

Indicator minerals in exploration for specialty metal deposits: A QEMSCAN[®] approach



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1. Introduction

Quantitative Evaluation of Materials by Scanning electron microscopy (QEMSCAN[®]) was used to assess carbonatite indicator minerals in fluvial sediments from the drainage area of the Aley carbonatite, in north-central British Columbia. QEMSCAN[®] is a viable method for rapid detection and characterization of carbonatite indicator minerals with minimal processing other than dry sieving. Stream sediments from directly above, and up to 11 km downstream, of the carbonatite deposit were selected for this indicator mineral study. The geology of the Aley carbonatite is described by Mäder (1986), Kressal et al. (2010), McLeish (2013), Mackay and Simandl (2014), and Chakhmouradian et al. (2015).

Traditional indicator mineral exploration methods use the 0.25–2.0 mm size fraction of unconsolidated sediments (Averill, 2001, 2014; McCurdy, 2006, 2009; McClenaghan, 2011, 2014). Indicator minerals are detectable by QEMSCAN[®] at particle sizes smaller than those used for hand picking (<0.25 mm). Pre-concentration (typically by shaker table) is used before heavy liquid separation, isodynamic magnetic separation, optical identification using a binocular microscope, and hand picking (McClenaghan, 2011). Following additional sieving, the 0.5-1 and 1-2 mm fractions are hand picked for indicator minerals while the 0.25-0.5 mm fraction is subjected to paramagnetic separation before hand picking (Averill, 2001; McClenaghan, 2011). Hand picking indicator minerals focuses on monomineralic grains, and composite grains may be lost during processing. Composite grains are difficult and time consuming to hand pick and characterize using optical and Scanning Electron Microscopy (SEM) methods. A single grain mount can take 6-12 hours to chemically analyse (Layton-Matthews et al., 2014).

Detailed sample analysis using the QEMSCAN[®] Particle Mineral Analysis routine allows for 5-6 samples to be analyzed per day. When only mineral identification and mineral concentrations and counts are required, the use of a Bulk Mineral Analysis routine reduces the analysis time from ~4

hours to ~30 minutes per sample.

2. Potential indicator minerals

Pyrochlore supergroup minerals (as defined by Atencio et al., 2010), columbite-tantalite series minerals (as defined by Černý and Ercit, 1985; Černý et al., 1992), rare earth element (REE)-bearing fluorocarbonates (such as bastnaesite and synchysite), monazite, and apatite (Bühn et al., 2001; Belousova et al., 2002) are ideal carbonatite indicator minerals because of their high density and high content of key pathfinder elements such as Nb, Ta, LREE (ΣLa, Ce, Pr, and Nd), and P.

3. Methodology

3.1. Sampling, portable XRF analysis, and processing

Sediments in streams draining the area near the Aley carbonatite contain high concentrations of Nb, LREE, and P (Mackay and Simandl, 2014). Within carbonatites, Nb is predominantly incorporated in pyrochlore and columbite-(Fe), LREE in REE-fluorocarbonates, LREE, P, Th, Y in monazite, and P in apatite.

High pathfinder element (Nb, Ta, LREE, Y, P, Ba, Sr, U, and Th) concentrations determined by pXRF in the 125-250 µm dry sieved fraction (herein referred to as 'RAW') of stream sediments (Mackay and Simandl, 2014) and extent of mineralization in the Aley deposit (Kressal et al., 2010; Jones et al., 2014) gave the expectation that indicator minerals would be detectable by QEMSCAN[®].

The Mozley C800 laboratory mineral separator and Wilfley #13 shaking table were tested. Both can consistently concentrate the pathfinder elements and, by extension, targeted indicator minerals; however, the Mozley C800 is more suitable for processing smaller samples (Mackay et al., 2015a, b).

