Scanning Electron Microscope (SEM) Study of Filters with Residue

June, 2021
Kevin Tkachuk
WO#: 21A2037

AGAT Laboratories
2730 – 39th Avenue N.E.
Calgary, Alberta T1Y 7H6

AGAT Laboratories

SERVICE BEYOND ANALYSIS

TABLE OF CONTENTS

EXECUTIVE SUMMARY

Two filter samples were provided to AGAT for study by SEM. One filter sample was unused and provided a blank reference, and the other heavily used and visually much darker. The objective of the study was to determine the composition of the material on the dark, used filter. The blank filter consists mainly of carbon (C) and oxygen (O), with several additional minor to trace constituents including chlorine (Cl), sodium (Na), neon (Ne), fluorine (F), sulfur (S), silicon (Si) and phosphorous (P). This indicates that the filter material is a complex artificial polymer.

The used filter is heavily coated in collected material, to a thickness of at least 18µm, and in places over 100µm. This collected material varies in composition but is dominantly carbon (55-81%), with lesser oxygen (11-20%). From location to location additional minor elements were found, including chlorine (up to 22.9%), zinc (Zn) (up to 13.1%), nitrogen (up to 9.7%), aluminum (Al) (up to 7.3%), phosphorus (up to 5.2%), calcium (Ca) (up to 2.8%), and sulfur (up to 1.4%). Sodium was also detected but only at trace levels.

Interference from the underlying filter polymer may have affected measurement of chlorine, sodium, phosphorus, and sulfur, but in all cases except sodium the detected quantities of these elements was much higher in some locations of the used filter.

SEM imaging shows a mix of amorphous agglomerated material with irregular texture, and rare blocky particles. The collected material is consistent with the composition of exhaust particulate.

INTRODUCTION

This Scanning Electron Microscope (SEM) study describes the morphological and chemical characteristics of residual compounds captured on a filter. The composition of the clean blank filter was also included for comparison. Representative Energy Dispersive X-ray Spectrometry (EDX) data providing semi-quantitative elemental analysis is also provided. An overview of general sample information can be found below within **Table A:**

Sample ID.	Sample Type	Analyses*
1	Blank Filter	SEM EDX
2	Filter with Residue	SEM EDX

Table A - General sample information. * SEM - Scanning Electron Microscope analysis, EDX - Energy Dispersive X-ray analysis.

Figure A - Samples as submitted by the client. The blank filter is shown on the left, while the filter with captured residue is shown of the right.

METHODS OF ANALYSIS

Scanning Electron Microscopy/ Energy dispersive X-ray Analysis

The filter with the captured residue was slowly air dried to remove excess moisture. A representative portion of each sample was adhered onto an aluminum stub specimen mount with conductive carbon tape. The stubs were then sputter-coated with a conductive gold-palladium alloy for detailed Scanning Electron Microscopy (SEM) analysis and imaging. Energy dispersive X-ray Spectrometry (EDX) was also used in conjunction with SEM observation in order to determine the elemental composition of the observed constituents. The photomicrographs are taken in backscatter electron (BSE) mode to highlight variances in density. Heavy elemental compounds appear brighter than lower density compounds. It should be noted that elements with an atomic number lesser than 6 (carbon) cannot be detected by the EDX system.

SEM PHOTOMICROGRAPHS AND DESCRIPTIONS

Sample ID	1	**Sample Preparation**	Aluminum Stub Specimen Mount with conductive carbon tape and gold/palladium coating
Sample Description	Blank Filter	**Analysis**	SEM
			EDX

