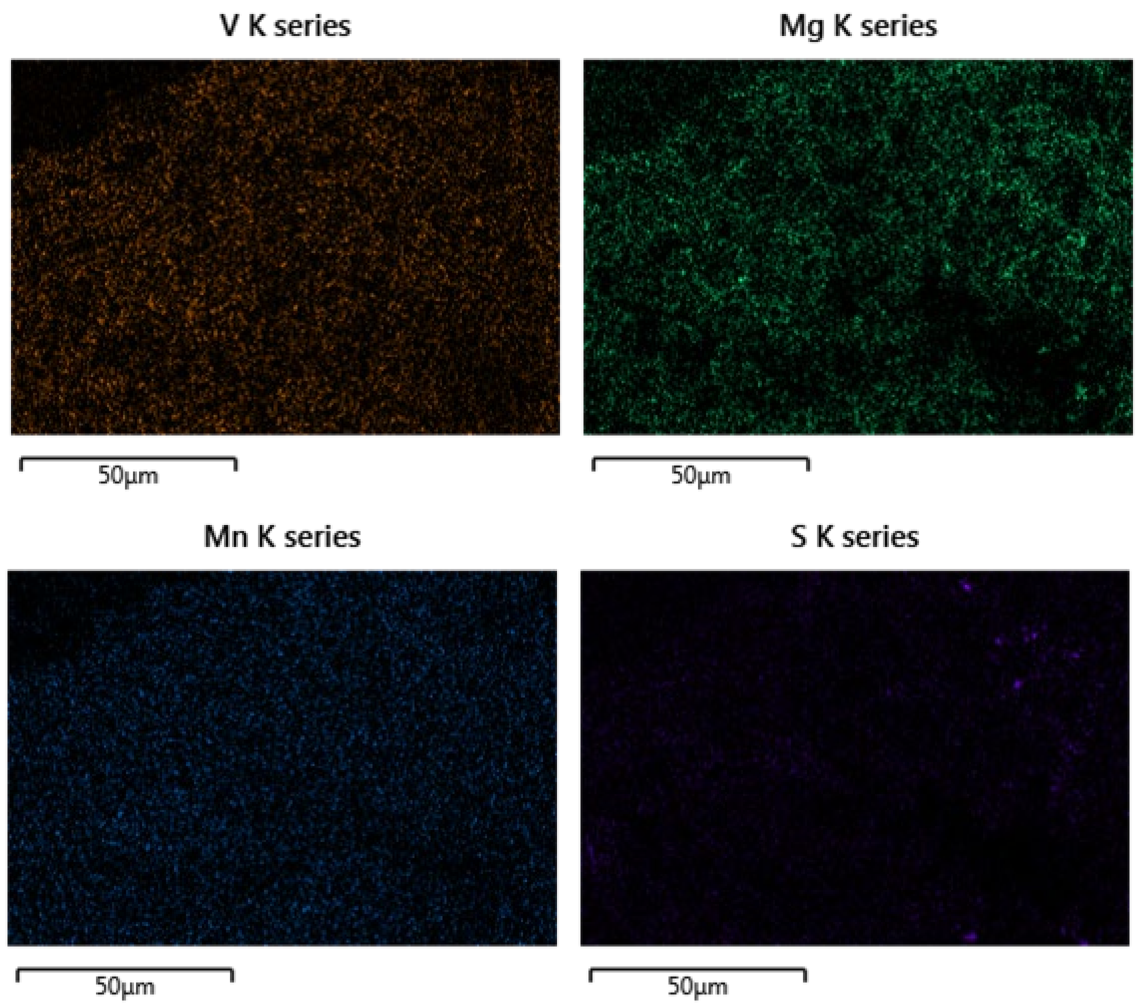

Spectrum 13
Wt% σ
O 44.8 0.2
Al 22.7 0.1
Si 18.2 0.1
Fe 10.0 0.1
Ti 1.8 0.1
Zn 0.4 0.1
K 0.4 0.0
Ni 0.3 0.1
Mn 0.3 0.1
Mg 0.3 0.0
Na 0.3 0.1
S 0.2 0.0
Cr 0.2 0.0
Ca 0.1 0.0
counts
keV



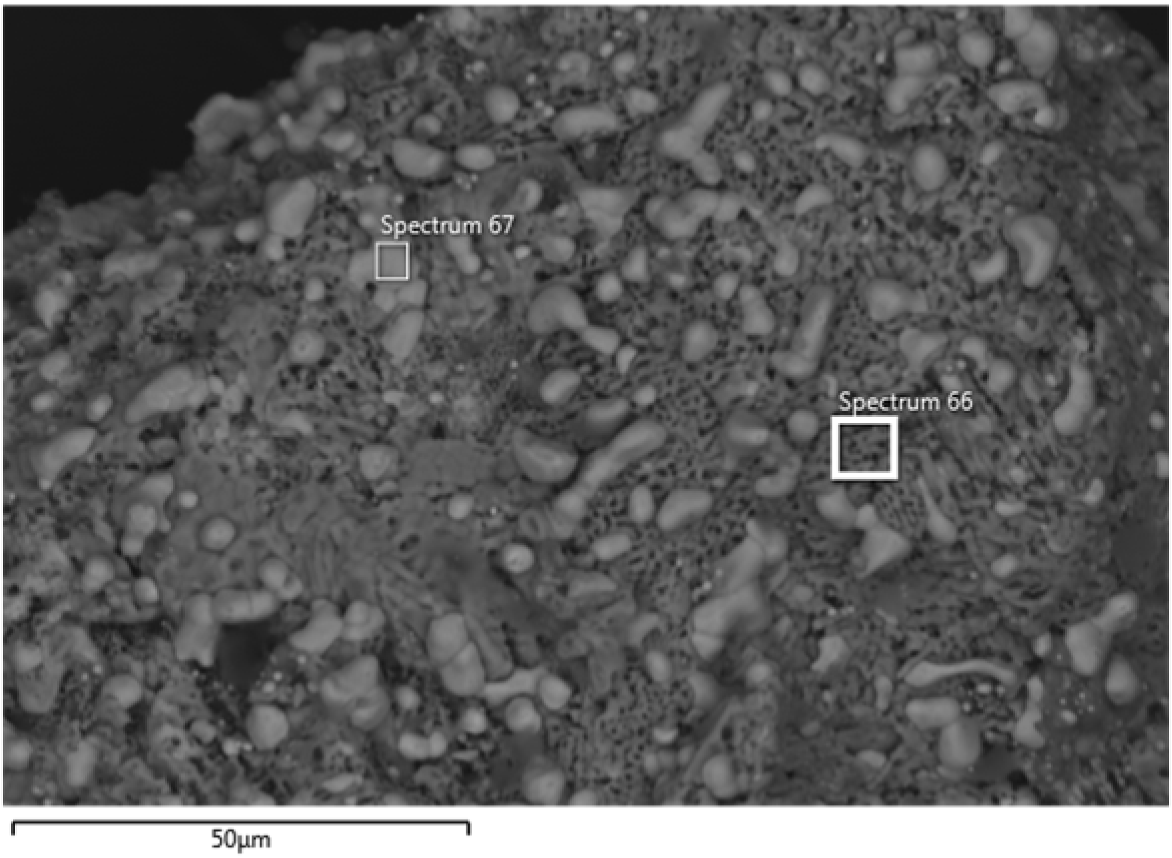
Appendix D: Elemental map Area 2 – Coal Ash