3.2. QEMSCAN[®] methodology

Approximately 2 g of RAW sample and/or corresponding Mozley C800 concentrate (CON) were mounted on polished smear sections (Fig. 1). Analysis was performed using an

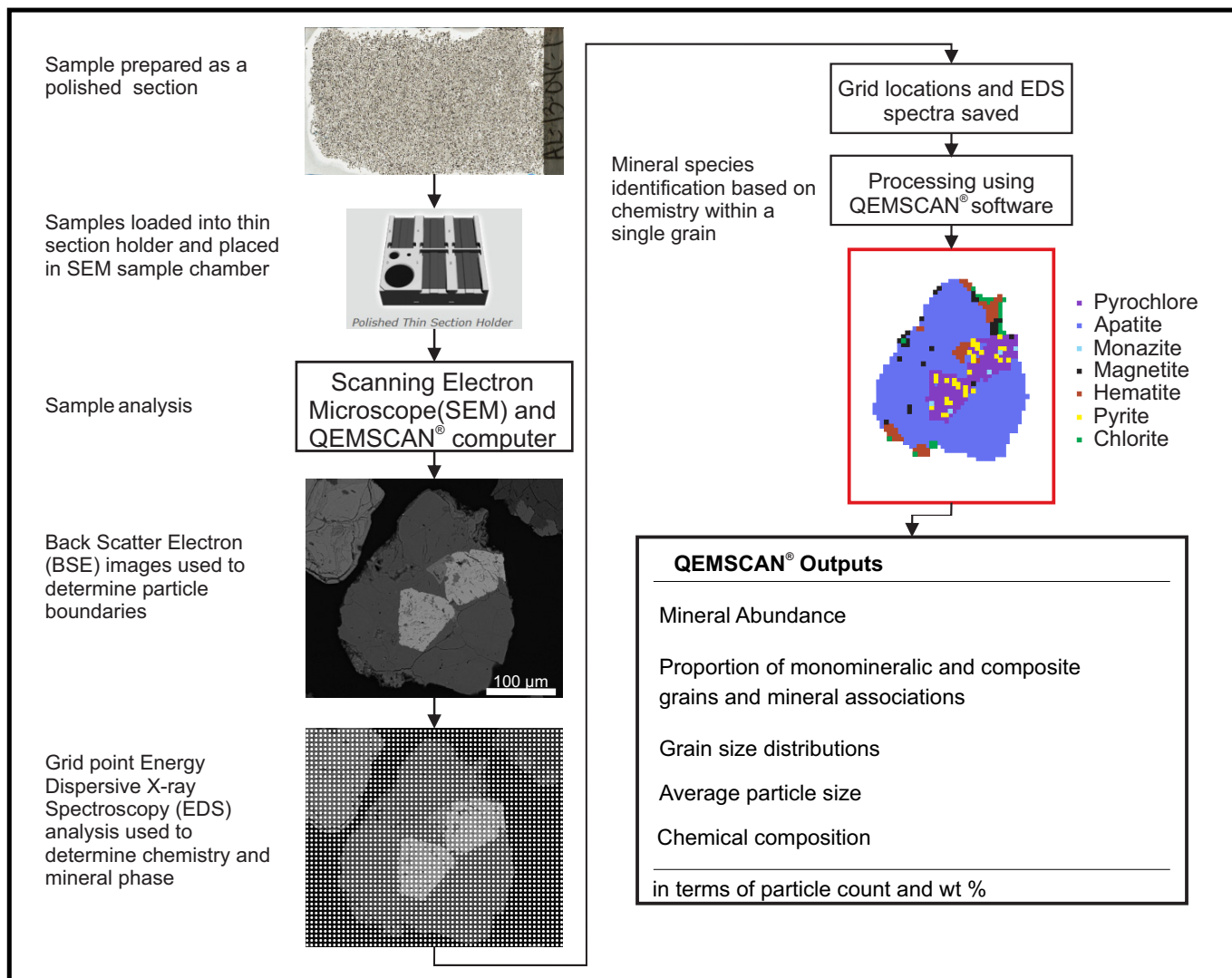


Fig. 1. Flow chart for QEMSCAN® analysis using the Particle Mineral Analysis routine. Based on QEMSCAN® software help file. Example grain is a polyminerallitic, or composite grain, with seven different mineral phases (identified in red box).

automated FEI Quanta Scanning Electron Microscope (SEM) with a tungsten filament operating at 10.00 nA and a maximum voltage of 25 keV.