* SEM: Scanning Electron Microscopy, EDX: Energy-Dispersive X-ray

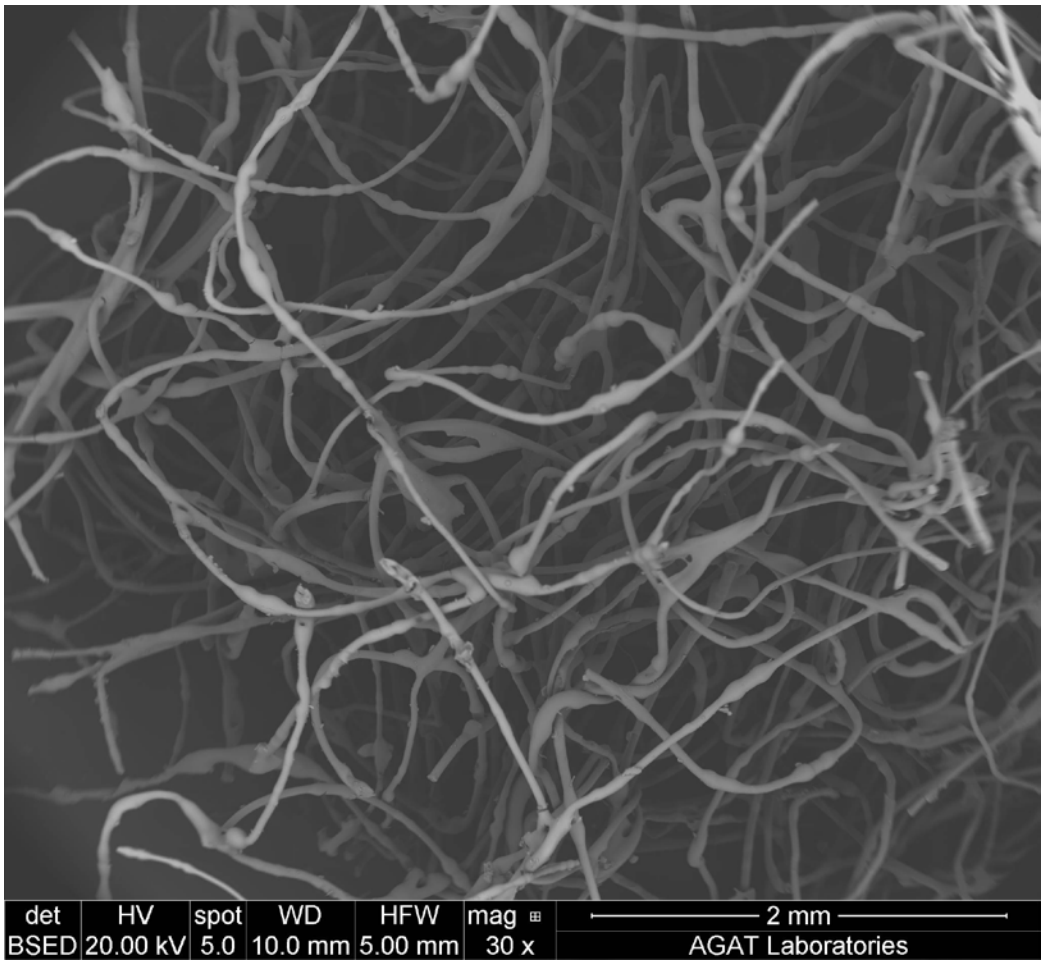

Figure 1.1 - Overview BSE photomicrograph of the blank filter fibers. See **Figure 1.1a** for representative semi-quantitative EDX data.

Element	Wt %	At %
C K	62.98	71.55
O K	29.40	25.07
F K	00.33	00.24
NeK	00.52	00.35
NaK	00.82	00.48
SiK	00.09	00.05
P K	00.08	00.03
S K	00.21	00.09
ClK	05.57	02.14

