SEM-EDX for Erionite Detection in Environmental Leaf Sample Protocols

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Erionite in New Zealand
Assessing and Managing Risk

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Purpose

To detect and characterise erionite fibres from environmental surface dust collected on tree leaves using SEM-EDX, based on morphology, elemental composition, and fibre size analysis.

1. Sample Preparation for SEM-EDX

1.1 Leaf Sample Handling

- Remove leaf samples from the freezer after **at least 1 month of dehydration at -18°C**.
- Cut leaves into small sections, **2.62 to 128 mm²** in area.
- Mount each leaf section **adaxial side up** (top surface) on an SEM stub using **double-sided conductive carbon tape** inside a **fume hood**.

⚠️ *Do not use the abaxial side — no mineral particles were detected on this side during initial trials.*

1.2 Drying and Coating

- Allow the mounted samples to **air dry at room temperature** for **3 days** inside a **plastic box under a fume hood**.
- Sputter-coat each sample with **platinum (Pt)** for **100 seconds** using a **Hitachi E-1045 or equivalent** coater.

2. SEM-EDX Analysis

2.1 Instrumentation

- Use a **Hitachi SU-70 Schottky field emission SEM** coupled with **Noran System 7 (NSS)** EDS or equivalent.

2.2 Operating Conditions

- **Accelerating voltage:** 15 kV
- **Acquisition time:** 60 seconds

3. Erionite Identification Criteria

3.1 Morphological Features (SEM-Based)

A particle is classified as a potential erionite fibre if it shows:

- A **fine, consistently elongated shape** with **aspect ratio > 3:1**,
  OR

- A **bundle or aggregate** structure containing **elongated fibrils**

These features are important for distinguishing erionite from non-fibrous dust and for identifying signs of **weathering, erosion, or fragmentation**, which indicate potential for airborne dispersal.