The QEMSCAN® procedure and IDiscover® software were used to collect and process data. As part of the automated QEMSCAN® procedure (Particle Mineral Analysis routine), a Back Scatter Electron (BSE) image of each grain was acquired, with individual particle boundaries determined by contrast in brightness (Fig. 1). Energy Dispersive X-Ray Spectroscopy (EDS) analysis, performed on a grid (6.5 µm spacing in this study), provided chemical composition and identified each mineral based on a customized Species Identification Protocol. Mineral abundances in weight percent (wt.%) were calculated using particle volume (based on surface area) and density of the identified mineral. QEMSCAN® analysis output included particle count, mineral wt.%, particle size distribution(s), chemistry, and proportion of monomineralic (or liberated) and composite grains with mineral associations (Fig. 1).

QEMSCAN® bulk chemical composition was derived from the chemical composition of mineral particles determined by EDS, volume measurements (which assume the polished smear section has representative mineral composition and each particle is uniform in shape), and idealized mineral densities.

3.3. Chemical analysis

A 3.5-10.5 g split of each RAW sample and corresponding Mozley concentrates was analysed using lithium metaborate fusion followed by ICP-MS for trace elements, Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) for major elements, and XRF for Nb (when in concentrations >2500 ppm Nb).

4. Results

4.1. Geochemistry

The geochemical composition of the unprocessed 125-250 µm fraction of stream-sediment samples and corresponding

Mozley concentrates from the Aley carbonatite drainage were determined by ICP-MS, ICP-AES, and XRF analyses. Carbonatite pathfinder elements associated with prospective indicator minerals were detected in all samples. Concentrations of Nb in RAW samples from the Aley drainage range from 3000-10,000 ppm with an average of 5900 ppm. Concentrations in corresponding Mozley concentrates are substantially higher, ranging from 11,900-25,900 ppm Nb and averaging 17,600 ppm Nb. Similarly, concentrations of Ta, Zr, LREE, U, Th, and P_2O_5 increased following Mozley processing.

4.2. Indicator mineral abundances

Mineral abundance by wt.% and particle count for each polished smear section were determined using QEMSCAN[®] analysis. The main carbonatite indicator minerals were detected in RAW samples from the Aley carbonatite without additional processing (Fig. 2). However, minimal processing by Mozley C800 table effectively concentrated heavy indicator minerals (Fig. 2). Specifically, pyrochlore abundance increased on average by 12.9 x, columbite-(Fe) by 2.4 x, monazite by 3.2 x, REE-fluorocarbonates by 2.6 x, apatite by 1.7 x, magnetite by 5.8 x, and zircon by 3.2 x.

5. Discussion

5.1. Particle size distribution

QEMSCAN[®] provides particle size distributions for individual minerals in terms of particle count and weight percent in the 125-250 μm size fraction of RAW stream sediments and corresponding Mozley concentrates from the Aley drainage area. Particle size distributions measured by particle count for some trace minerals (eg. monazite and REE-fluorocarbonates), are skewed towards smaller (<20 μm) particle diameters. This is most likely due to the presence of small inclusions of these trace minerals in composite grains. The particle count for pyrochlore (Fig. 3a), columbite-(Fe) (Fig. 3c), monazite, and apatite show an overall decreasing trend with increasing distance downstream of source rocks in the Aley carbonatite. Abundant mineral constituents and ore minerals (e.g., pyrochlore and columbite-[Fe]) occur as larger particles with nearly normal distributions of particle diameters (Figs. 3b, d).