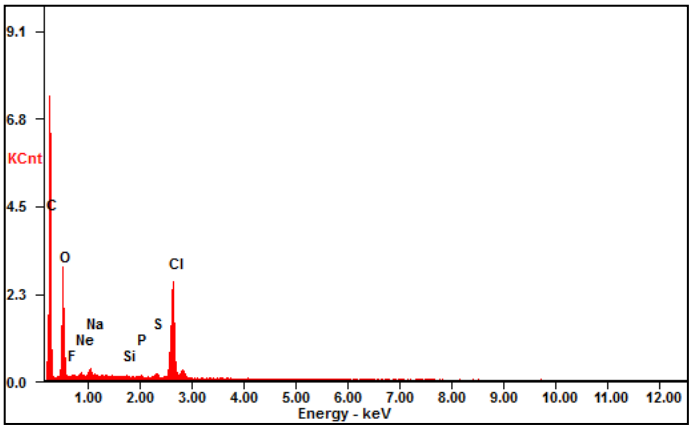

Figure 1.1a - Representative semi-quantitative EDX data representing the blank filter which consists of abundant carbon (C) and oxygen (O), along with minor to trace amounts of chlorine (Cl), sodium (Na), neon (Ne), fluorine (F), sulfur (S), silicon (Si) and phosphorous (P).

Sample ID	2	**Sample Preparation**	Aluminum Stub Specimen Mount with conductive carbon tape and gold/palladium coating
Sample Description	Filter with Residue	**Analysis**	SEM
			EDX