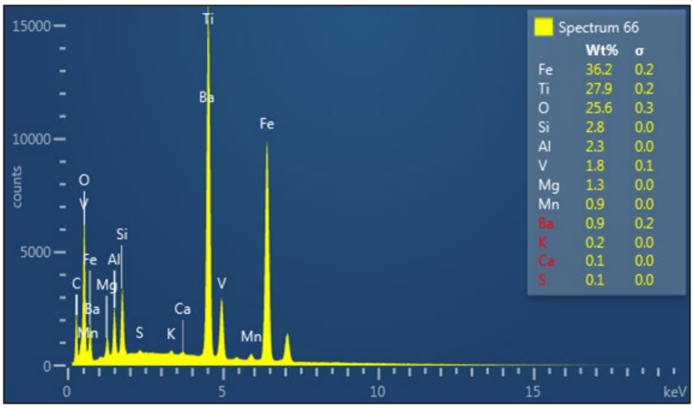


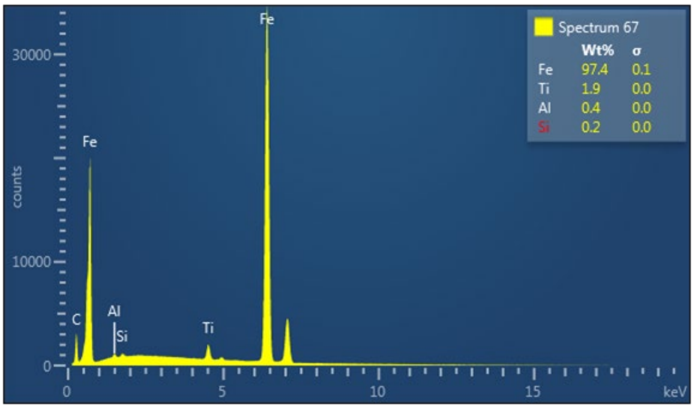

Fe K series
Ti K series
50µm
50µm
O K series
C K series
50µm
50µm
Al K series
Si K series
50µm
50µm


V K series
50µm
Mg K series
50µm
Mn K series
50µm
S K series
50µm

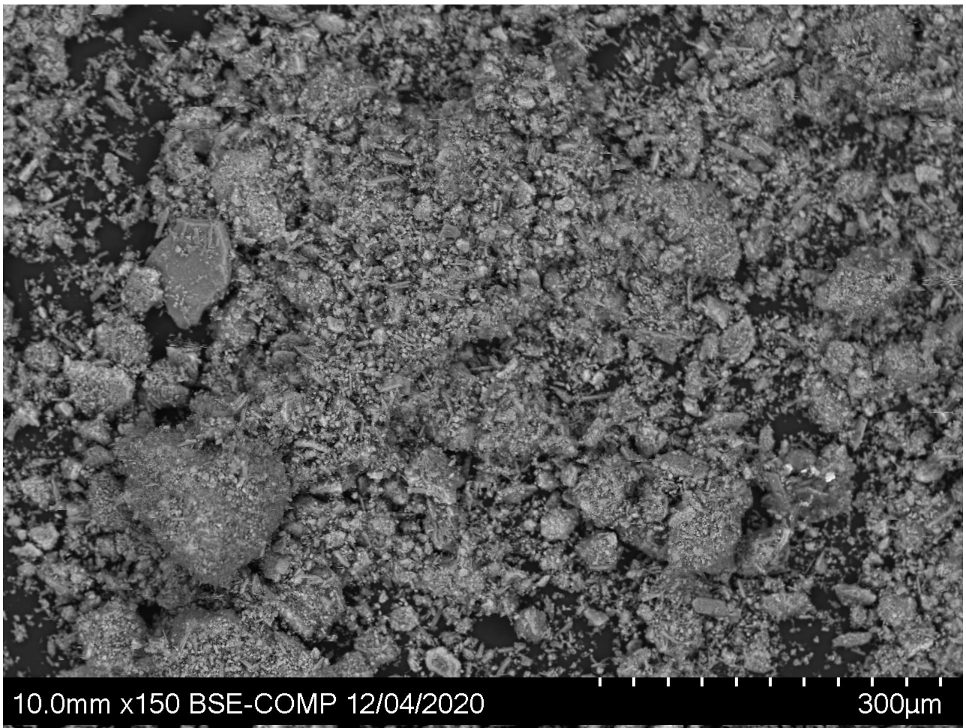
Appendix D: EDS spectra Area 2 – Coal Ash