5.2. Mineral abundance and pathfinder element concentration

There is a good fit between the concentration of carbonatite pathfinder elements determined using laboratory geochemical analysis and abundance of carbonatite indicator minerals determined by QEMSCAN[®]. The highest R^2 are obtained by comparing bulk sample chemistry and indicator mineral concentration in terms of wt.%. For example, Nb content is related to pyrochlore ($R^2=0.72$; Fig. 4a) and columbite-(Fe) ($R^2=0.78$; Fig. 4b) concentrations; fersmite is a minor constituent (Fig. 2). Total niobate (sum of pyrochlore, columbite-[Fe], and fersmite) concentration shows the best fit with Nb content of samples ($R^2=0.90$; Fig. 4c). As fersmite is a minor Nb-bearing

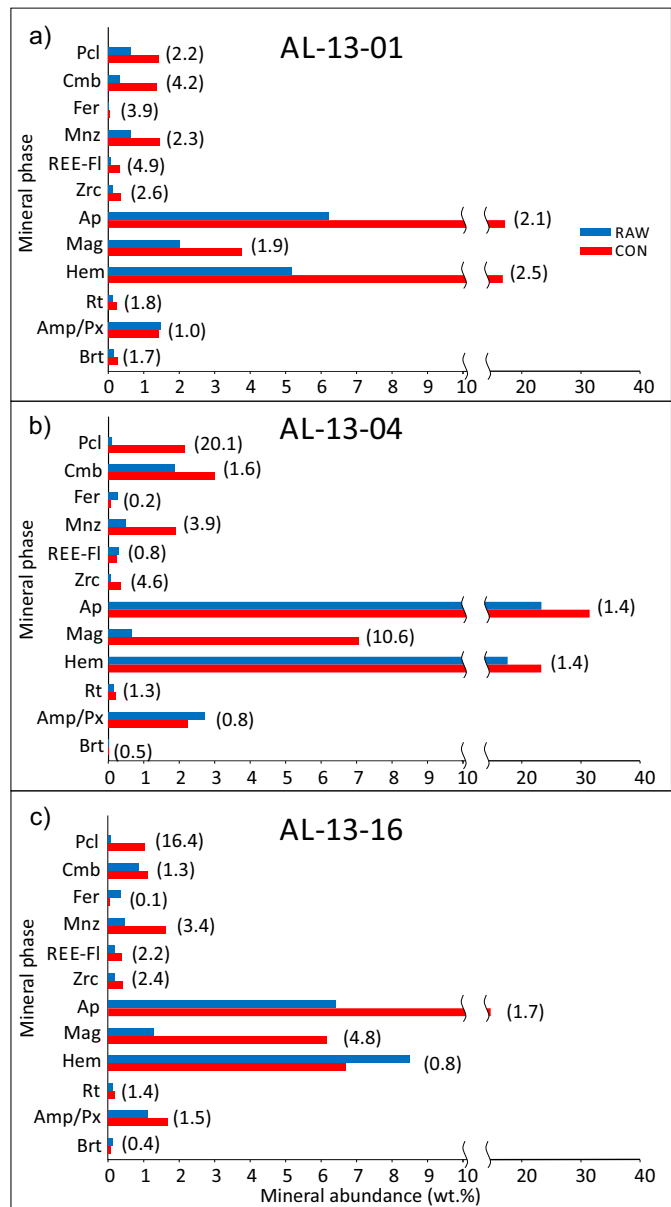


Fig. 2. Mineral abundances (wt.%; determined by QEMSCAN[®]) for RAW and corresponding Mozley C800 concentrates (CON) for selected samples **a)** AL-13-01, **b)** AL-13-04, and **c)** AL-13-16 from Aley (AL) carbonatite drainage. Concentration factors for CON relative to corresponding unprocessed samples (RAW) are shown in parentheses. Mineral abbreviations: pyrochlore (Pcl); columbite-(Fe) (Cmb); fersmite (Fer); REE-fluorocarbonates (REE-FI); zircon (Zrc); apatite (Ap); magnetite (Mag); hematite (Hem); rutile (Rt); amphibole/pyroxene (Amp/Px); and barite (Br).

constituent, a low R^2 between Nb content (XRF) and fersmite concentrations (QEMSCAN[®]) is expected.

