Mineral liberation analysis

Rita Kallio,
University of Oulu

Introduction

Automated mineralogy (AM) is a term that has been used to describe the automation of the analytical process of quantifying minerals, rocks, and production materials with Scanning Electron Microscopy (SEM). Measurements begin with collecting backscattered electron (BSE) images and handling them with image analysis software. Image data is complemented with collecting energy-dispersive X-ray spectra, which is then classified and post-processed for desirable outcomes.

With advances in computing power and speed, automated mineralogy techniques have been developed for the minerals industry and used in research institutes for several decades. Common to all SEM-AM systems is the combination of a hardware platform and a specific image analysis and processing software. Almost every scanning electron microscope (SEM) can be used as a hardware platform for AM. Commercially available lab-based platforms include:

- Mineral Liberation Analysis (**MLA**) (Thermo Fischer)
- Quantitative Evaluation of Minerals by Scanning Electron Microscopy (**QEMSCAN**) (Thermo Fischer)
- Tescan Integrated Mineral Analyzer (**TIMA**) (Zeiss Mineralogic)
- **INCAMineral** – **Aztec Mineral** (Oxford Instruments)
- Advanced Mineral Identification and Characterization System (**AMICS**), developed by Yingsheng Technology and now marketed through Bruker Inc.

Comparison of various platforms for SEM-AM

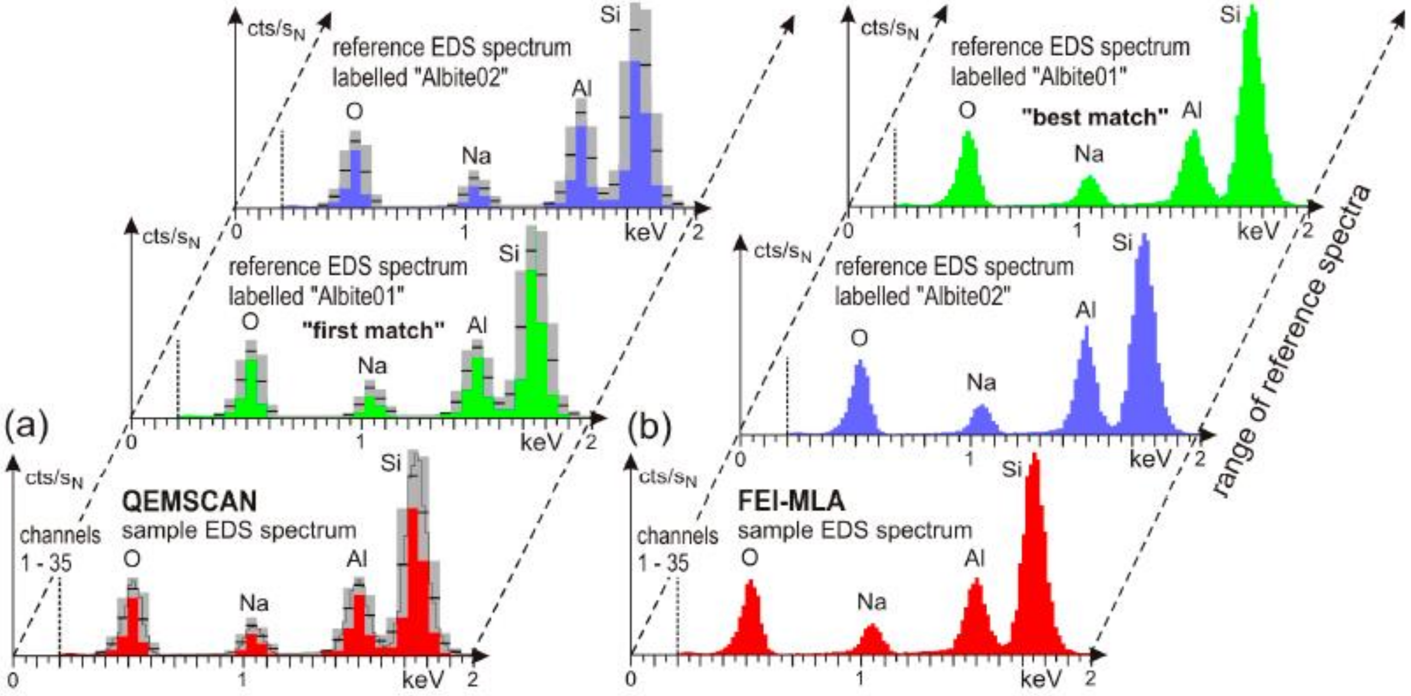


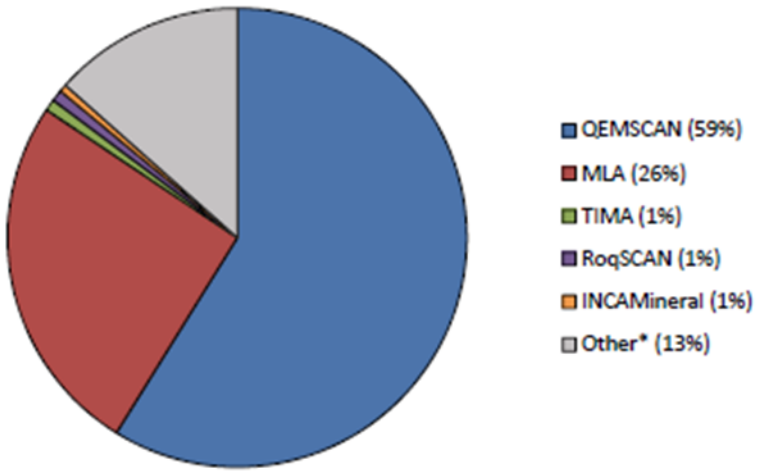
Image from Schulz et al., 2020

Table 5.2 Basic auto-SEM comparison among different platforms

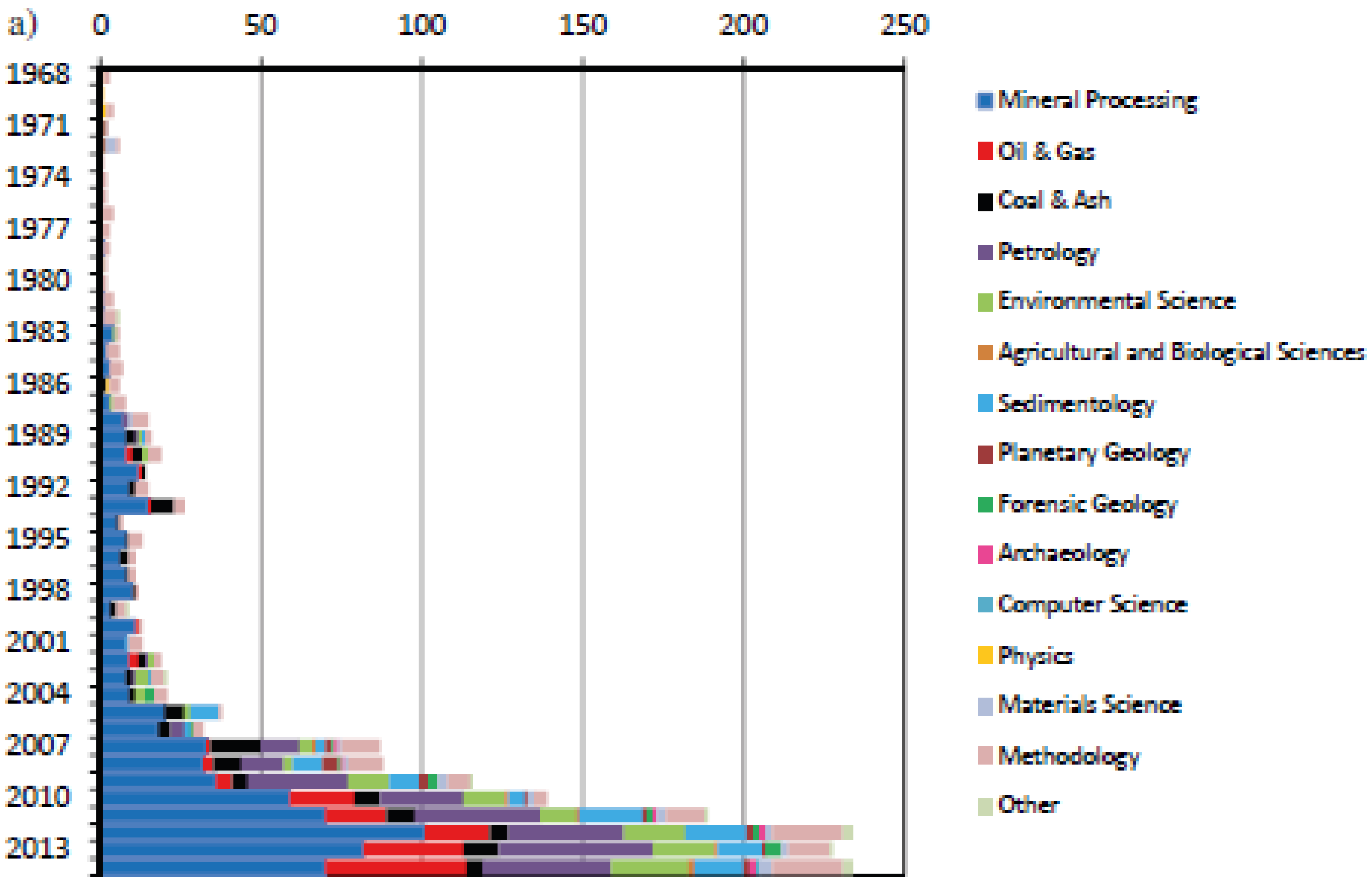
	QEMSCAN	MLA	Mineralogic	TIMA
Historic SEM platform	Oxford LEOs/ Carl Zeiss EVOs	FEI Quanta	N/A	N/A
Current SEM platform	FEI Quanta/ FEG	FEI Quanta/ FEG	Carl Zeiss EVO	TESCAN MIRA FEG/VEGA
EDX detection	Oxford/Bruker SDD	Bruker SDD	Bruker/Oxford SDD	PulseTor SDD
Number of detectors (historical)	4	1	N/A	N/A
Number of detectors (current)	Up to 4	Up to 4	Up to 4	Up to 4
Traditional mode of measurement	X-ray mapping	X-ray centroiding	N/A	N/A
Current mode of measurement	X-ray mapping/X-ray centroiding	X-ray centroiding/ X-ray mapping	X-ray centroiding/ X-ray mapping	X-ray mapping
Mineral ID	Species identification protocol	Standard file matching	Mineral recipe identification	Mineral classification rules/profiles
Accommodation of 30mm sample blocks	9–16	14	9–16 (Customizable)	7 (Customizable)
Consistent specimen current for BSE calibration	Yes	No (range very narrow)	No	?
Spectral matching	First match	Best fit	Priority/first match (best fit if priorities are equal)	?
Iron oxide distinction	Arbitrary yet consistent BSE cutoff	BSE histogram peak separation	Correlative microscopy	?
Typical X-ray count rates	1000	1000	>3000 (Variable)	?