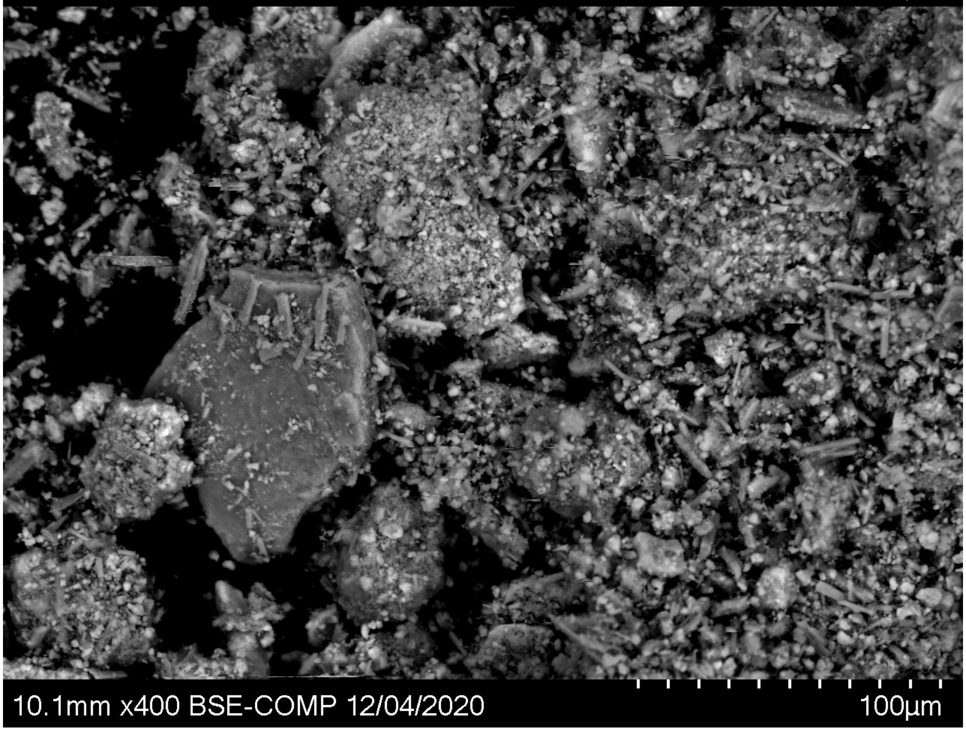


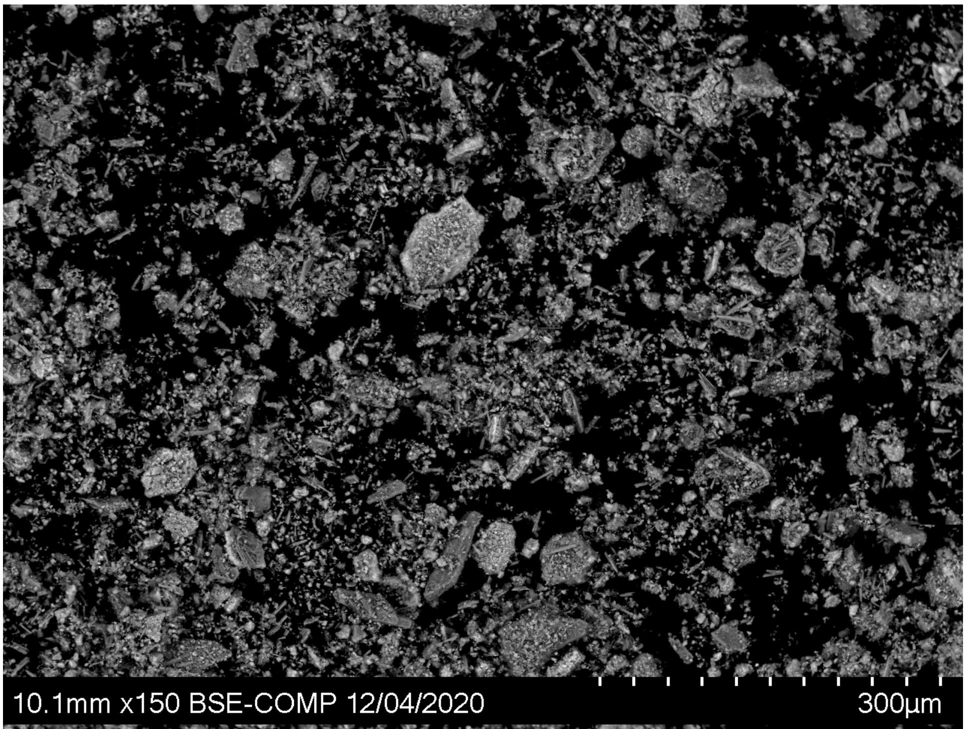


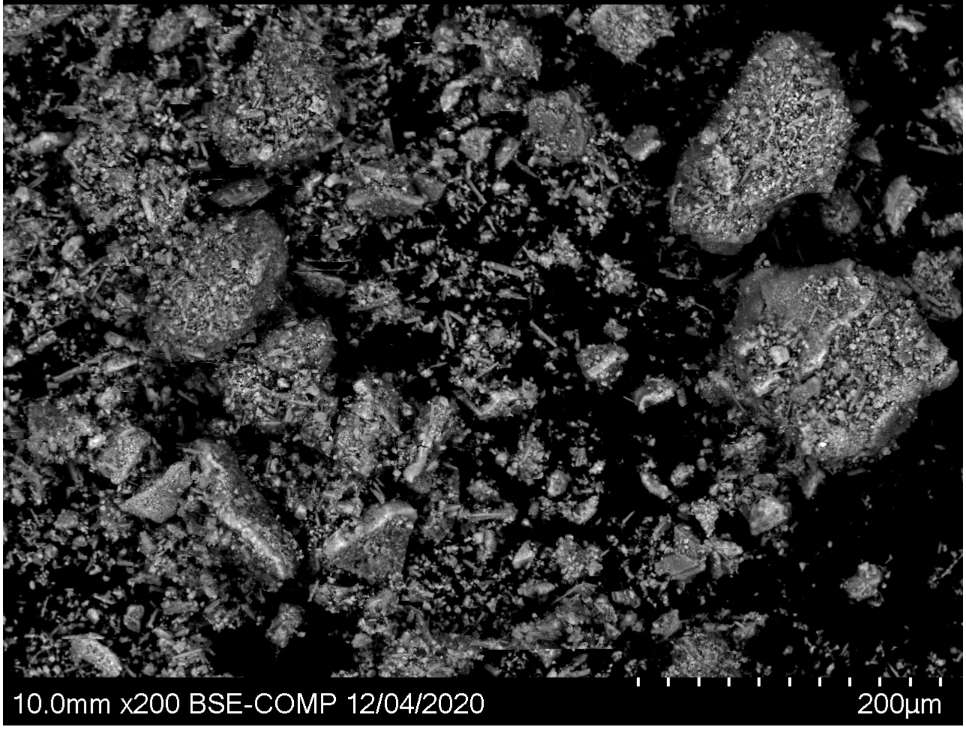


Appendix D: SEM Images – Metal Hydroxide Precipitation #1 Residue

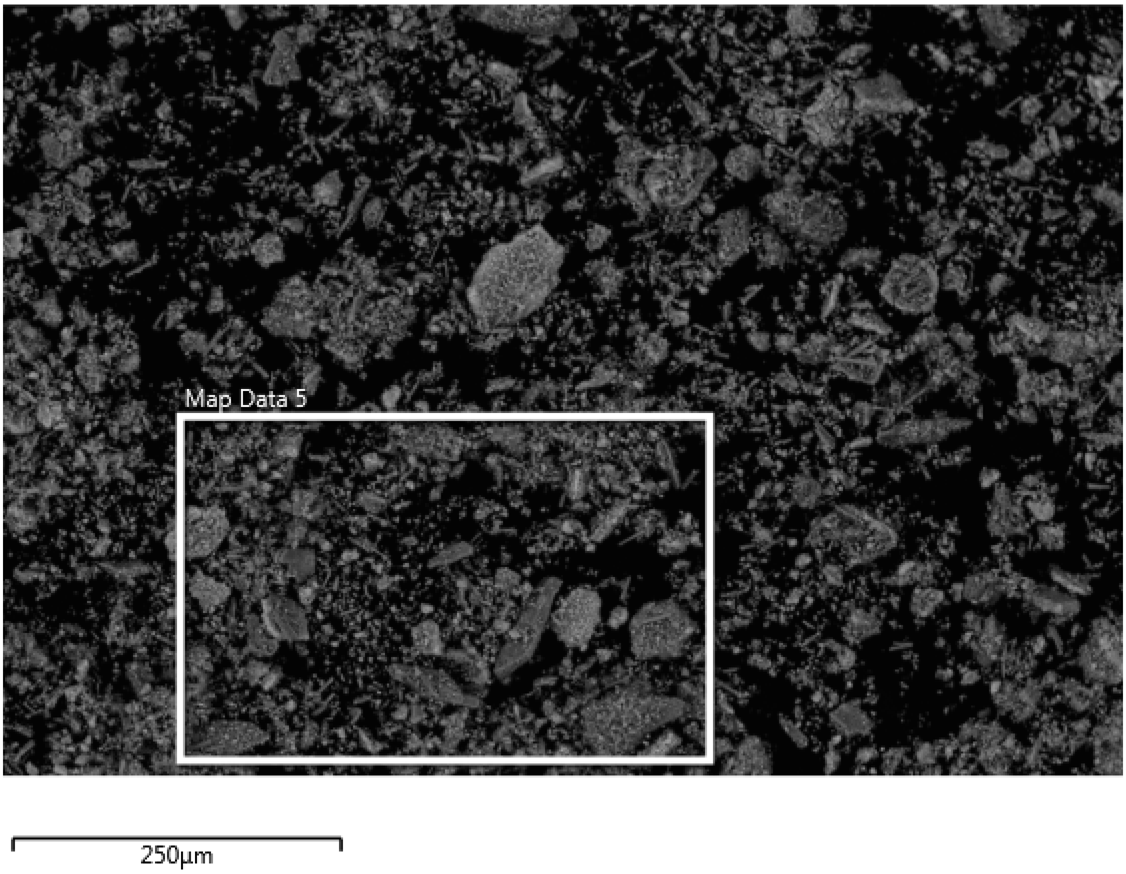


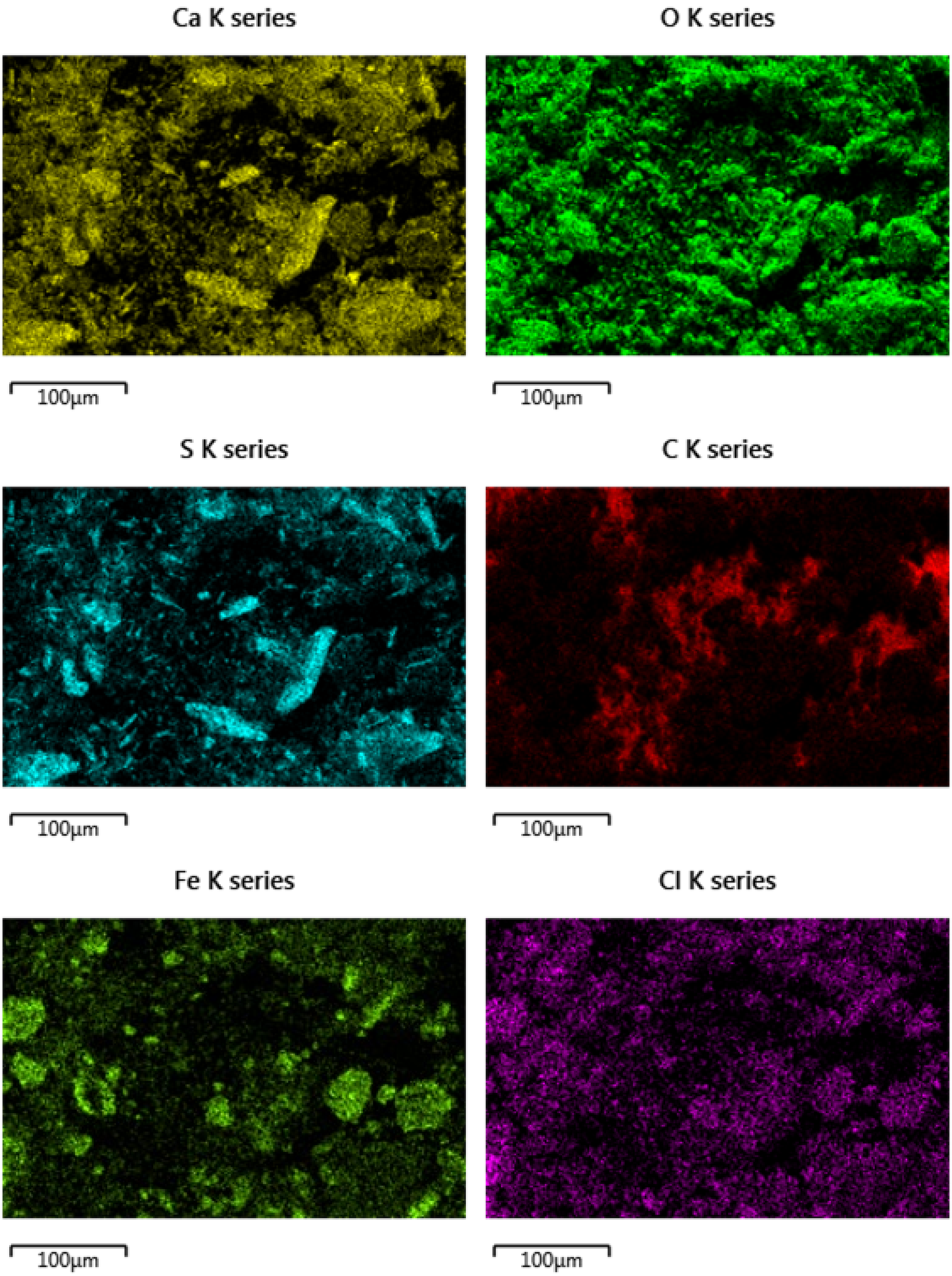