* SEM: Scanning Electron Microscopy, EDX: Energy-Dispersive X-ray

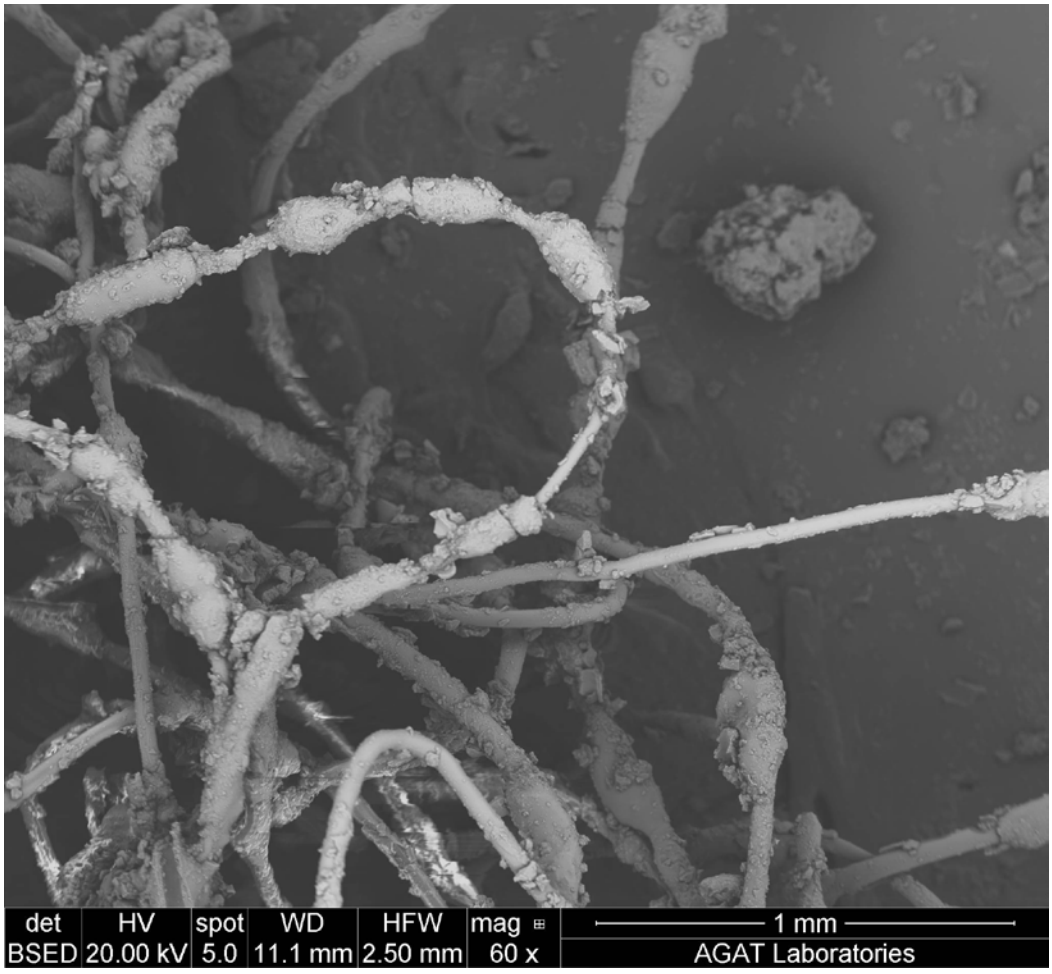

Figure 2.1 - Overview BSE photomicrograph of a portion of the filter with dried residual solids. See **Figures 2.1a and b** for representative semi-quantitative EDX data.

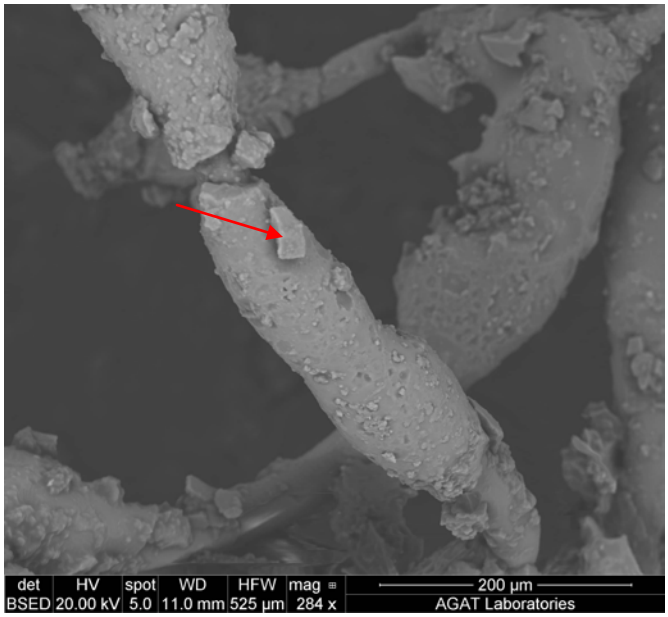

Figure 2.2 - BSE photomicrograph of dried residual solids on the filter fibers. The fiber coating materials, in addition to the blocky solids (highlighted by the red arrow), are fairly consistent in composition and consist predominately of carbon. See **Figures 2.1 a and b** for representative EDX data.

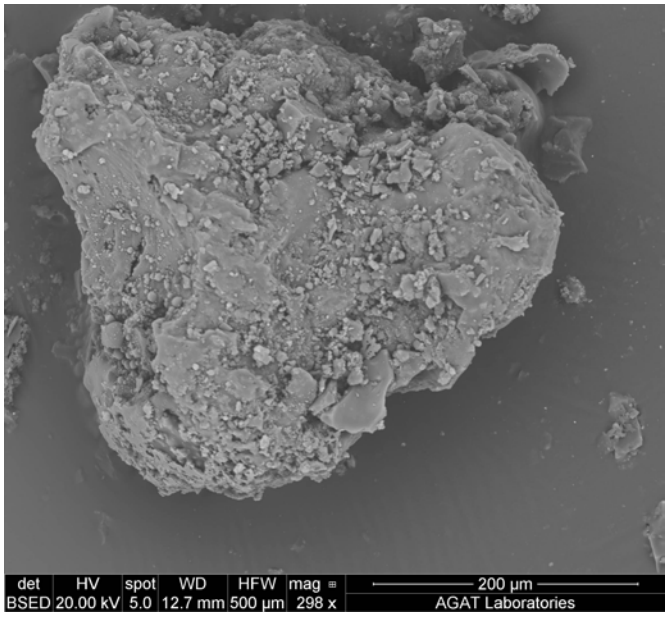

Figure 2.3 – BSE photomicrograph of a relatively large fragment of dried carbon-based solids. See **Figures 2.1 a and b** for representative EDX data.