Tonzetic, 2015

Publications related to automated mineralogy



Cumulative distribution of publications related to automated mineralogy by system type, end of 2014.



Both graphs from Sandmann, 2015

What kind of information can be obtained with automated mineralogy?

- Modal mineralogy – mineral abundance and sample elemental distributions
- Mineral associations
- Size-by-size **liberation analysis** and locking
- Digital textural mineral maps
- Bright-field search for precious metal
- Particle and grain size distributions
- Shape factors
- Porosity

Definition of particles and grains: a) PARTICLE consisting of various grains
b) Particle consisting of one GRAIN. Image from Sandmann, 2015

The advantages of automated digital image analysis techniques for mineralogical investigations over traditional microscopy techniques include fast acquisition time that provides a more statistically representative analysis of a sample as well as the ability to distinguish fine-grained or complex intergrown minerals at the micrometer scale. It also reduces the potential for operator bias and human error.

Hierarchy of material in mineral processing circuit

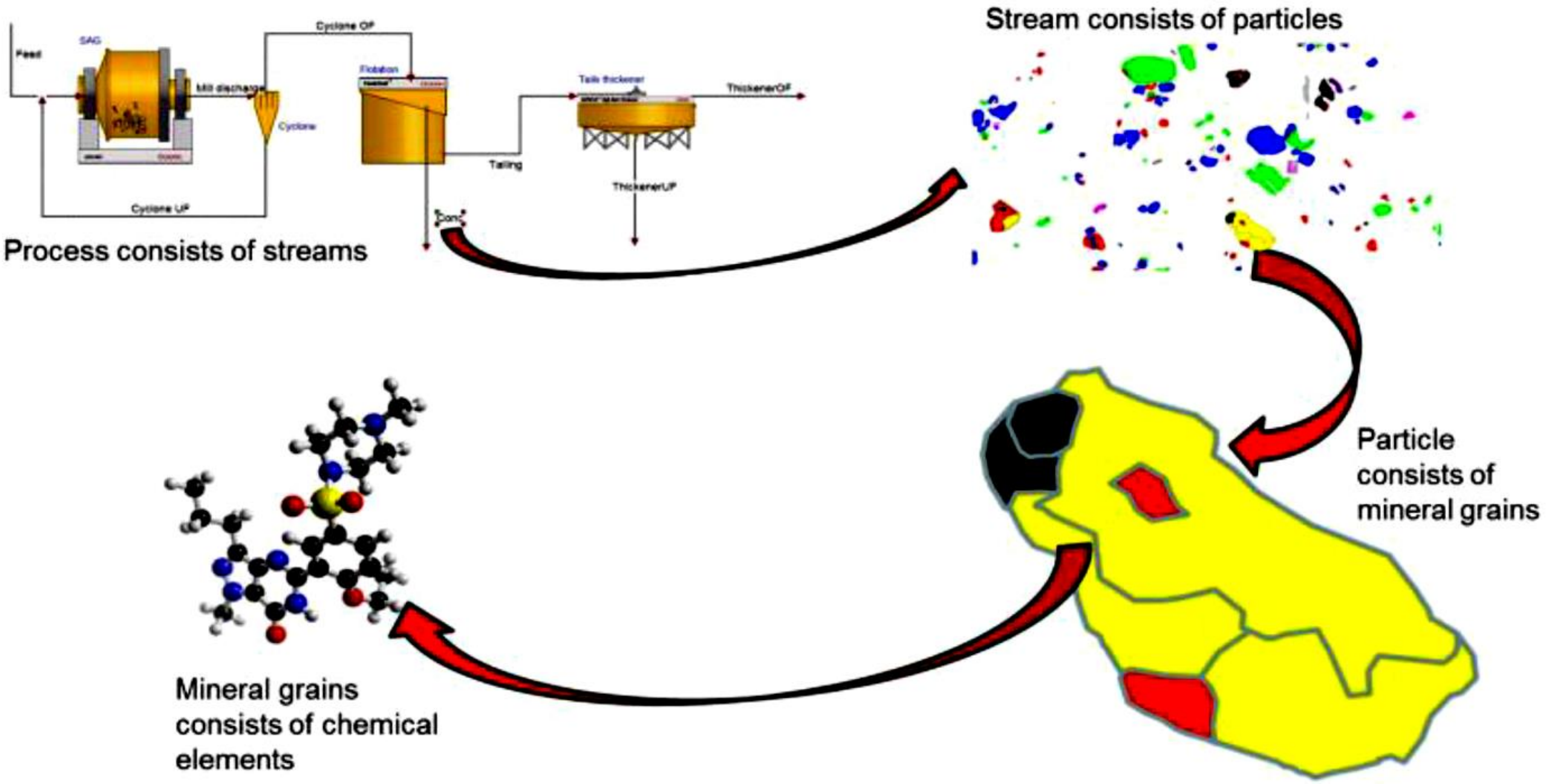
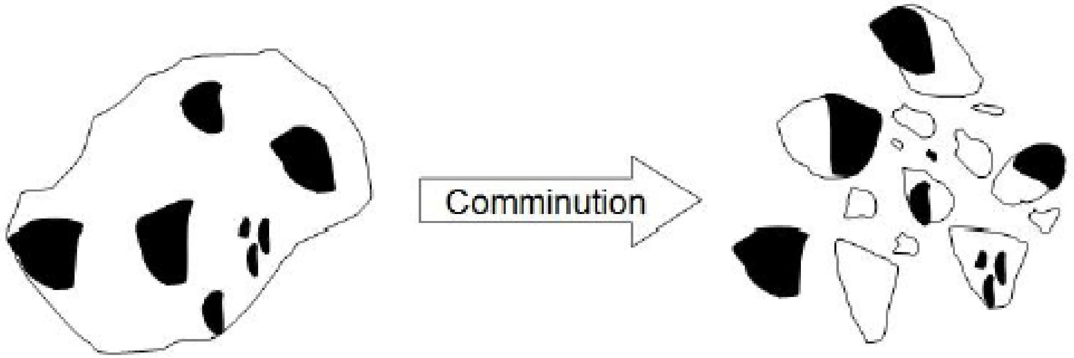


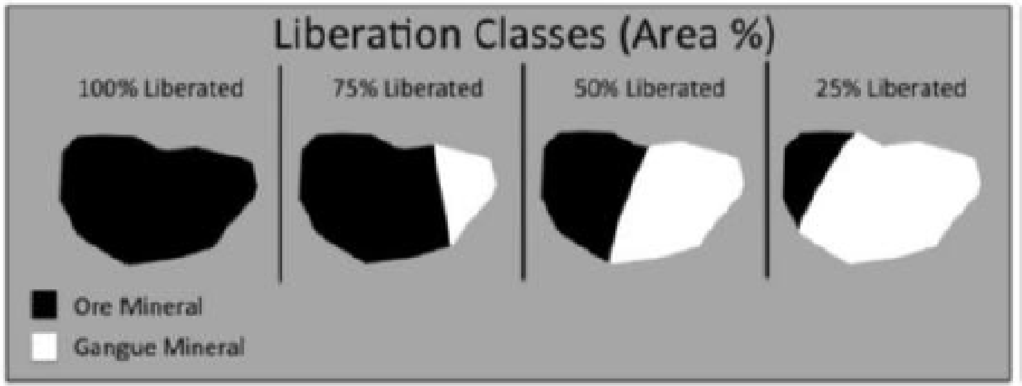
Image modified from: ”A way forwards in process mineralogy – using automated mineralogy for modelling and simulating beneficiation processes” P. Lamberg.

What is liberation?

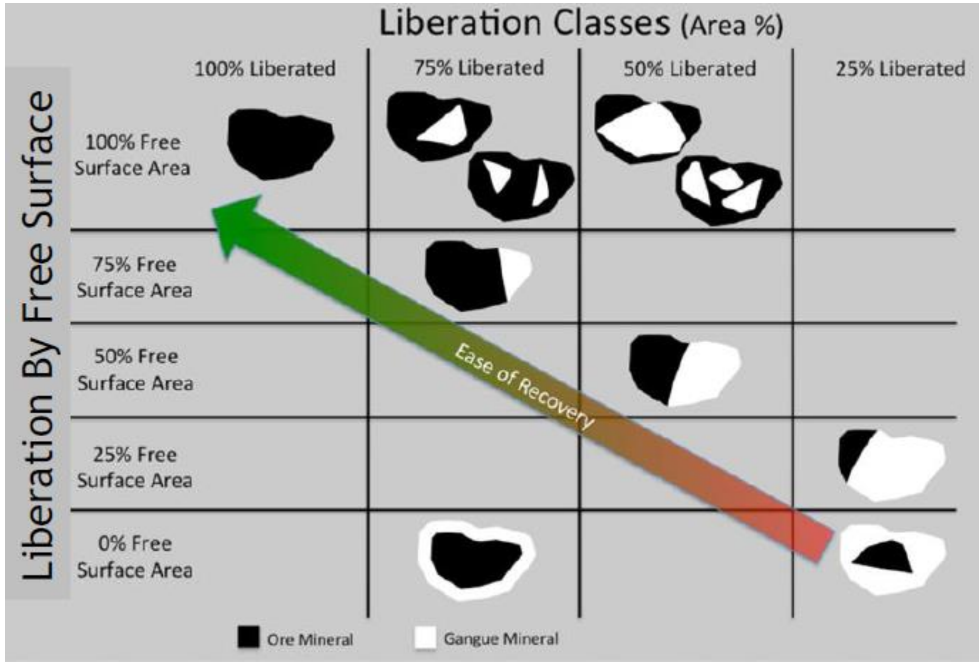
- Liberation is achieved in mineral processing by comminution - crushing and grinding – of the material to obtain the mixture of particles and grains.
- The **degree of liberation** refers to the percentage of the mineral occurring as free particles in the ore in relation to the total content. Liberation of a particle with respect to a given mineral of interest can be computed and expressed in one of the two following forms:

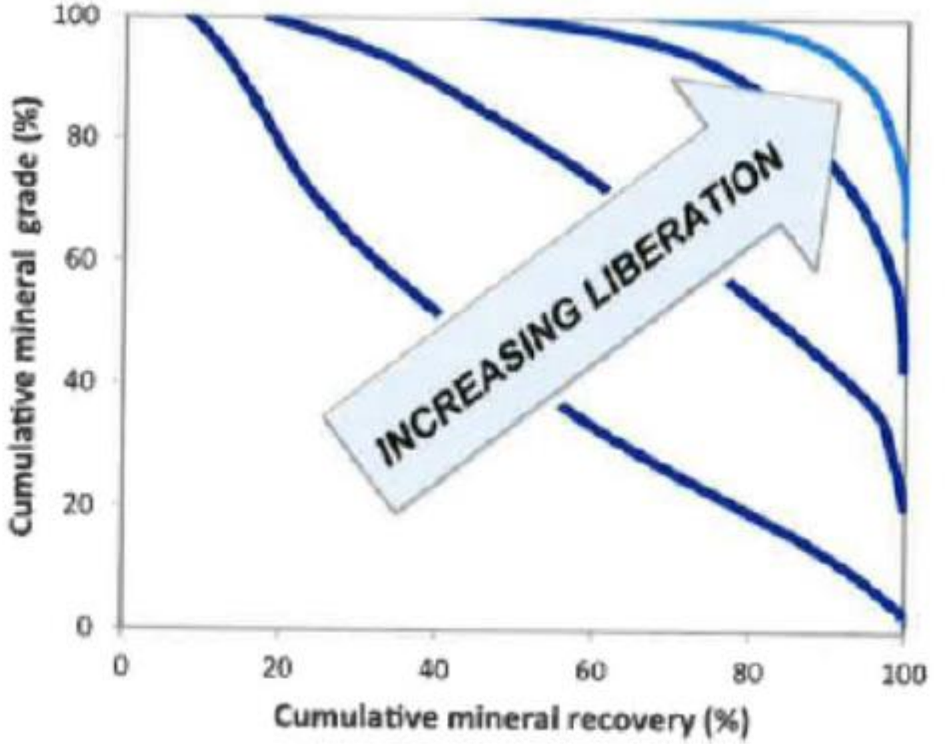
  1. **Surface area**. Liberation of a particle is the length fraction on the outer perimeter of the particle covered by a mineral(s) of interest with respect to the whole outer perimeter of the particle expressed as a percentage.
  2. **Volume**. Liberation of a particle is the area fraction of a mineral(s) of interest with respect to the total area of the particle normally expressed as a percentage.

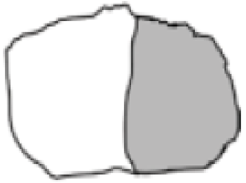
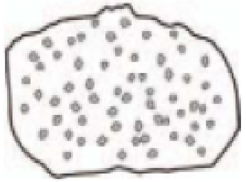
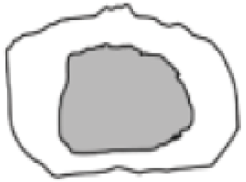




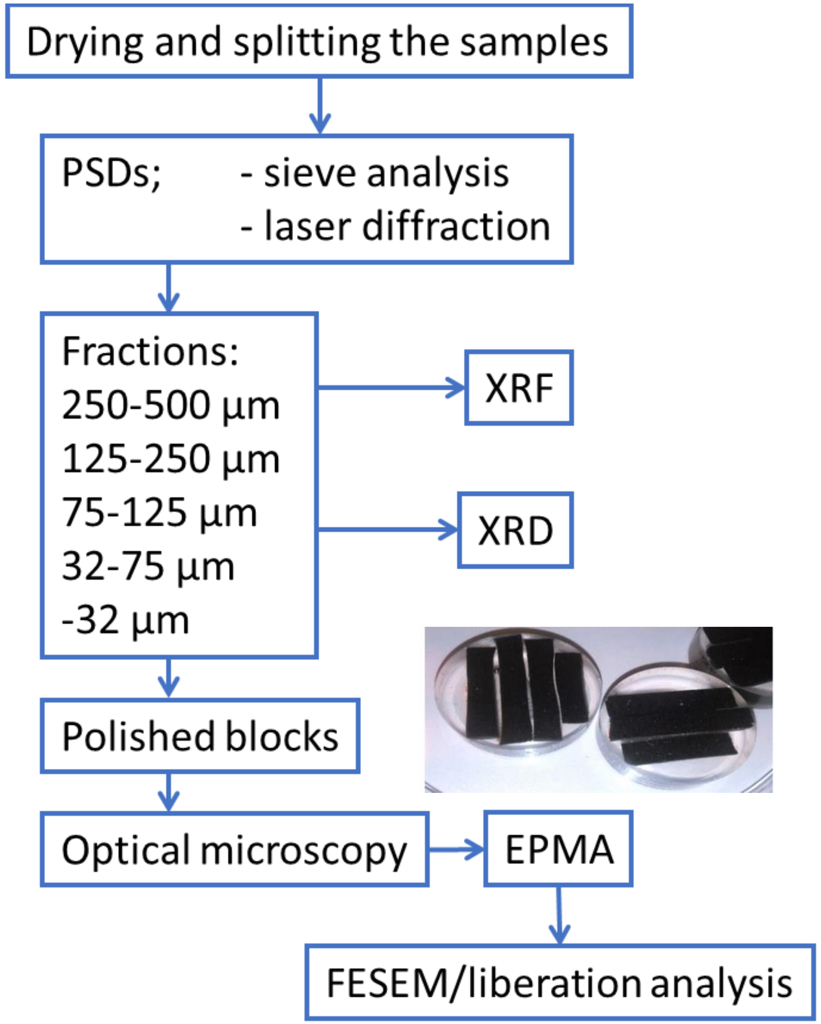
Liberation degree vs. recovery





Typical particles of valuable (dark phase) and gangue (light phase) shown in cross-section			
By flotation	✔ Recovered	Not recovered	Not recovered
By density separation	✔ Recovered	✔ Recovered	✔ Recovered
By leaching	✔ Recovered	Not recovered	Not recovered

The beginning for liberation analysis



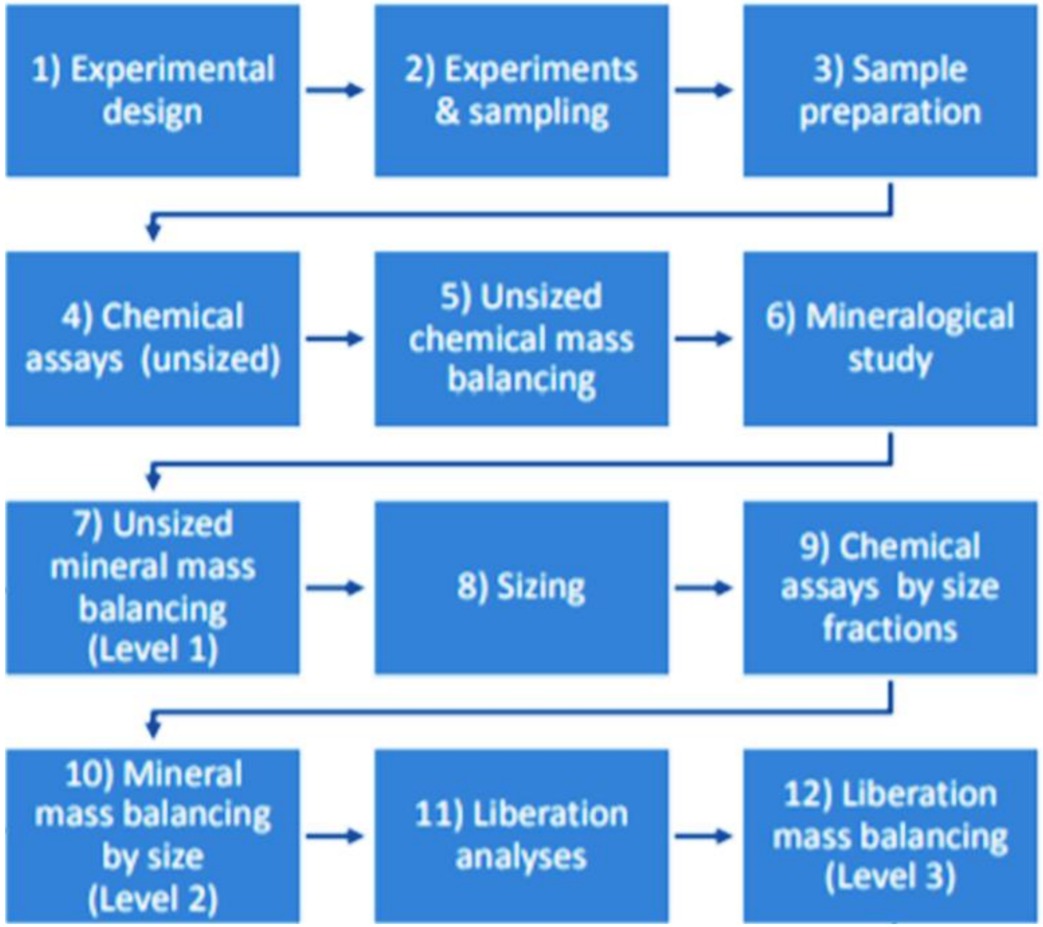


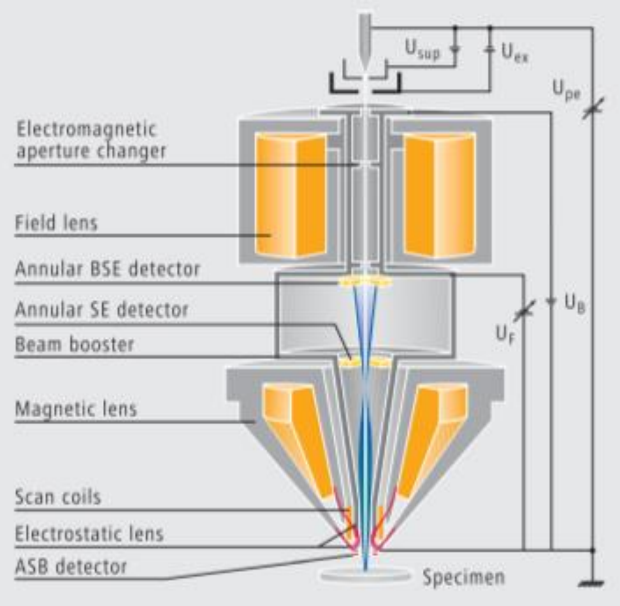
Image modified from: "A way forwards in process mineralogy – using automated mineralogy for modelling and simulating beneficiation processes" P. Lamberg

Instrumentation

Image from: https://microscopy.anu.edu.au/zeiss-ultraplus-analytical-fesem

- The platform is SEM hardware.
- A backscatter electron (BSE) detector is standard with SEM equipment.
- One to four EDX spectroscopy detectors are mounted on the SEM. Most digital imaging analysis systems now use much faster liquid nitrogen–free Si drift detectors (SDDs).
- User friendly operating and processing software with simplified or ready-made data outputs provide information on mineral speciation, composition, liberation, association, and size distribution.