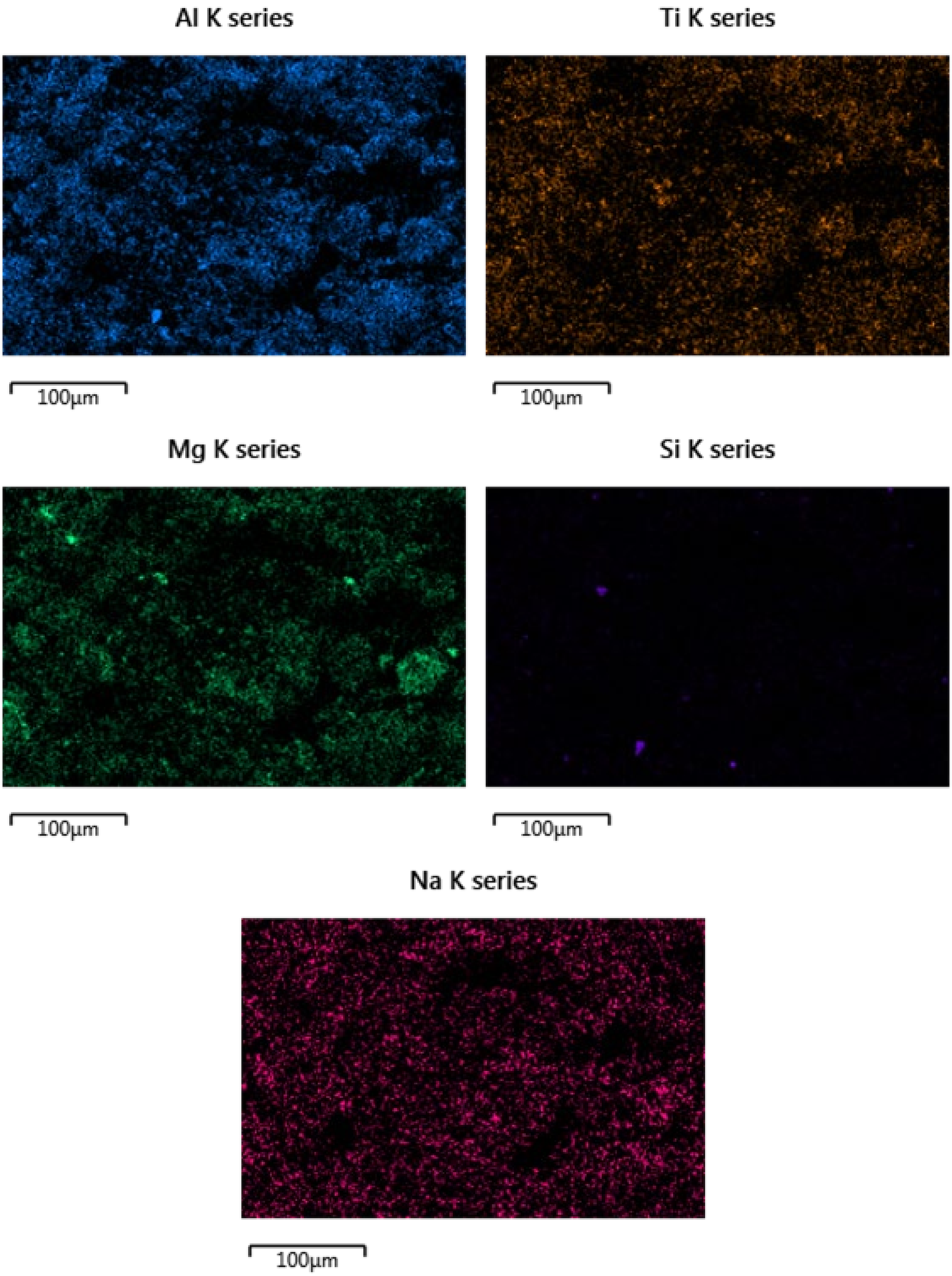

10.1mm x150 BSE-COMP 12/04/2020
300µm


10.0mm x200 BSE-COMP 12/04/2020
200µm

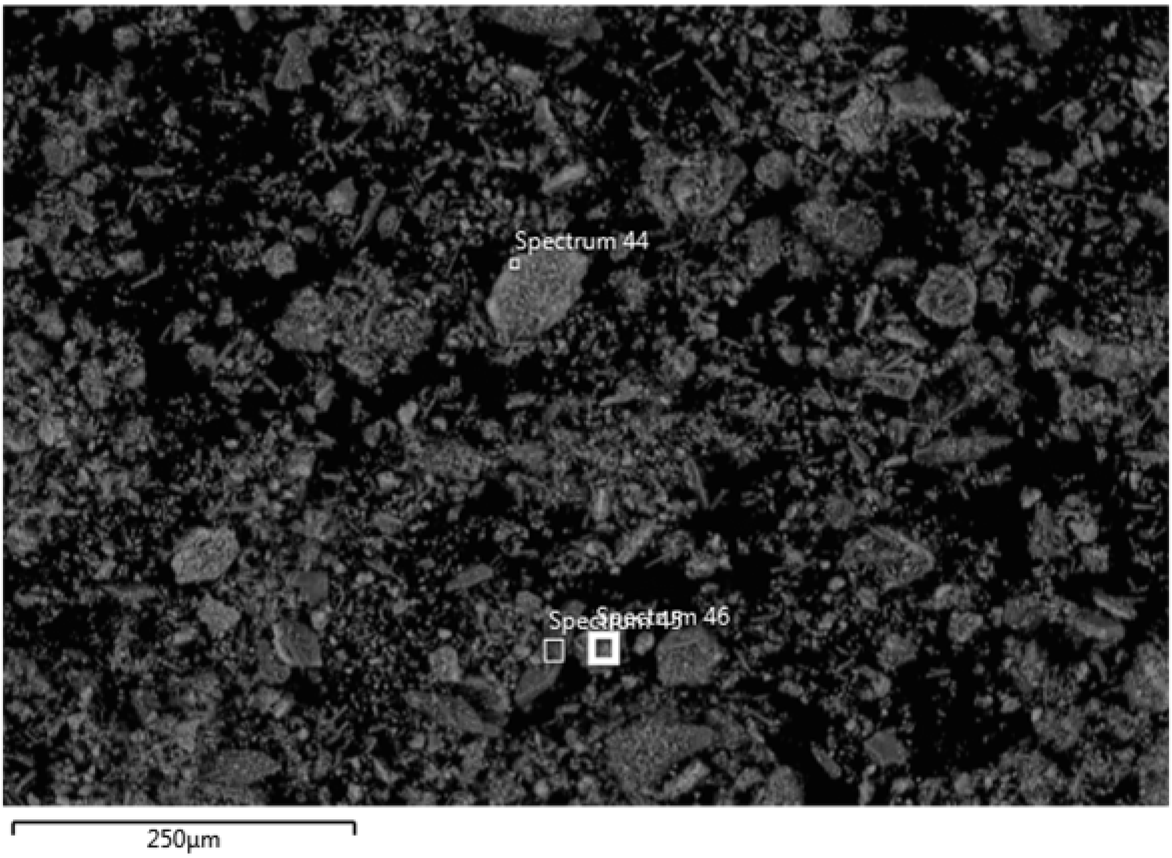
Appendix D: Elemental map Area 1 – Metal Hydroxide Precipitation #1 Residue

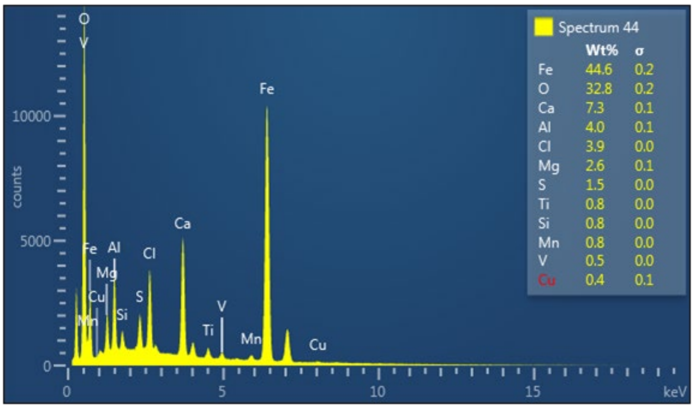