LightREE content is strongly related to monazite concentration ($R^2=0.91$) and less strongly to REE-fluorocarbonate concentration ($R^2=0.51$; Fig. 4d). Concentration of P_2O_5 in RAW samples and corresponding Mozley concentrates is strongly related to apatite concentration ($R^2=0.98$) and weakly to monazite content ($R^2=0.25$). Thus, overall indicator mineral

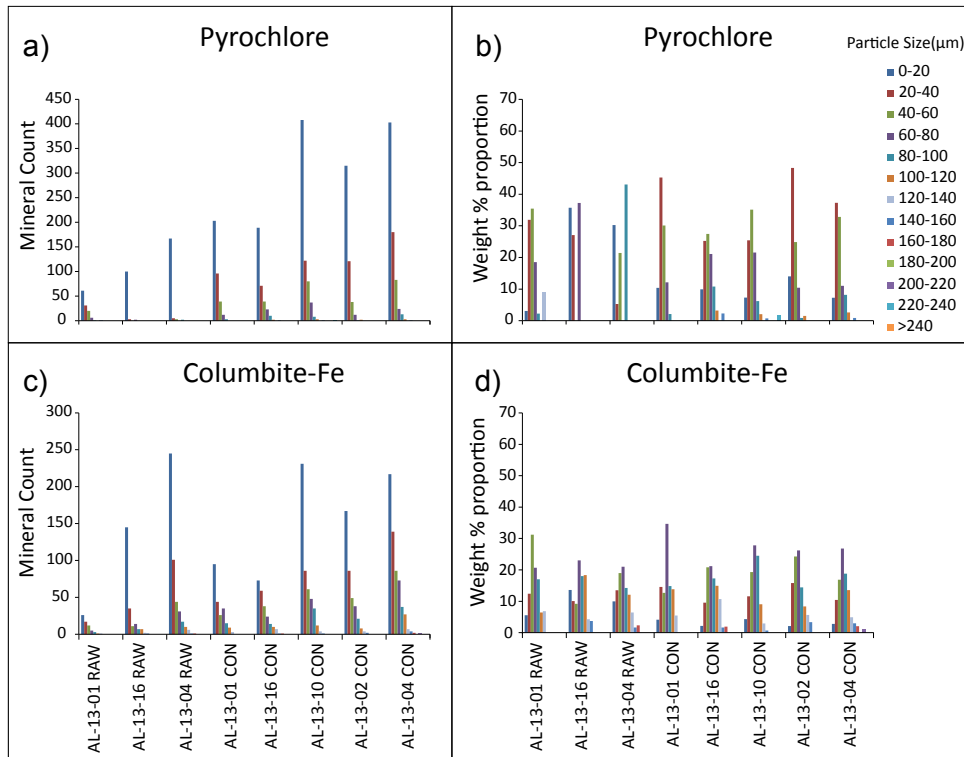


Fig. 3. Particle size distribution in terms of mineral count and wt.%. **a)** and **b)** pyrochlore; **c)** and **d)**, columbite-(Fe). RAW samples and corresponding Mozley concentrates (CON) samples are each ordered from west (away from the deposit) to east (nearest the deposit). Mineral sizes are in μm .