Principle



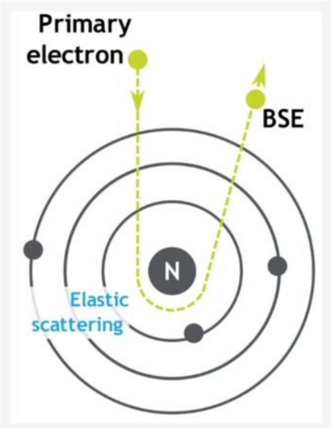


Image from thermofischer.com

Backscattered electrons, which are elastically scattered electrons with energies close to the primary electron beam energy generated from tens of nanometers at the surface, are used for imaging compositional variations between phases. Minerals composed of heavier elements (e.g., zircon) backscatter more of the incident electrons of the SEM and appear brighter in the BSE image, whereas minerals composed of lighter elements (e.g., quartz) backscatter fewer electrons and appear darker. In other words, BSEs are high-energy electrons used to obtain high-resolution images that show the distribution of various elements that make up a sample.

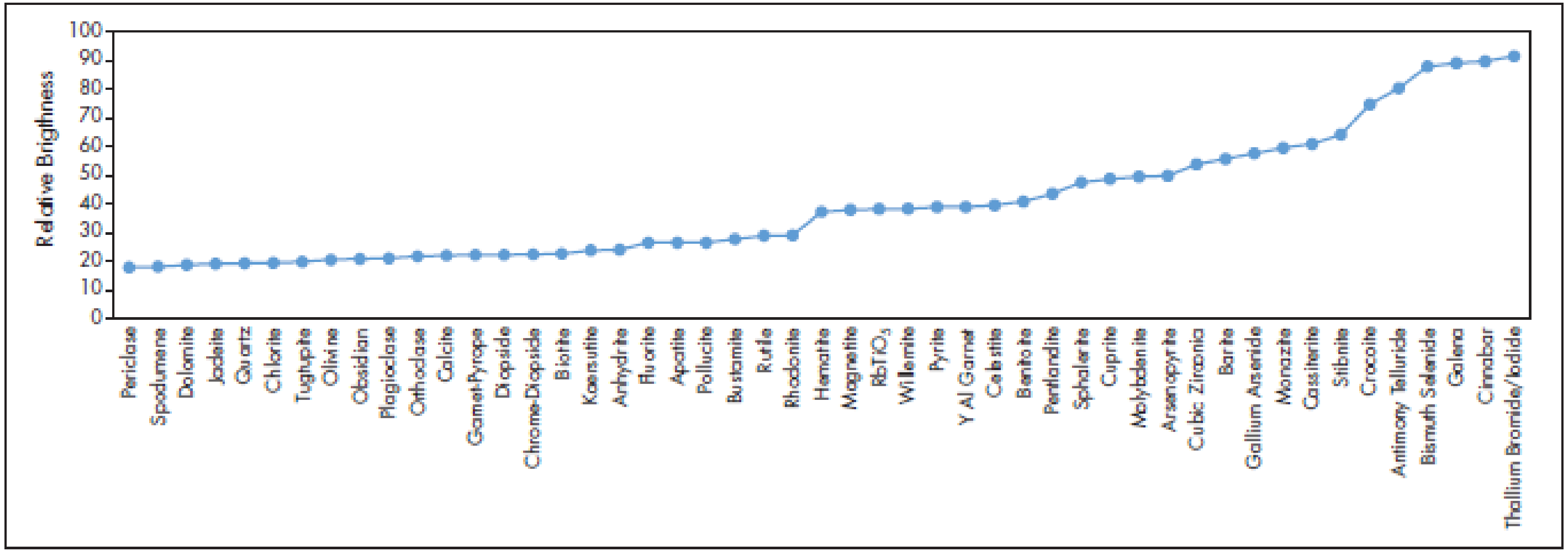


Figure 6 Relative backscatter electron intensity comparison

Image from Kawatra & Young

Principle

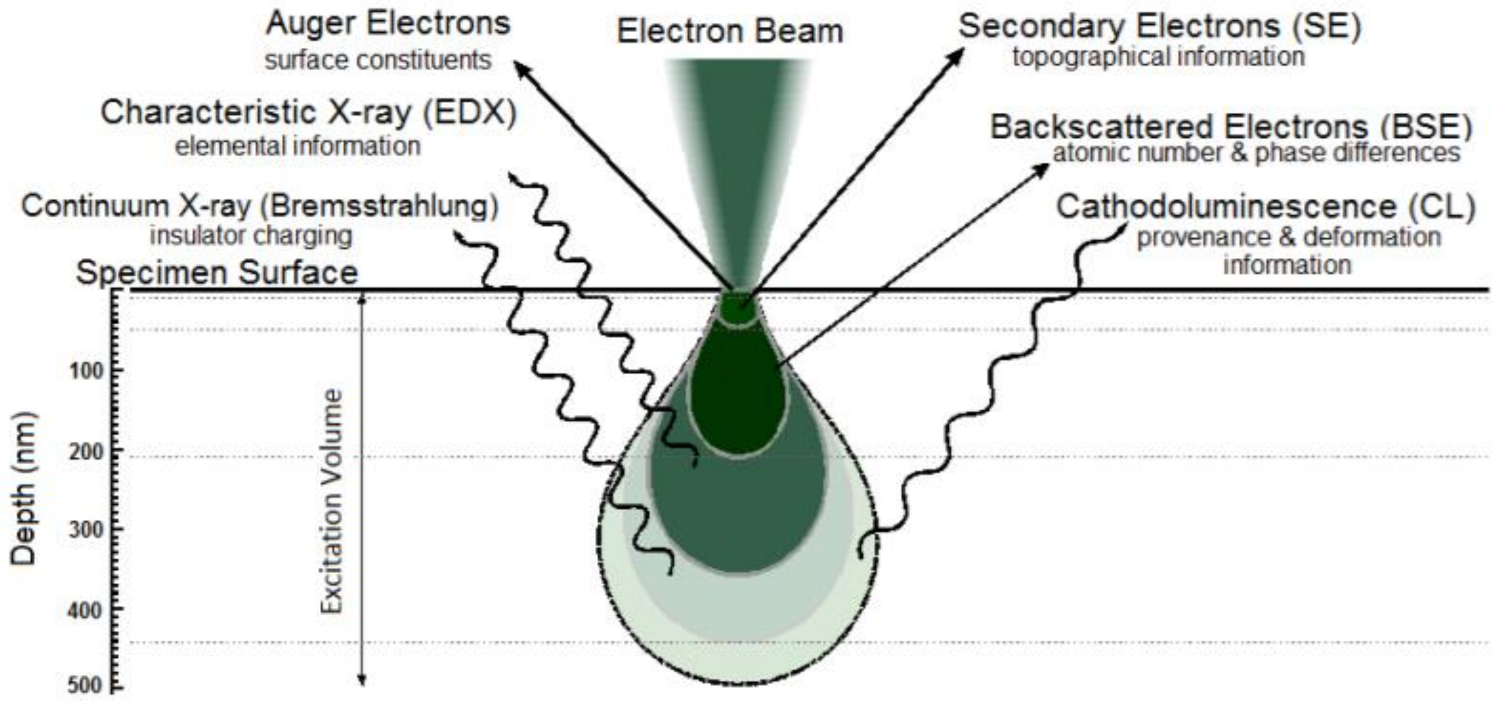
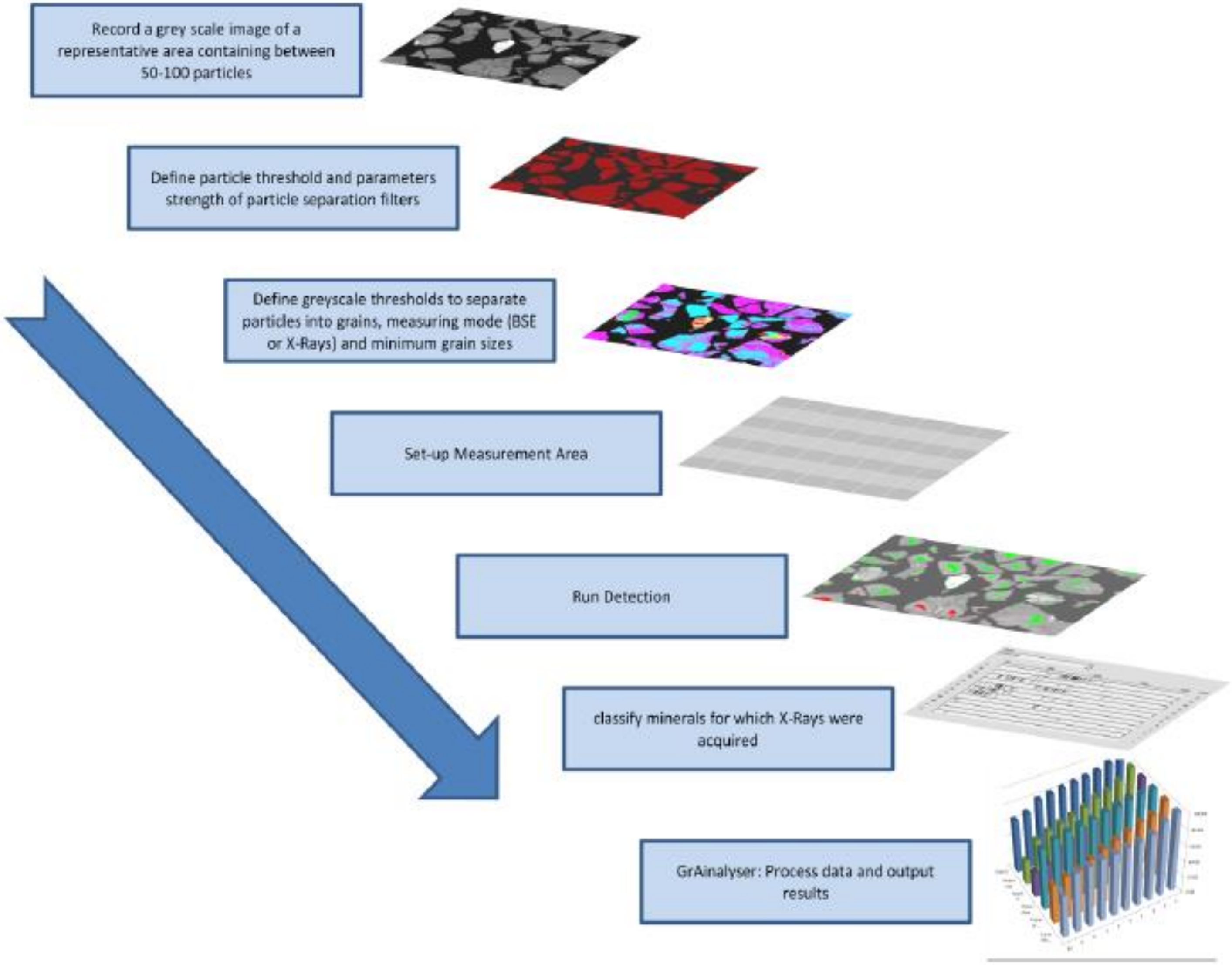