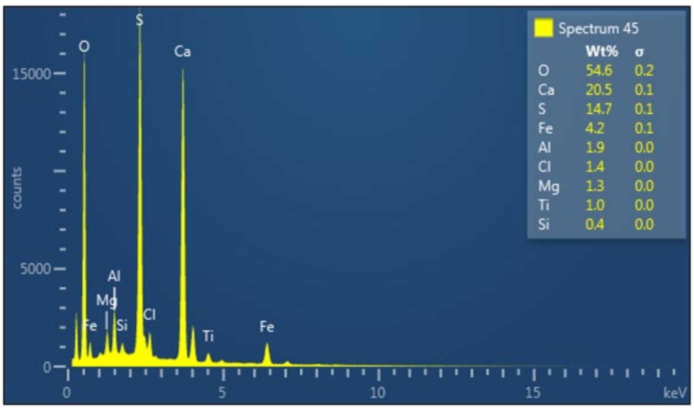

Ca K series
O K series
100µm
100µm
S K series
C K series
100µm
100µm
Fe K series
Cl K series
100µm
100µm

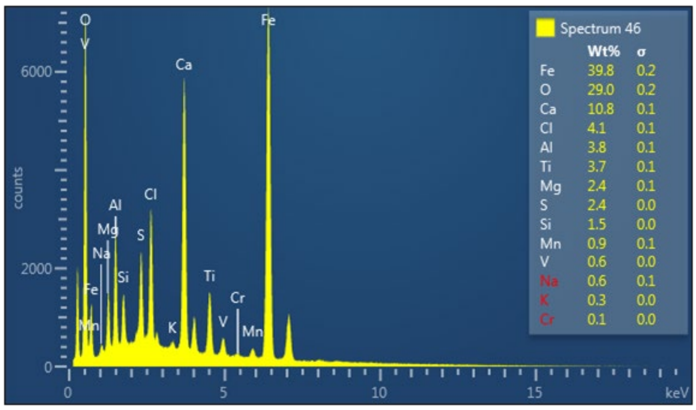

Al K series
100µm
Ti K series
100µm
Mg K series
100µm
Si K series
100µm
Na K series
100µm