Element	Wt %	At %
C K	81.38	85.92
O K	16.84	13.35
NaK	00.25	00.14
AlK	00.30	00.14
SiK	00.12	00.05
S K	00.28	00.11
ClK	00.70	00.25
CaK	00.13	00.04

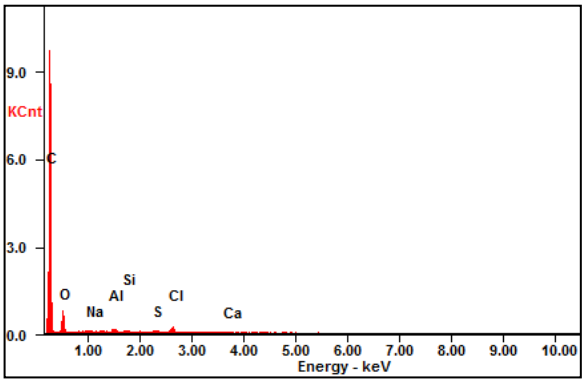

Figure 2.1a - Representative semi-quantitative EDX data representing the filter with residue before coating. The filter, plus residual coating, consists of abundant carbon (Ca) and oxygen (O), along with trace amounts of chlorine (Cl), aluminum (Al), sodium (Na),

Element	Wt %	At %
C K	76.55	84.93
O K	11.97	09.97
NaK	00.23	00.14
AlK	07.26	03.59
ClK	01.21	00.45
CaK	02.77	00.92

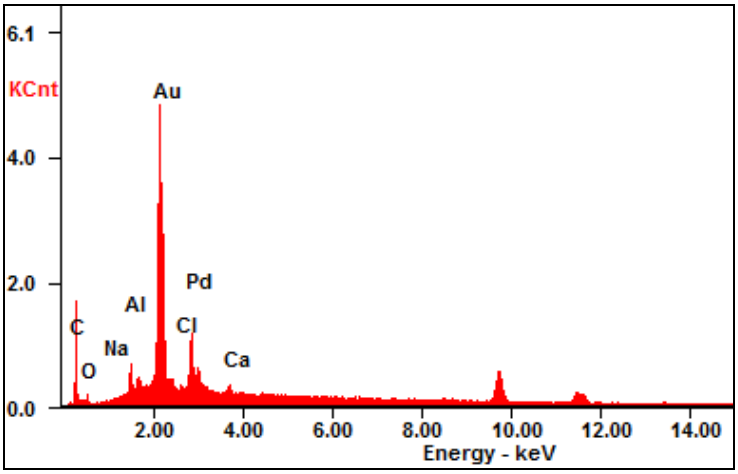

Figure 2.1a - Representative semi-quantitative EDX data representing the filter with residue after coating. Au and Pd are attributed to the conductive sputter coating and are not quantified.

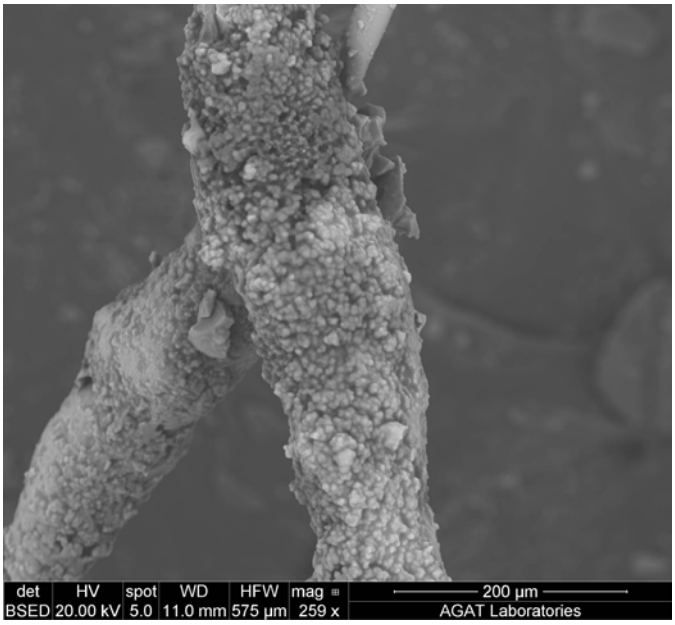

Figure 2.4 - BSE photomicrograph of dried residual solids on the filter fibers. The texture of the solids on this portion of the filter is more irregular in comparison to the smooth appearance of the residual solids shown in **Figure 2.2**. This is likely due to the presence of localize increased amounts of salts. See **Figure 2.4a** for representative EDX data.