Image from: Rodriguez et al. 2014

INCAMineral workflow



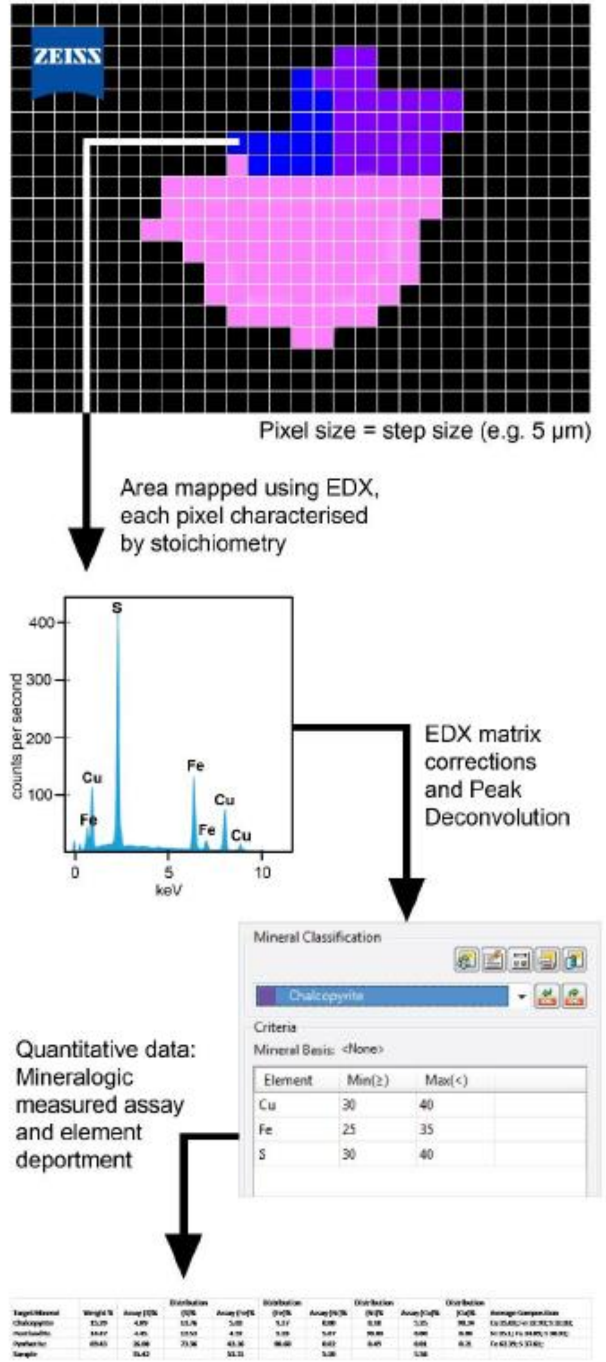


Image from Graham, 2017

Setting up particle separation parameters

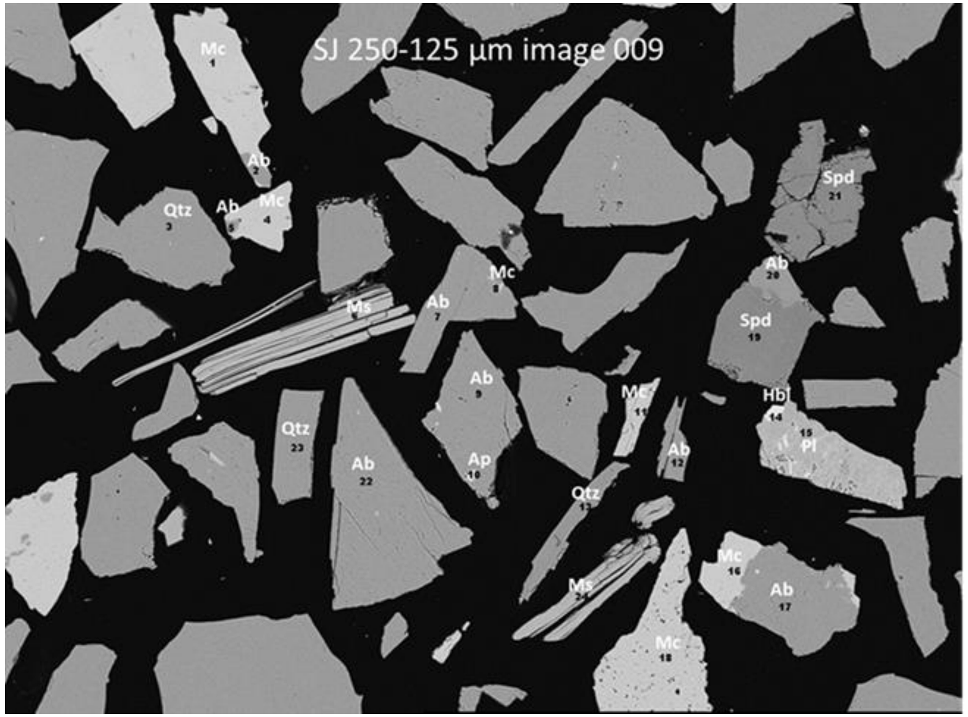
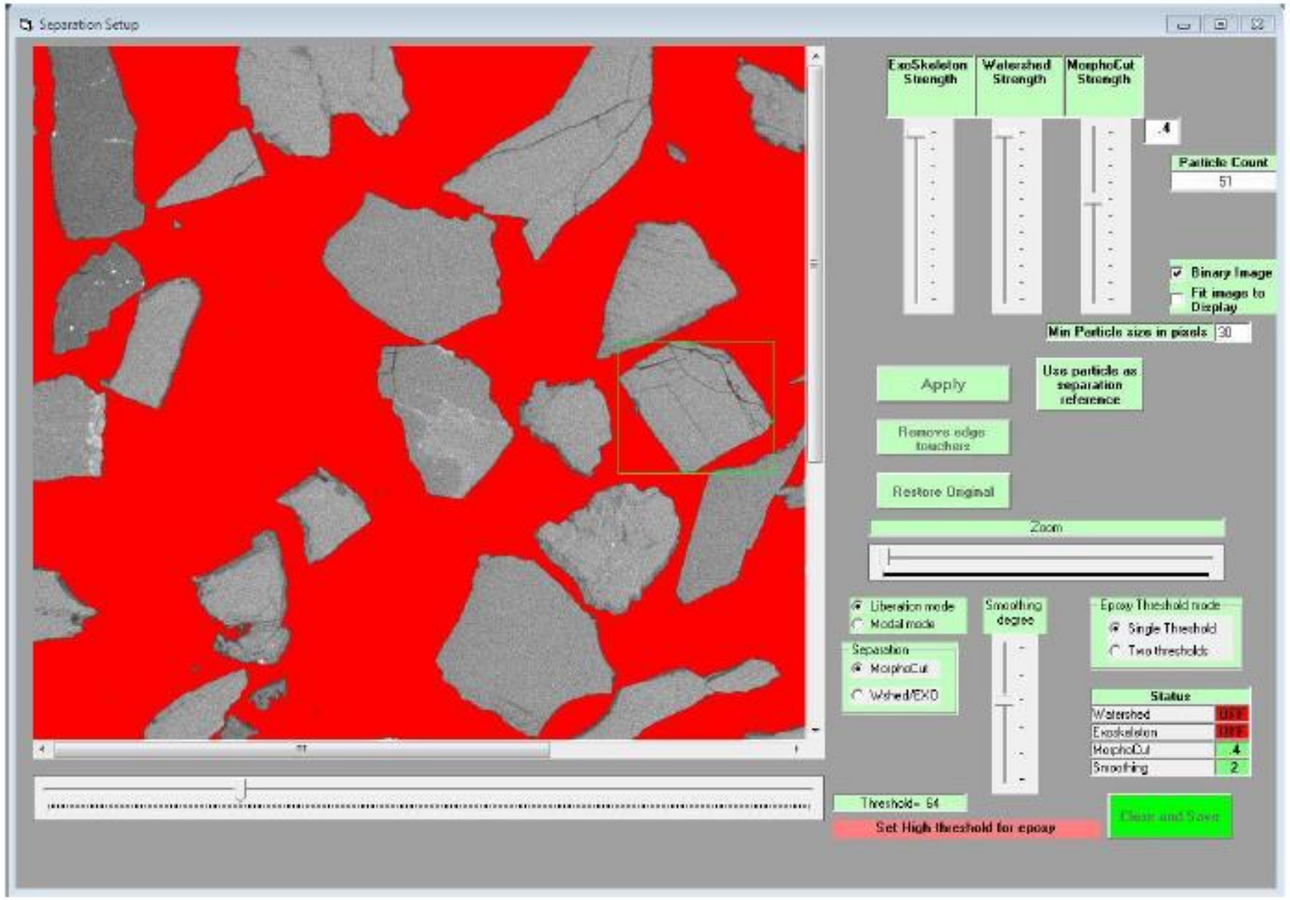