Appendix D: EDS spectra Area 1 – Metal Hydroxide Precipitation #1 Residue

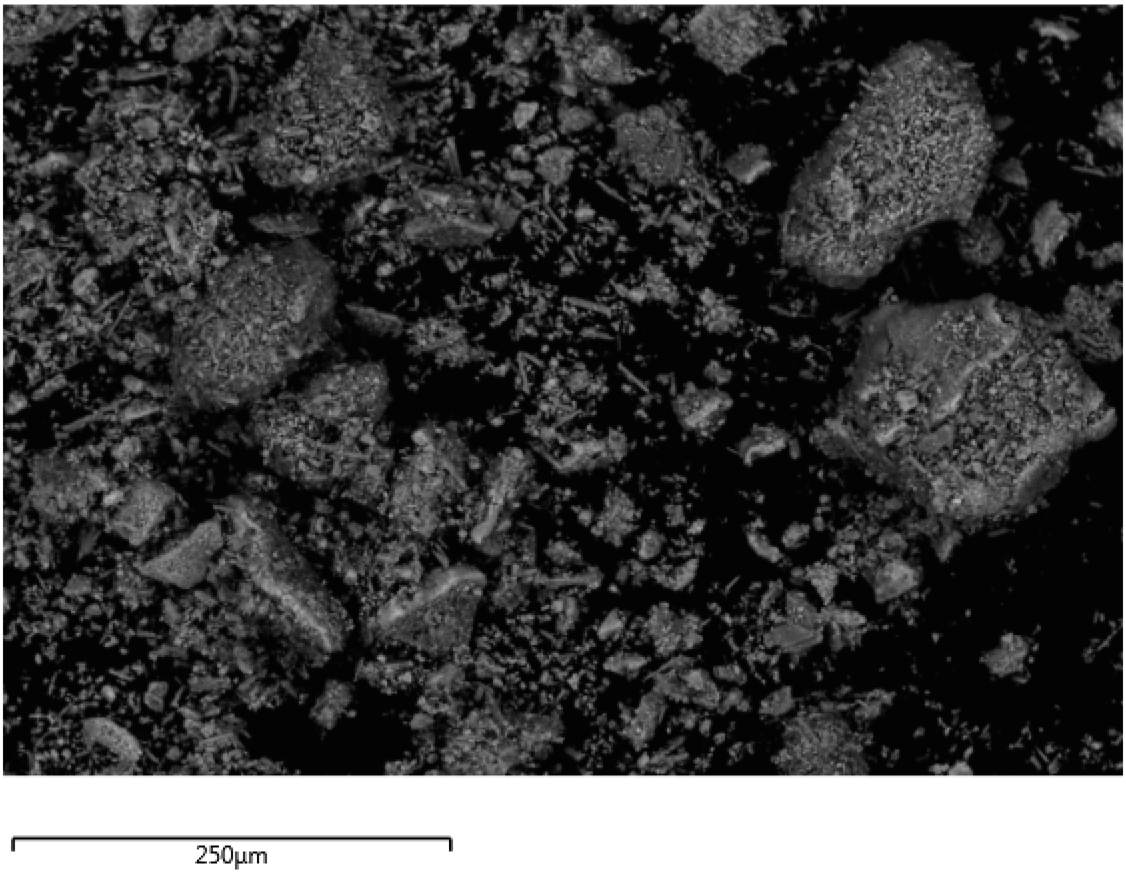


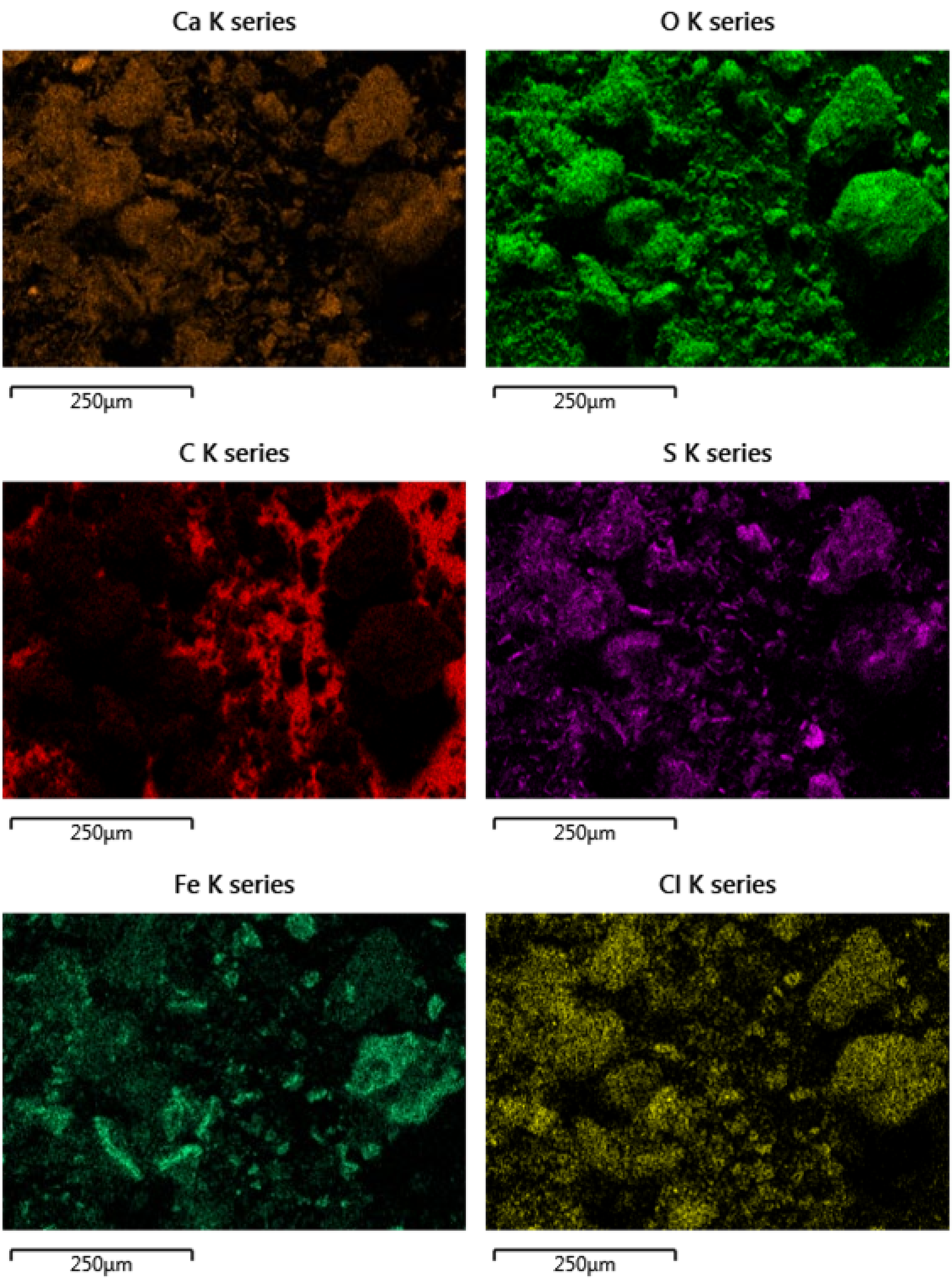