Element	Wt %	At %
C K	59.18	73.42
N K	09.65	10.27
O K	05.54	05.16
NeK	00.90	00.67
NaK	00.11	00.07
AlK	00.84	00.46
ClK	22.92	09.63
CaK	00.86	00.32

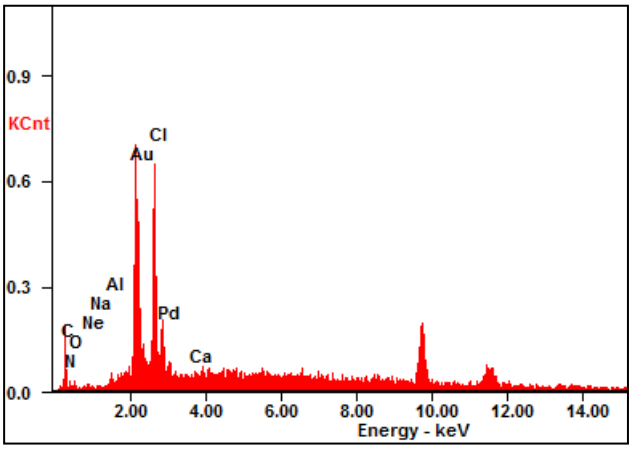

Figure 2.4a - Representative semi-quantitative EDX data for solids with potentially increased salt content. Au and Pd are attributed to the conductive sputter coating and are not quantified.

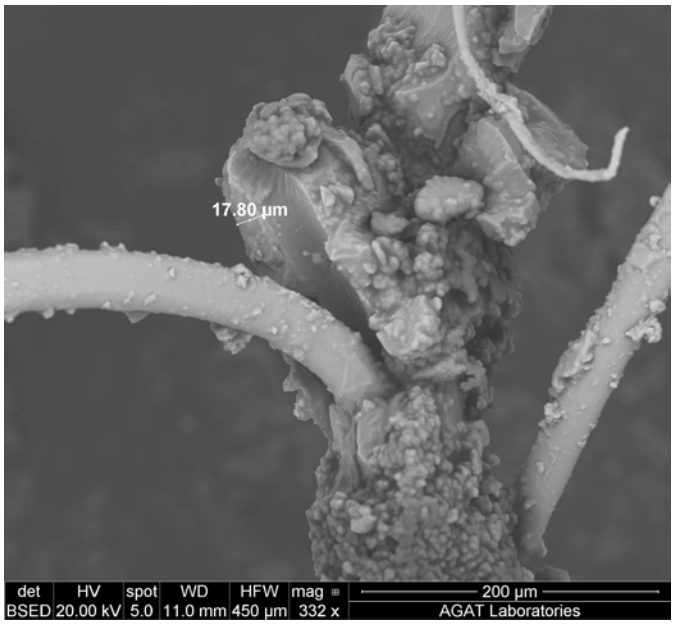

Figure 2.5 – BSE photomicrograph of dried residual solids which have pulled away from a filter fiber revealing the coating thickness to be a minimum of ~18µm.