Image R. Kallio



Work in progress

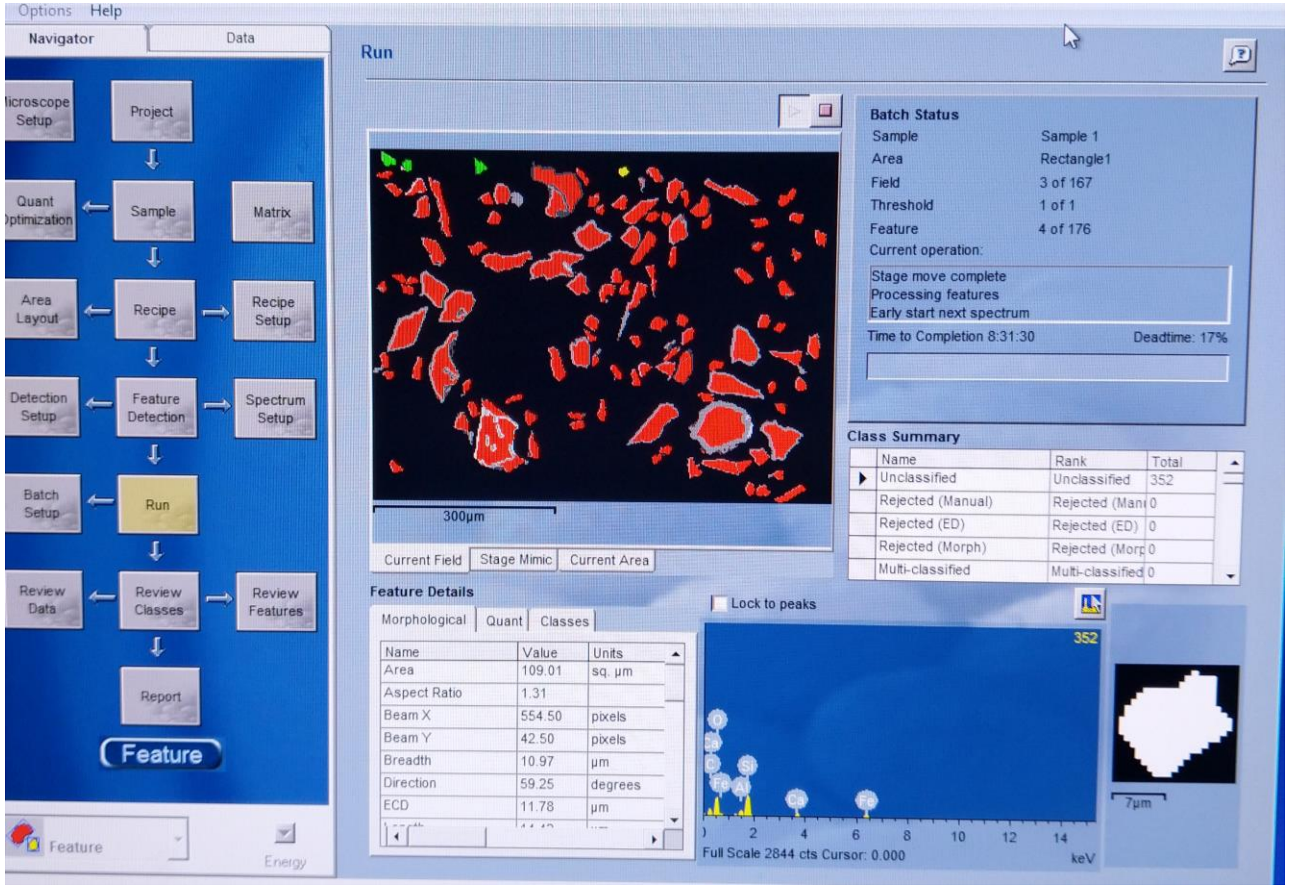
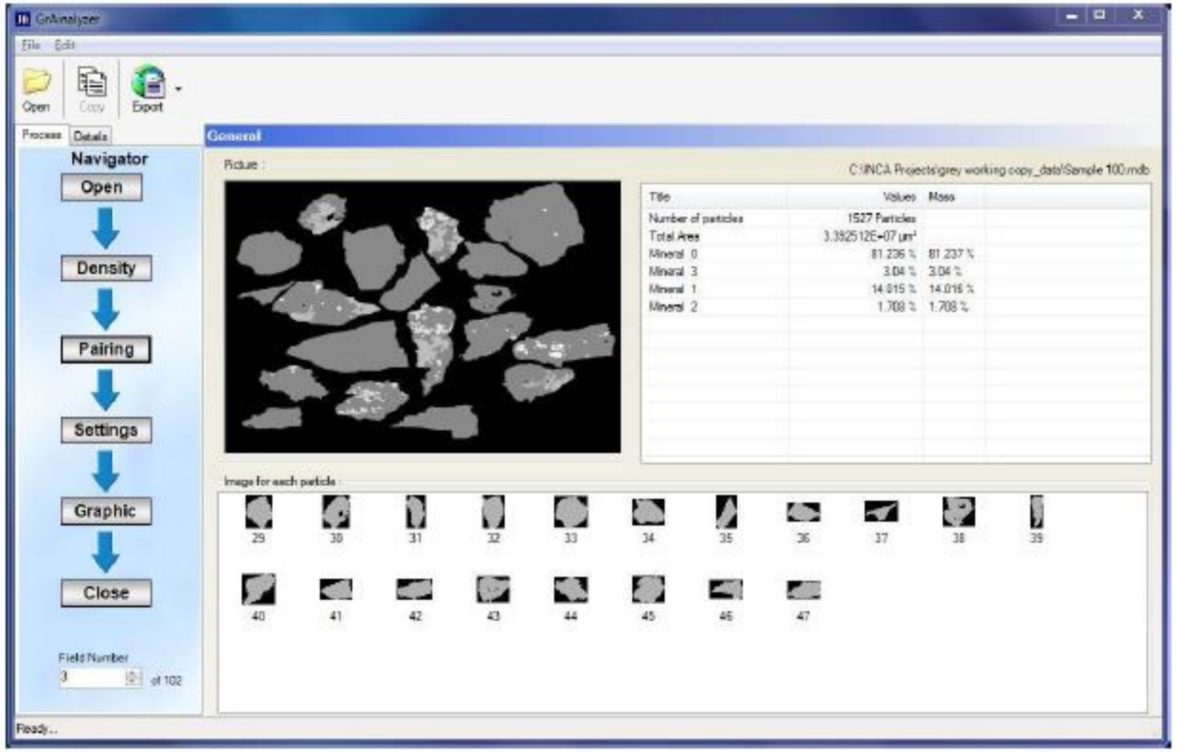
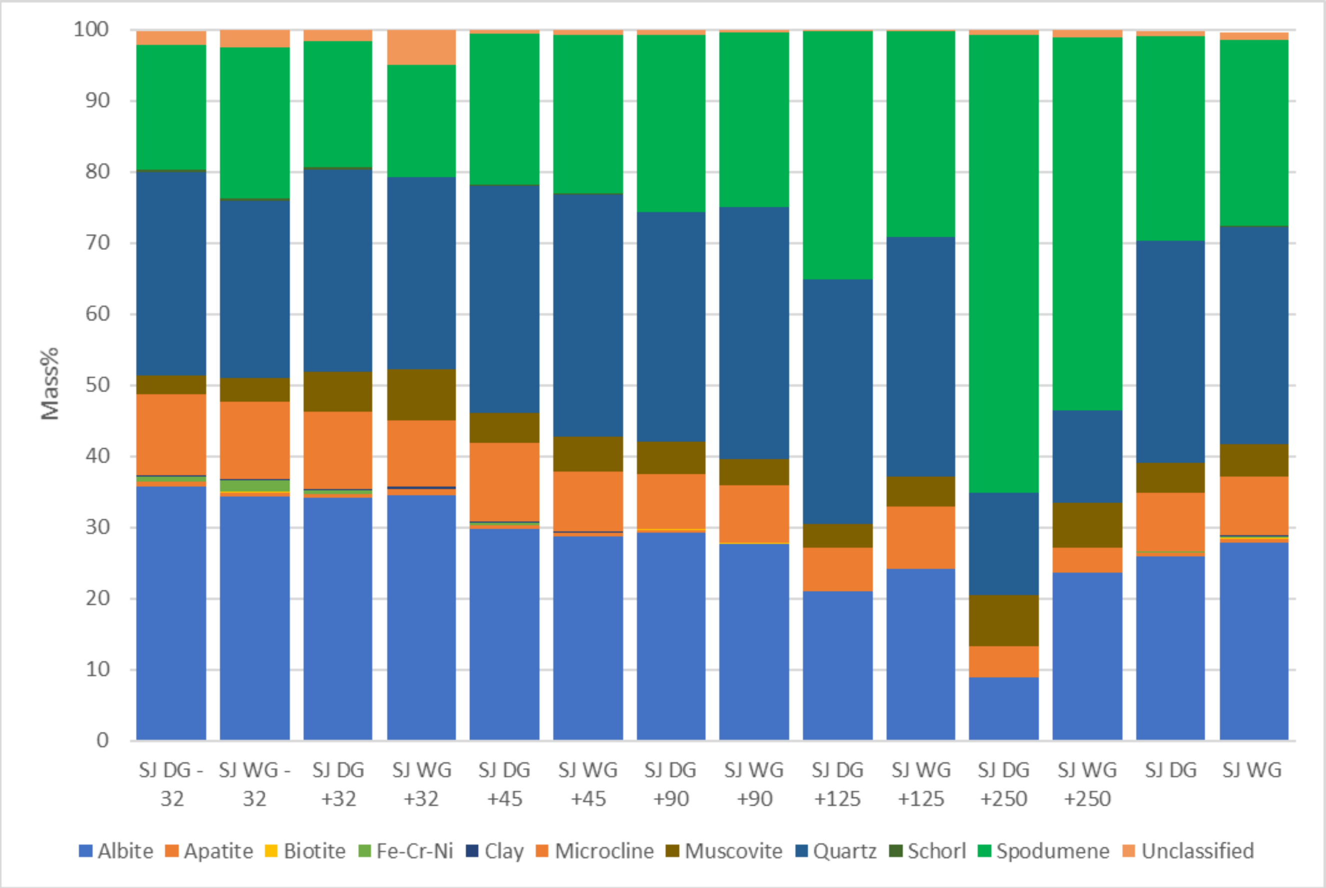