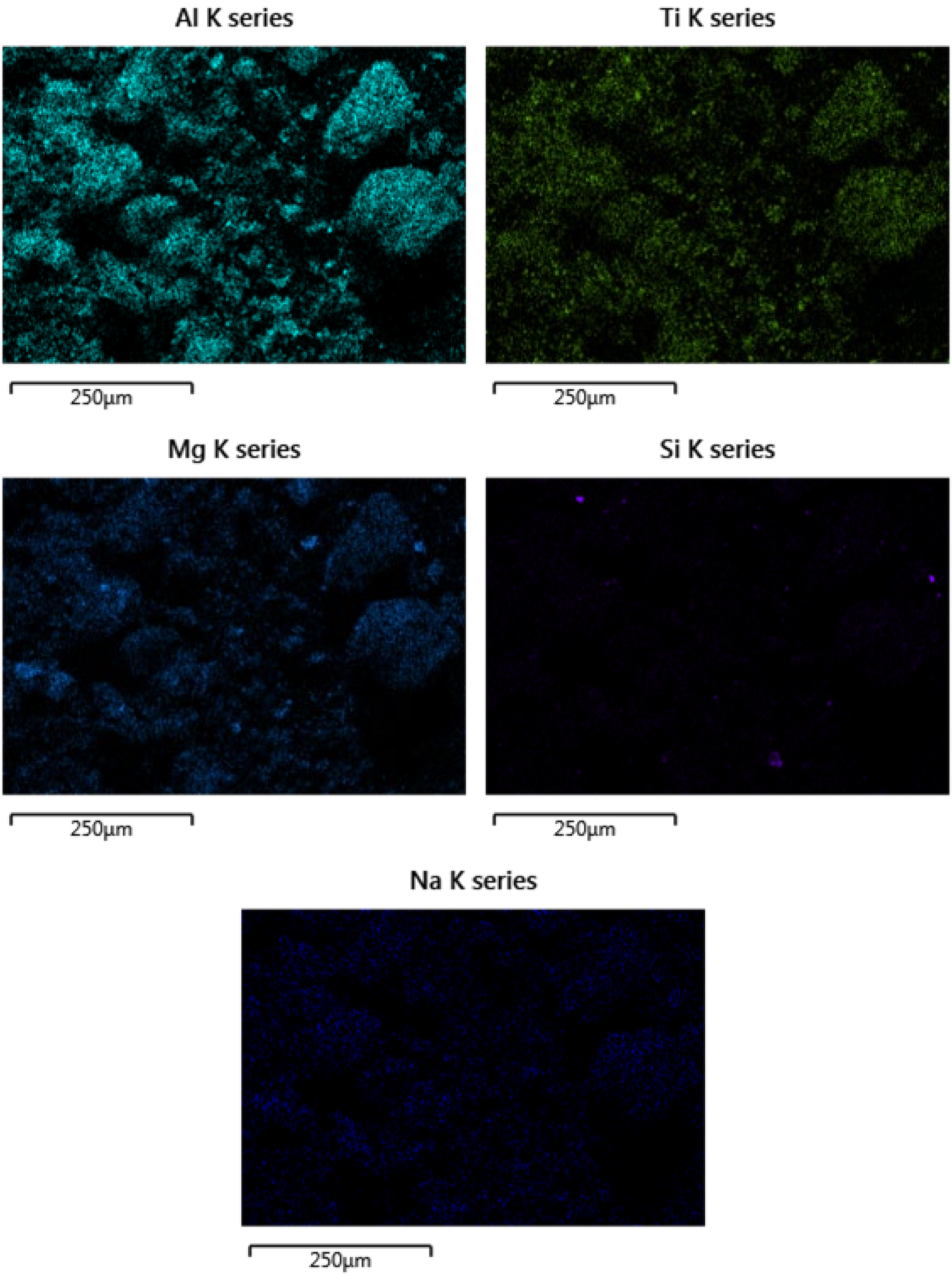




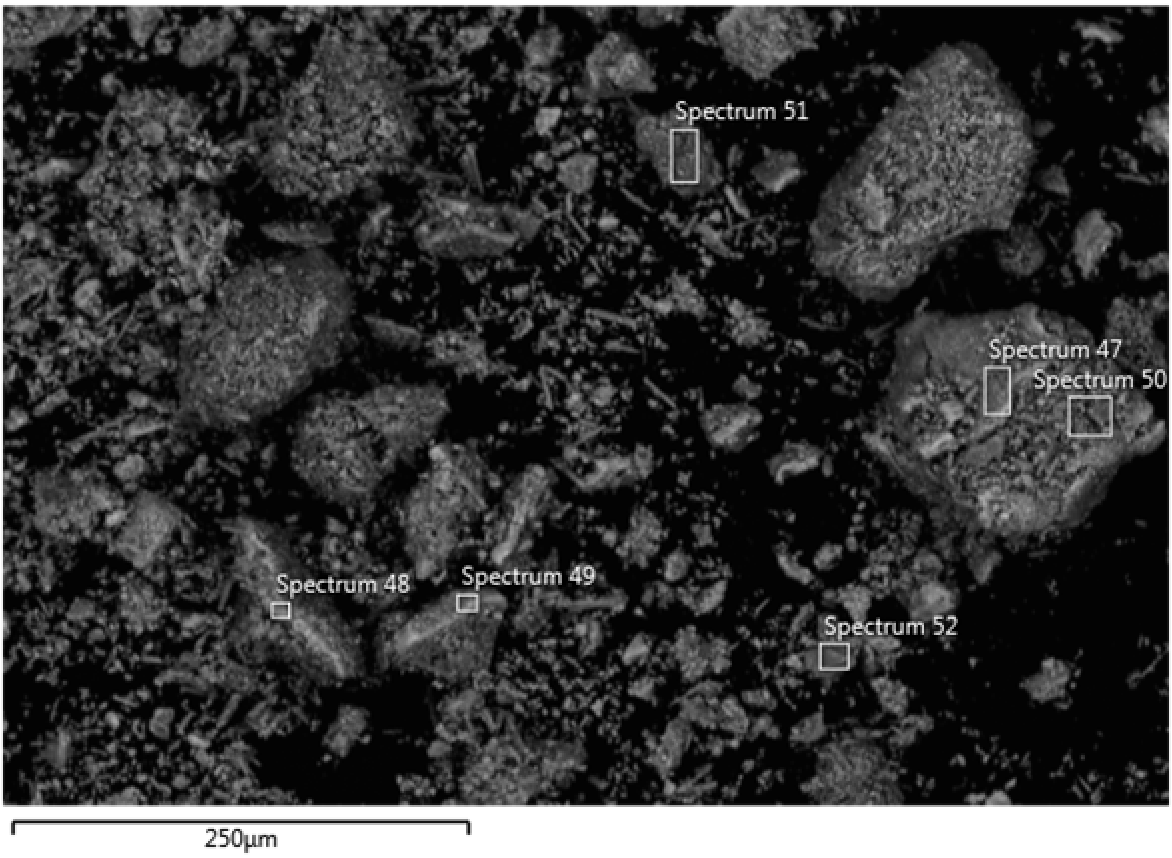
Appendix D: Elemental map Area 2 - Metal Hydroxide Precipitation #1 Residue