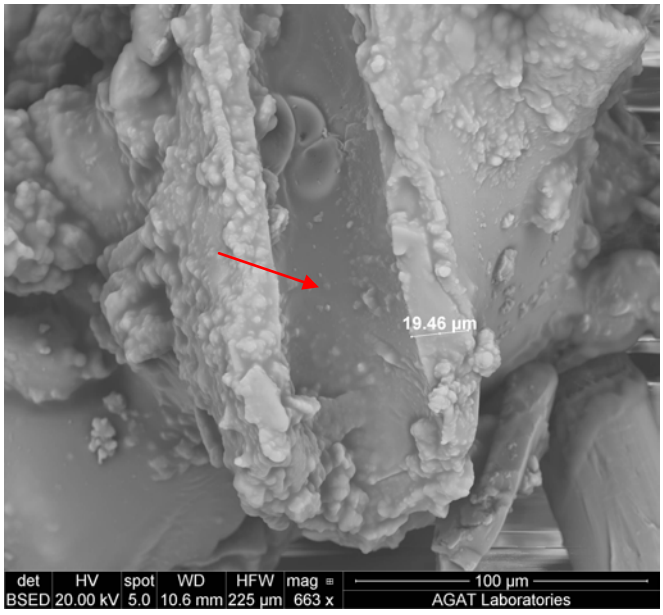

Figure 2.6 – Alternate high magnification BSE photomicrograph highlighting the texture of the dried residual solids. The hollow depression (highlighted by the red arrow) shows where the solids have become detached from the filter fiber. The solids have a minimum coating thickness of ~20 µm.

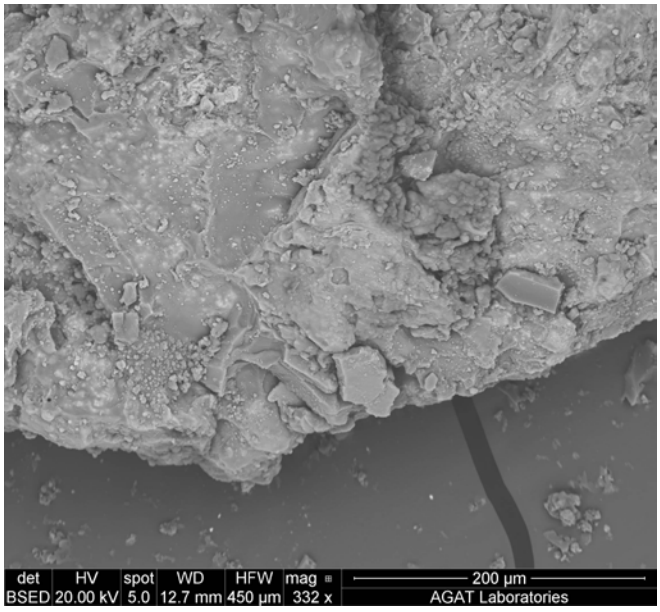

Figure 2.7 – BSE photomicrograph of a loose fragment of dried solids which consists predominately of carbon, oxygen, zinc, phosphorous, sulfur and calcium. See **Figure 2.7a** for semi-quantitative EDX data.

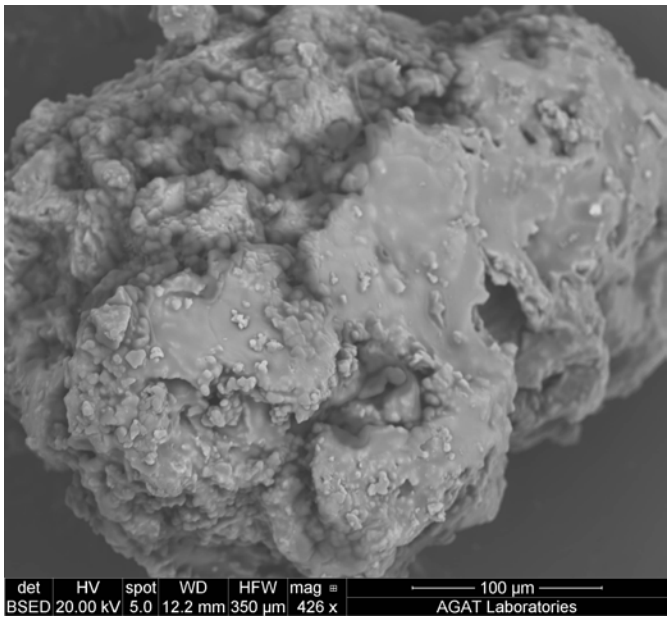

Figure 2.8 – Alternate BSE photomicrograph of a loose fragment of dried solids which consists predominately of carbon, oxygen, zinc, phosphorous, sulfur and calcium. See **Figure 2.8a** for semi-quantitative EDX data.