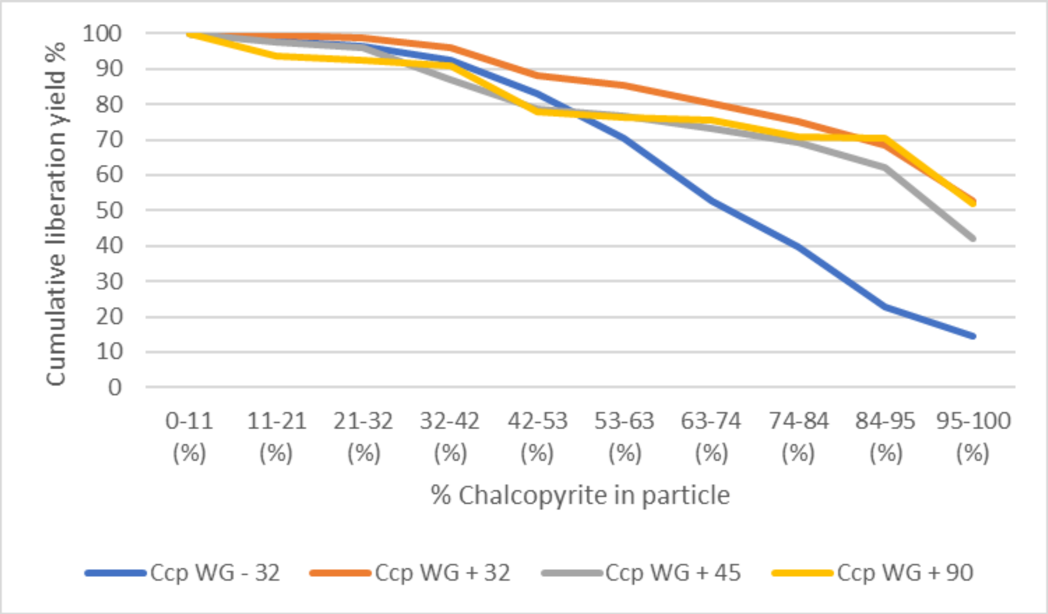
Image R. Kallio

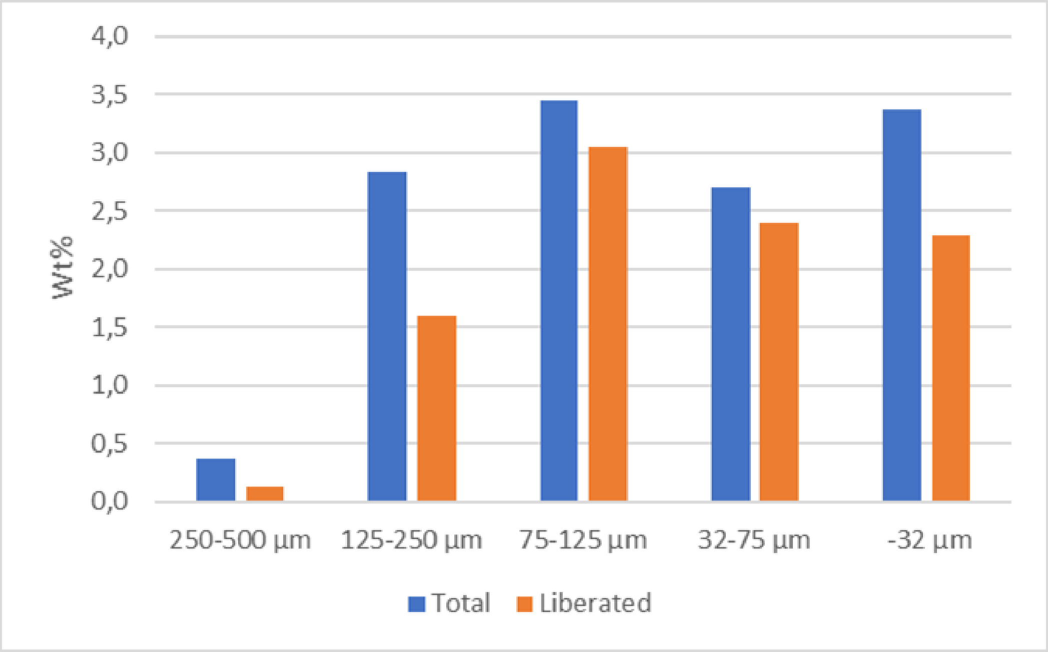


The results

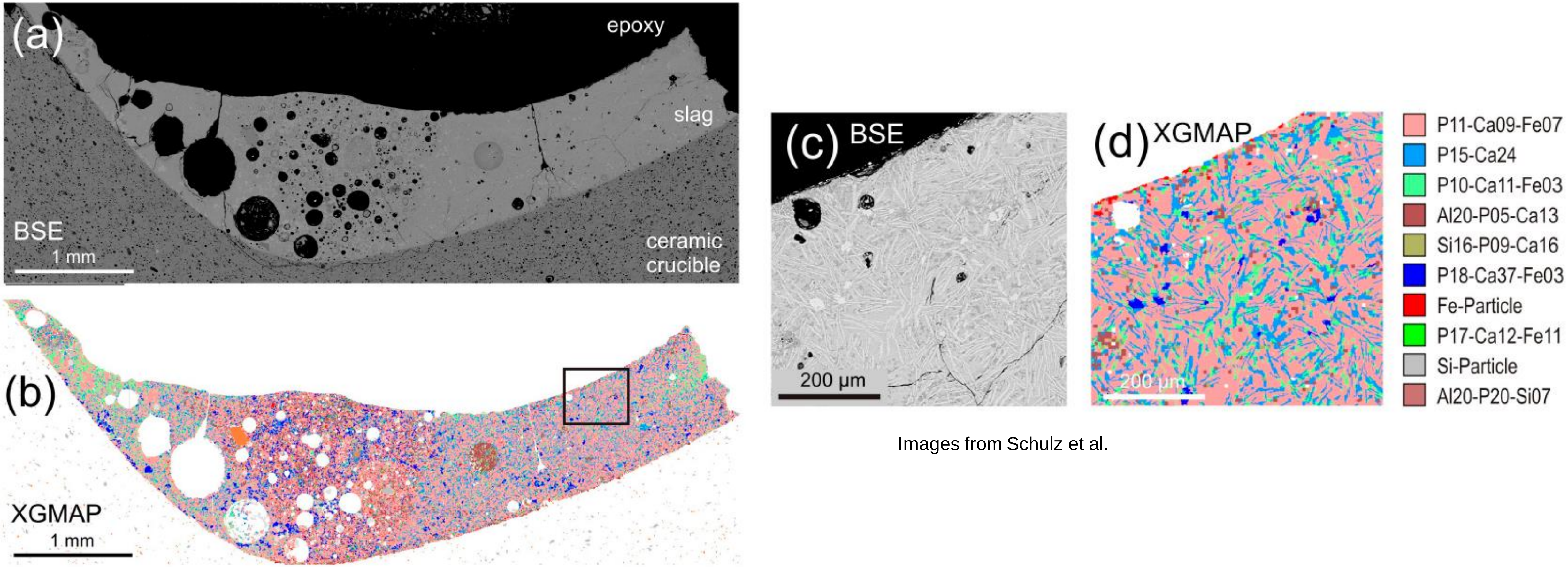


All images R. Kallio



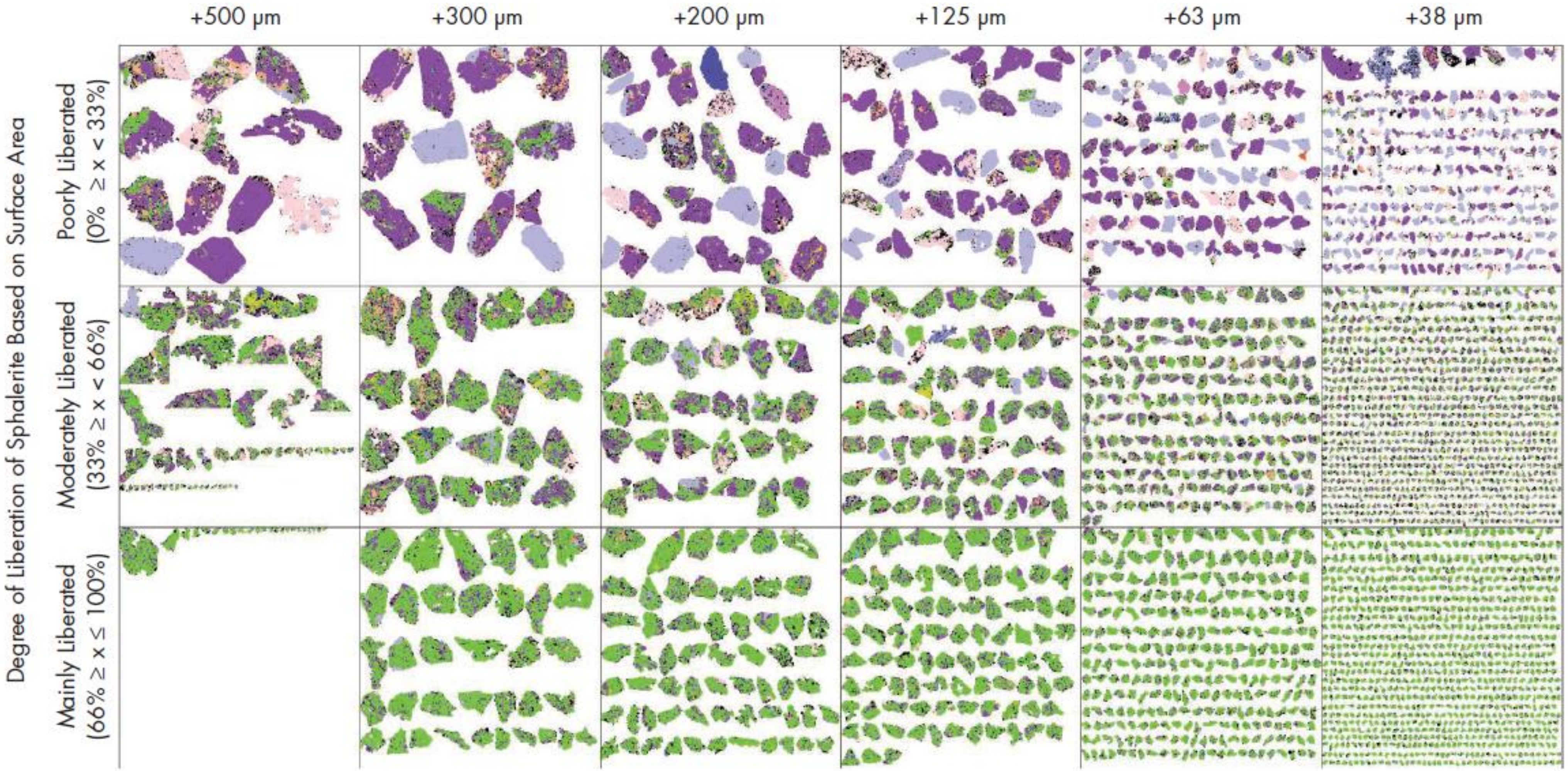


Other examples I



Images from Schulz et al.