4. Elemental Composition (EDX-Based)

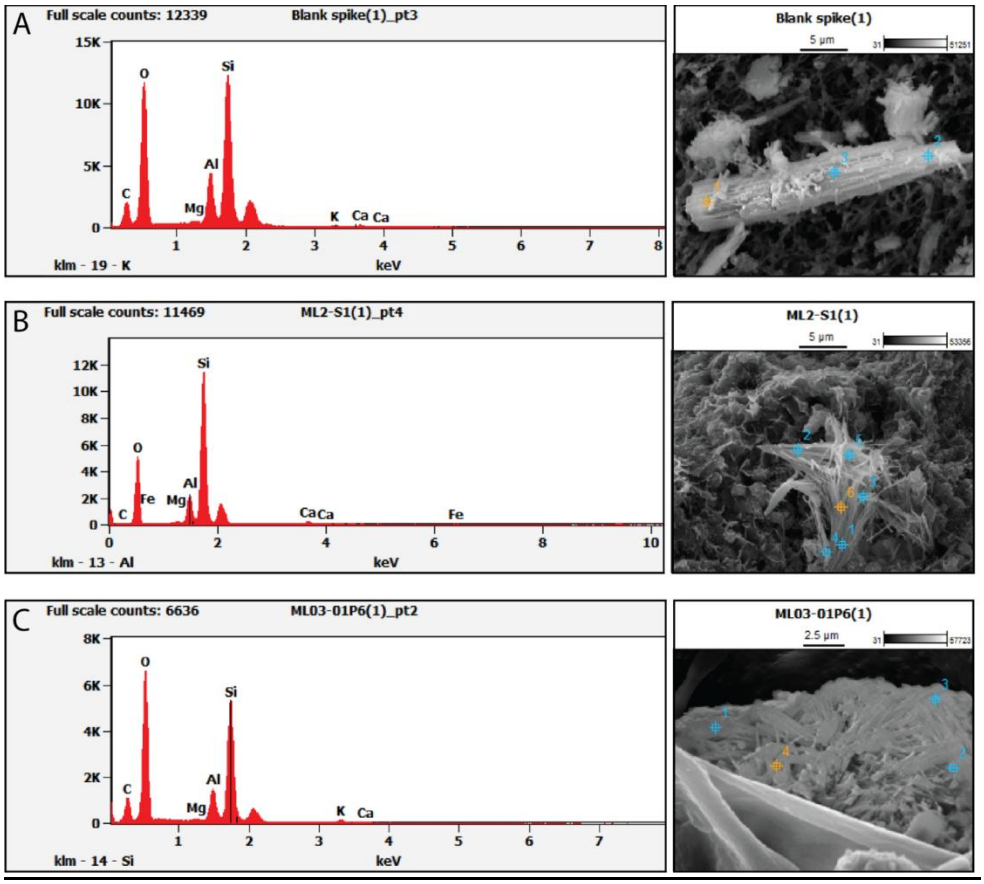
4.1 Framework Element Confirmation

- Confirm **strong peaks for Si (silicon)** and **Al (aluminium)** in the EDX spectrum.
- Also check for **extra-framework cations**: K (potassium), Na (sodium), Ca (calcium)

⚠️ Note: EDX spectra alone cannot differentiate erionite from similar zeolites like **mordenite** or **offretite**, as they can have overlapping chemical signatures.

4.2 EDX spectrum:

- **Major peaks:** O, Si, Al
- **Minor peaks:** K, Na, Ca, Mg



Examples of the SEM-EDX elemental spectrum of three types of samples tested. A. erionite. B. Mordenite containing rocks. C. Fibrous particles detected on leaf surface. Each panel shows the EDX spectra and the corresponding SEM images of the particles that were tested

5. Chemical Screening Indices

5.1 Tetrahedral Si Ratio (Tsi)

- Use the **Tsi ratio = Si / (Si + Al)** as a preliminary indicator.
- Measure **weight % (wt%) of Si and Al** from flat, central points on fibre surfaces.
- Compare Tsi values to literature ranges:

Zeolite	**Ideal Formula**	**Tsi Range**
Erionite	$K_2(Na,Ca_{0.5})_8[Al_{10}Si_{26}O_{72}]\cdot 30H_2O$	0.68–0.79
Mordenite	$(Na_2,Ca,K_2)_4[Al_8Si_{40}O_{96}]\cdot 28H_2O$	0.80–0.86
Offretite	$CaKMg[Al_5Si_{13}O_{36}]\cdot 16H_2O$	~0.74

Framework elements (Si, Al) are more stable in EDX analysis than extra-framework cations in small or weathered fibres.

5.2 Balance Error (E%)

- Optional: Use the **Passaglia et al. (1970) E% equation** (based on normalised 72 O atoms).
- Acceptable range for erionite: **±10%**.
- Note: E% may be unreliable for airborne fibres due their smaller size, which causes lack of extra-framework cation detection.

6. Fibre Size Measurement

6.1 Micrograph Capture

- Capture SEM images of identified fibres.

6.2 Measurement Software

- Use **ImageJ v1.53g62** for fibre measurement.

6.3 Measurement Approach

- Use the scale bar on the SEM micrograph to set up scale for the measurement first.
- Use **maximum Feret diameter** as fibre **length**
- Use **minimum Feret diameter** as **width**
- Calculate **aspect ratio** = length / width

These are equivalent to calliper lengths used in volcanic SEM studies (e.g., Bagheri et al., 2015).

7. Fibre Abundance Estimation

7.1 Subsampling Strategy

- Use **quadrant sampling** to estimate fibre abundance:
    - For each leaf sample, randomly select **six 0.1 mm² plots** using **400x magnification** on the SEM.

7.2 Fibre Counting

- Identify and count fibres in each plot using higher magnification.
- Process SEM images with **ImageJ** to count particles.

Follow **NIOSH 7400** fibre-counting rules: if fibres are agglomerated in air-dispersed form, count as one aggregate.

7.3 Fibre Level Calculation

Use the following formula:

$$L_f = \frac{\sum n_p}{N_p} \times 100$$

Where:

- **Lf** = fibres per cm² (surface abundance)
- **np** = number of fibres in each 0.1 mm² plot
- **Np** = total number of plots (6 per sample)

References

Fan, W., Gualtieri, A.F., Hamilton, A., Patel, J., Salmond, J. (2025). Determining factors affecting the accuracy of SEM-EDX data-based quantitative chemical analysis for identifying naturally occurring individual carcinogenic erionite fibers. Scientific Reports. https://www.nature.com/articles/s41598-025-09551-5