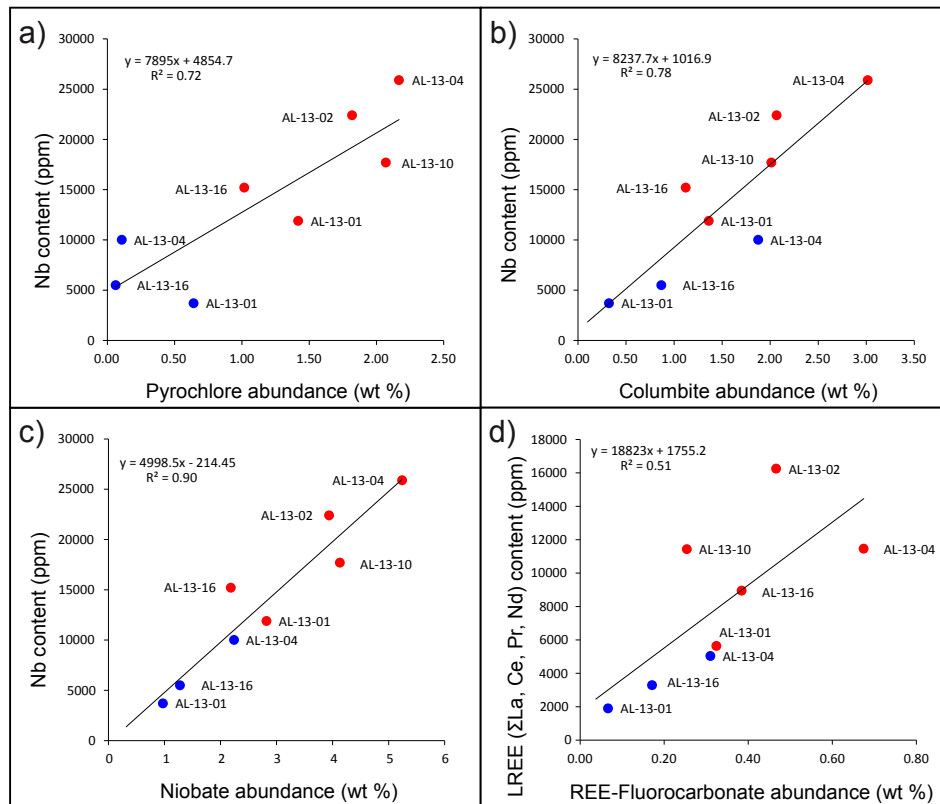


Fig. 4. Relationship between indicator mineral concentrations (determined by QEMSCAN[®]) and corresponding pathfinder element content for: **a)** Nb vs. pyrochlore; **b)** Nb vs. columbite-(Fe); **c)** Nb vs. niobates (includes pyrochlore, columbite-(Fe), and fersmite); **d)** LREE vs. REE-fluorocarbonates. Results are for unprocessed samples (blue circles) and Mozley C800 concentrates (red circles).