Other examples II



C. Degree of liberation of sphalerite represented graphically in different size fractions

Image from Kawatra & Young

Other examples III

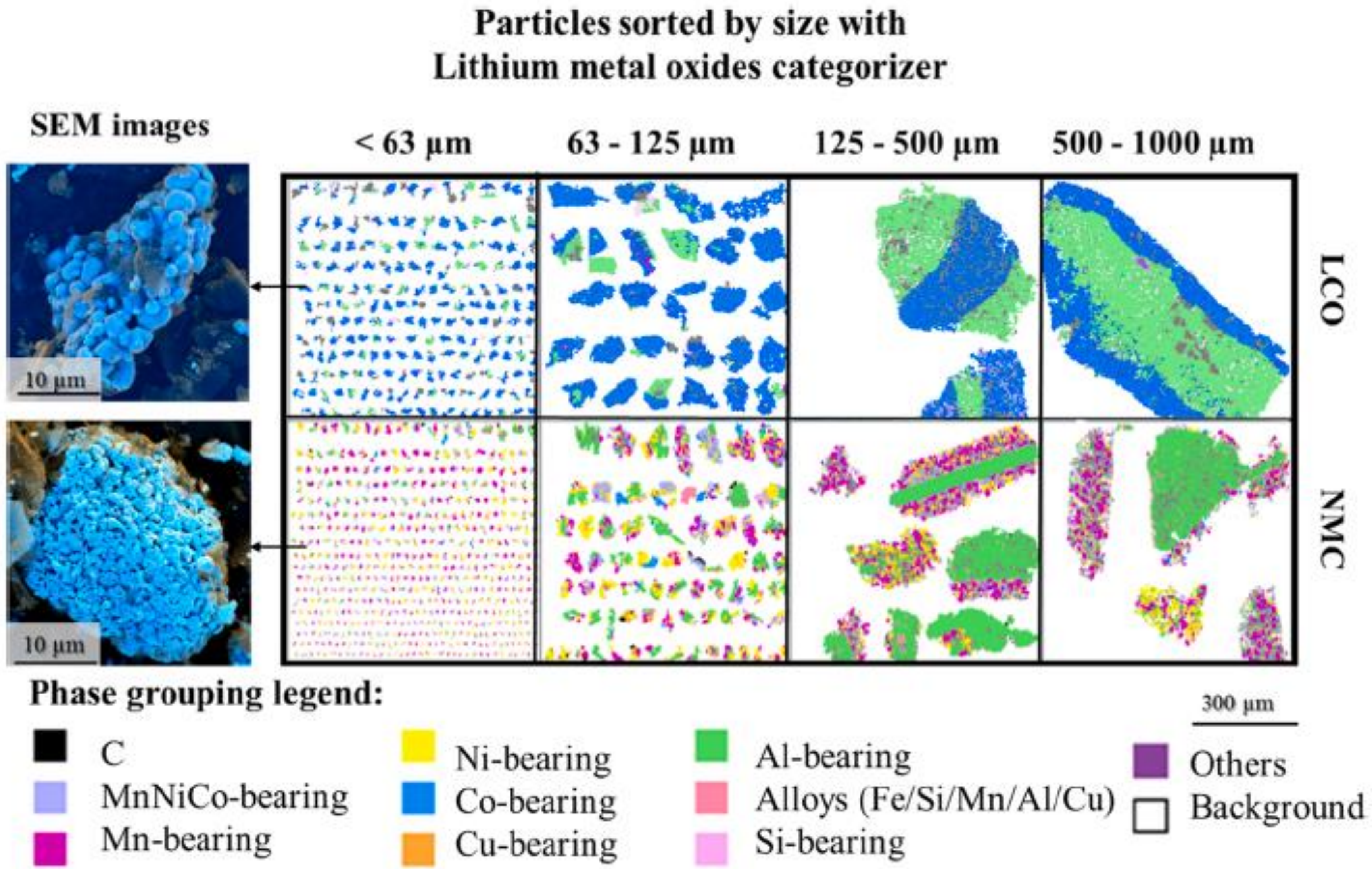


Image from Vanderbruggen et al.

Sources of error and limitations

- The number of coarse particles (> 500 μm) that can be mounted in a polished section are low and can lead to issues associated with statistical analysis representative of the size population.
- Very fine particles <~15 μm pose practical problems for mounting because of possible bias caused by particle loss during sieving and polishing and tend to agglomerate during the sample preparation. Generally, very fine-particle sizes are not measured and rely on other techniques such as XRD to measure the mineral composition data or can be inferred to some extent from data at a coarser fraction size.
- Variation occurs in the specific gravity of mineral and phase components within a sample. Samples require careful sample preparation to prevent segregation of heavy particles from lighter gangue occurring within the section yielding a biased distribution. The process of double mounting to get representative samples can be used.
- The measurements of the size and shape of elongated particles can be biased by variable orientations.
- For studies of minor to trace heavy minerals in rock or metallurgical samples, the sample should be concentrated to increase the number of heavy mineral grains available prior to mounting and analysis. In particular, the dense nature and low concentration of gold found in ores requires careful sample preparation to monitor.

Sources of error and limitations

For liberation analysis, it is important to remove agglomerated particles. Agglomerated particles affect the results of mineral liberation assessment, because they are considered as one particle. Samples of dry flotation concentrates commonly have agglomerated particles. Samples can be subjected to a strong attrition treatment process (e.g., ultrasonic bath) to remove the agglomerated material prior to mounting. Alternatively, many automated software systems have particle separation routines that set boundaries between touching particles within agglomerates.

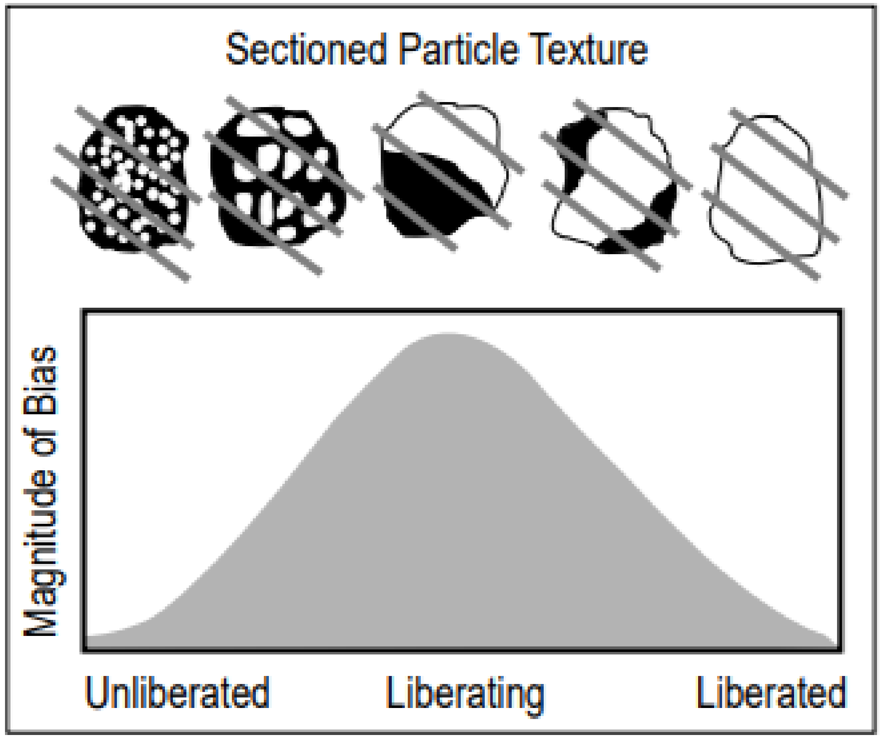
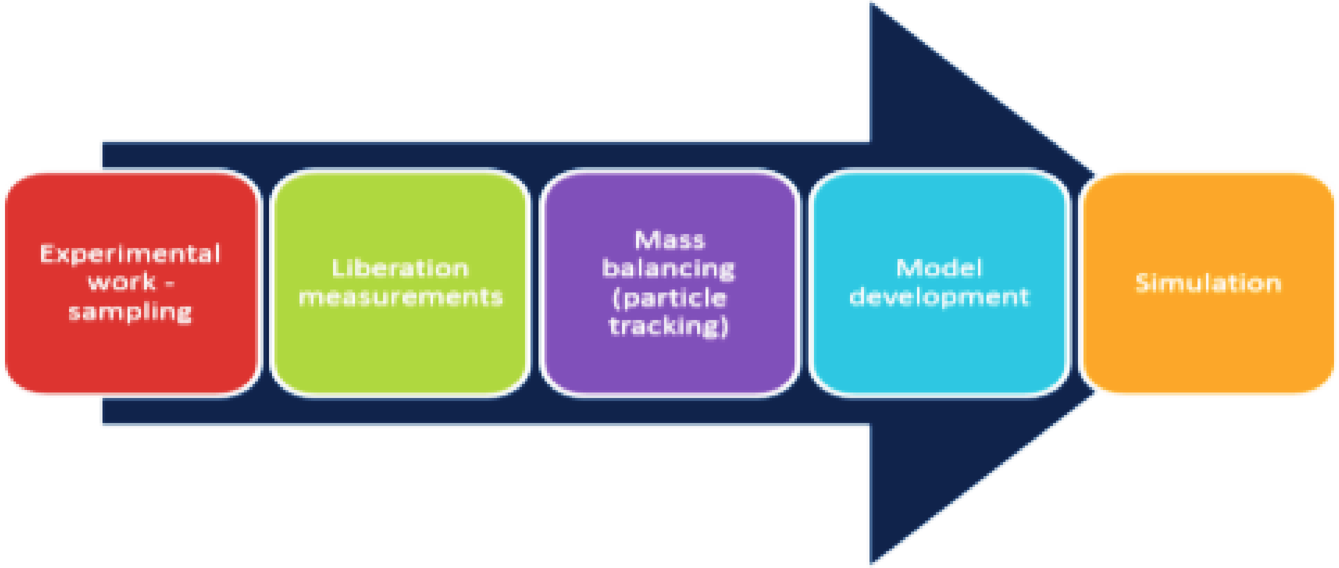


Figure 1. A schematic of the basis of stereological error, with sections (gray lines) through liberated and composite particles of varying texture and the corresponding magnitude of bias.

The future prospects for automated mineralogy/application of liberation data



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