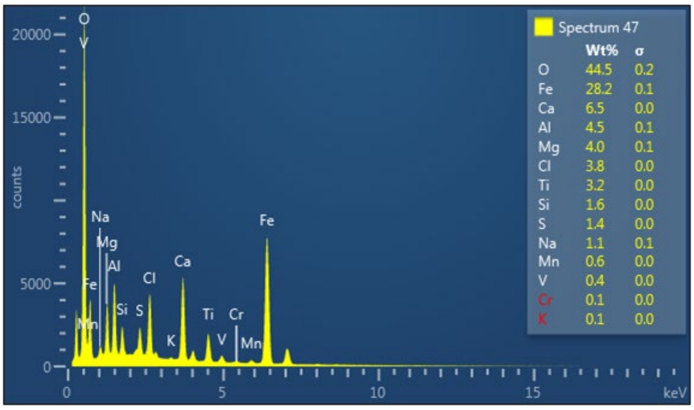


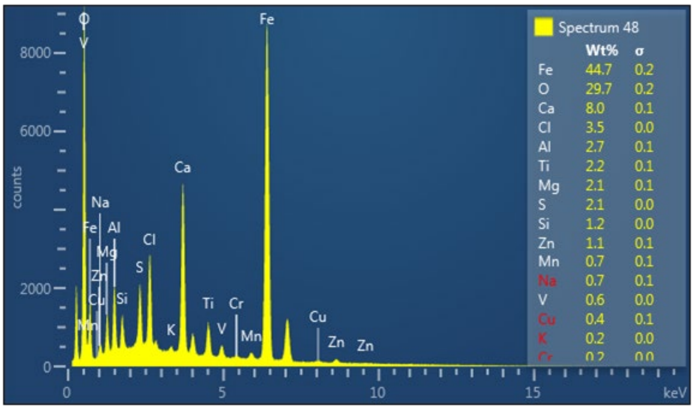

Ca K series
O K series
250µm
250µm
C K series
S K series
250µm
250µm
Fe K series
Cl K series
250µm
250µm

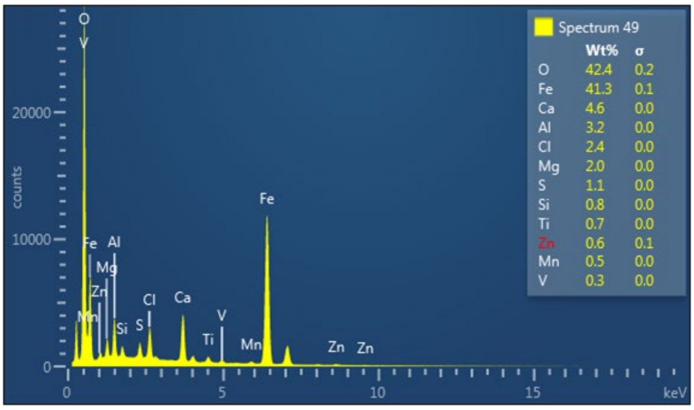

Al K series
250µm
Ti K series
250µm
Mg K series
250µm
Si K series
250µm
Na K series
250µm

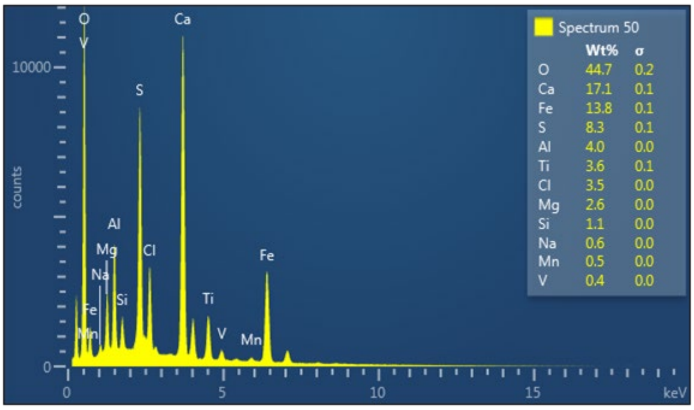
Appendix D: EDS spectra Area 2 – Metal Hydroxide Precipitation #1 Residue

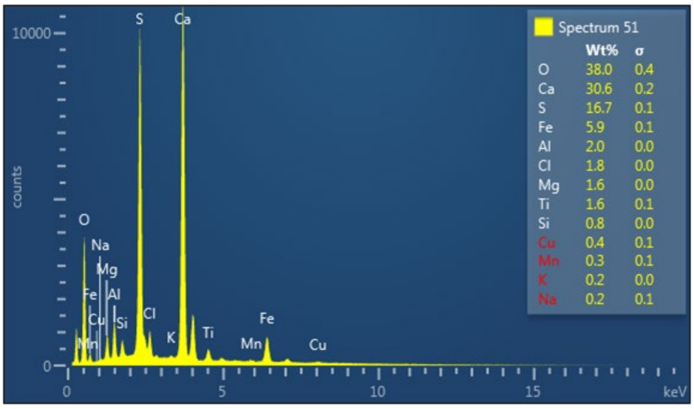


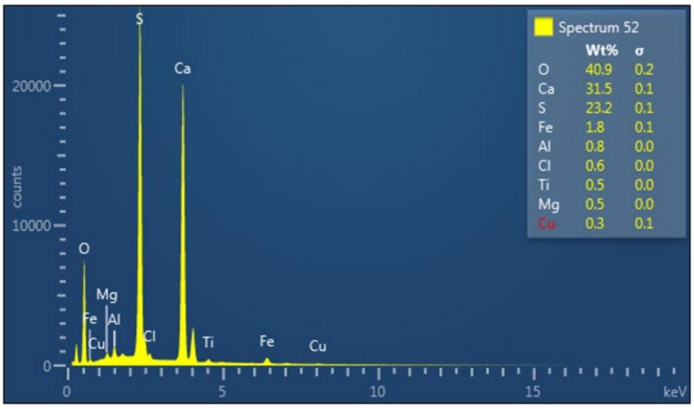









Spectrum 51
Wt% σ
O 38.0 0.4
Ca 30.6 0.2
S 16.7 0.1
Fe 5.9 0.1
Al 2.0 0.0
Cl 1.8 0.0
Mg 1.6 0.0
Ti 1.6 0.1
Si 0.8 0.0
Cu 0.4 0.1
Mn 0.3 0.1
K 0.2 0.0
Na 0.2 0.1
counts
keV


Spectrum 52
Wt% σ
O 40.9 0.2
Ca 31.5 0.1
S 23.2 0.1
Fe 1.8 0.1
Al 0.8 0.0
Cl 0.6 0.0
Ti 0.5 0.0
Mg 0.5 0.0
Cu 0.3 0.1
counts
keV

Appendix D: SEM Images – Artificial Solution #2 Neutralisation #1 Residue