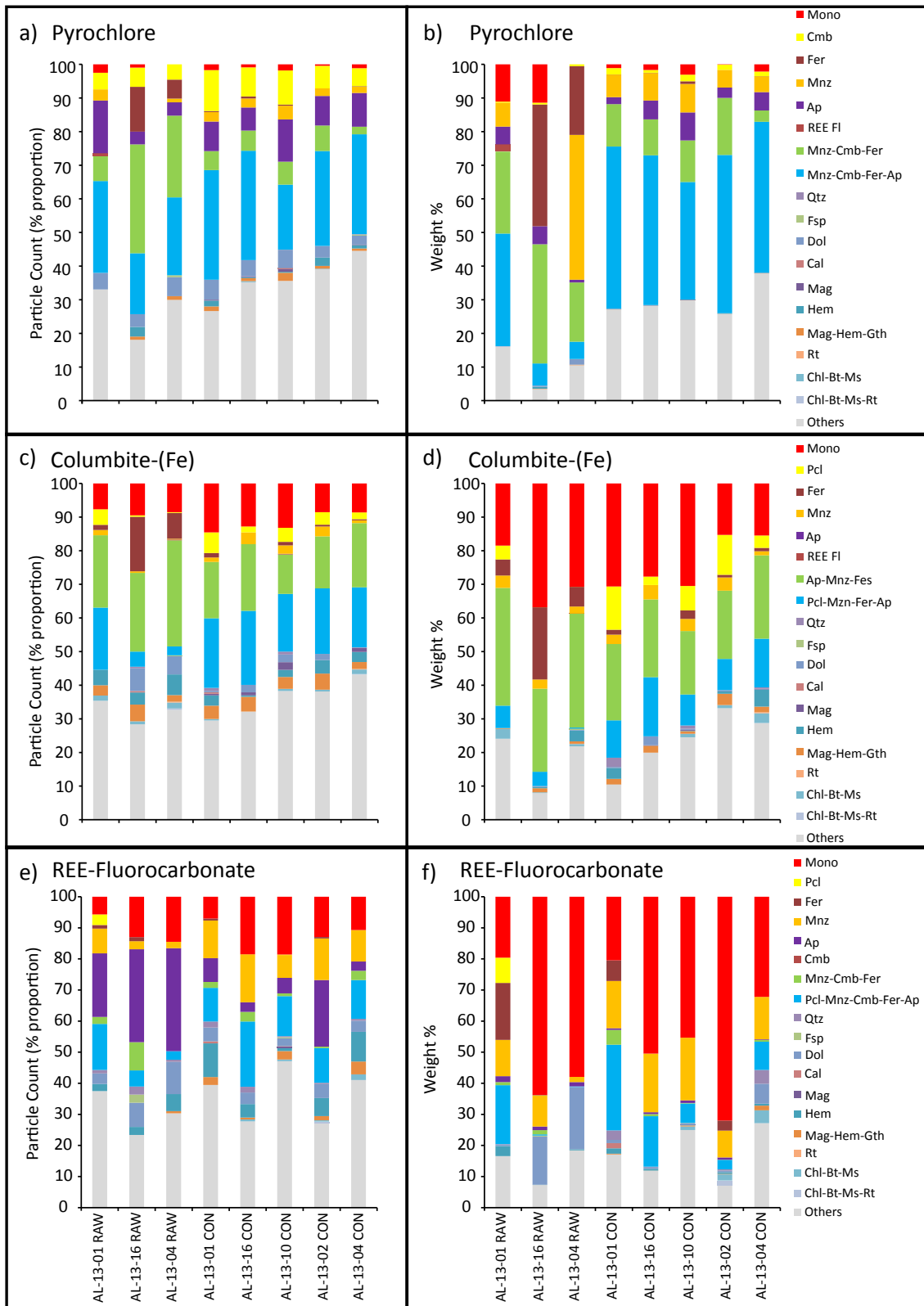


Fig. 5. Proportions of monomineralic (Mono) and composite grains with predominant mineral associations (in terms of mineral count and wt.%) for: **a)** and **b)** pyrochlore; **c)** and **d)** columbite-(Fe); **e)** and **f)** REE-fluorocarbonates. Results are for RAW samples and corresponding Mozley concentrates (CON). Sample distance increases from directly above the deposit (AL-13-04) to 11.5 km downstream (AL-13-01). ‘Others’ refers to complex composite grains, and those containing unidentified mineral phases. Abbreviations: pyrochlore (Pcl); columbite-(Fe) (Cmb); fersmite (Fer); REE-fluorocarbonates (REE-FI); apatite (Ap); magnetite (Mag); hematite (Hem); rutile (Rt); quartz (Qtz); dolomite (Dol); calcite (Cal); feldspar (Fsp); hematite (Hem); goethite (Gth); chlorite (Chl); biotite (Bt); and muscovite (Ms).

abundance measured by wt.% is representative of bulk sample composition determined using ICP-MS, ICP-AES, and XRF.

5.3. Mineral association in composite grains

QEMSCAN[®] determines the quantitative proportions of monomineralic (or liberated) grains and minerals in composite grains. Key mineral associations in composite grains and the proportion of monomineralic grains are shown in Fig. 5. Pyrochlore (wt.%) is predominantly contained in composite grains composed of minerals associated with carbonatite magmatism and alteration (columbite-[Fe], apatite, fersmite, \pm monazite; Figs. 5a, b). Apatite (wt.%) is contained predominantly in monomineralic grains. Columbite-(Fe) (Fig. 5d), REE-fluorocarbonates (Fig. 5f), and monazite fall in between these two extremes.

6. Conclusions

QEMSCAN[®] can detect indicator minerals in the dry sieved 125-250 μ m size fraction without additional processing from stream-sediment samples taken near the Aley carbonatite. Minimal processing using a Mozley C800 mineral separator increases the concentration of indicator minerals found in low concentrations in a predictable way. QEMSCAN[®] can detect and characterize particle sizes too small for hand picking and provides quantifiable proportions of monomineralic grains and mineral associations in composite grains. QEMSCAN[®] can fully characterize a sample in 3.5-4.5 hours using the 'Particle Mineral Analysis' routine, much faster than the 6-12 hours required for traditional techniques to characterize a single sample with mineralogically complex grains.

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