Element	Wt %	At %
C K	55.43	71.06
N K	02.15	02.36
O K	19.59	18.86
ZnL	12.82	03.02
MgK	00.24	00.15
AlK	00.13	00.08
SiK	00.31	00.17
P K	05.13	02.55
S K	01.42	00.68
ClK	00.02	00.01
CaK	02.75	01.06

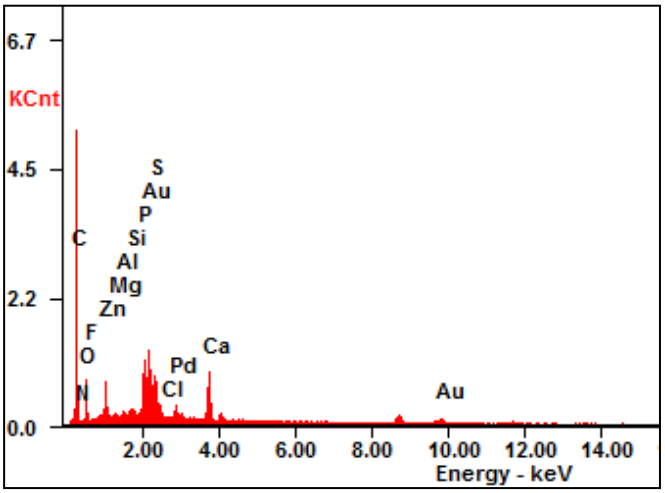

Figure 2.7a - Representative semi-quantitative EDX data of the loose solid fragment shown in Figure 2.7. Au and Pd are attributed to the conductive sputter coating and are not quantified.

Element	Wt %	At %
C K	55.49	71.10
N K	02.18	02.39
O K	19.87	19.11
ZnL	13.07	03.08
P K	05.17	02.57
S K	01.44	00.69
CaK	02.78	01.07

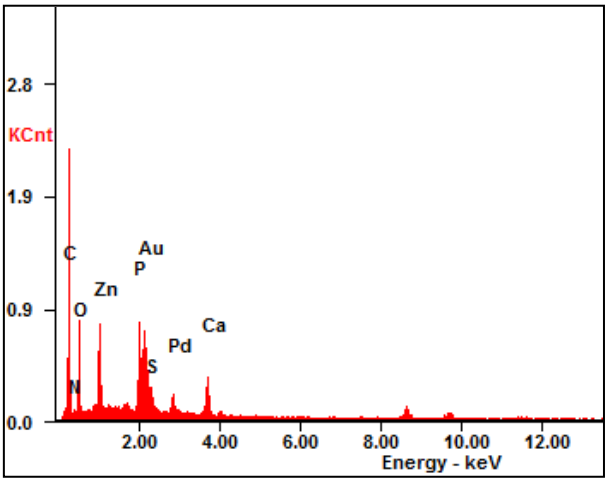

Figure 2.8a - Representative semi-quantitative EDX data of the loose solid fragment shown in Figure 2.8. Au and Pd are attributed to the conductive sputter coating and are not quantified.

PERMIT TO PRACTICE
AGAT LABORATORIES LTD.
Signature
Date June 18, 2024
PERMIT NUMBER: P 3939
The Association of Professional Engineers,
Geologists and Geophysicists of Alberta

PROFESSIONAL GEOSCIENTIST ALBERTA
EMILY VANDERSTAAL

Emily Vanderstaal

Prepared by Emily Vanderstaal, B.Sc., P.Geo.