Automated particle analysis of aerosols formed during biomass combustion by SEM/EDX
Overview

- General information about SEM
- Fundamentals of particle analysis
- Principles of the automated particle analysis (APA)
- APA of aerosols from biomass plants
The Scanning electron microscope

Primary electrons (up to 30keV)

- Secondary electrons (SE)
- Backscattered electrons (BSE)
- Characteristic x-rays (EDXS)
- Augerelectrons (AE)
- Cathodoluminescence (CL)

Electron-specimen interaction and generated signals

Specimen current
Monte Carlo simulations of electron scattering of an 100 nm particle of PbCl$_2$:
left $E_0 = 20$ keV and right $E_0 = 7$ keV
Experimental

- pure chemicals ($\text{K}_2\text{SO}_4$, $\text{PbSO}_4$ and $\text{PbCl}_2$) were ground in a ball mill
- suspended in pure sec-butanol
- filtered through a 3 µm Nucleopore filter
- the filtrate was filtered through a 0.05 µm (C-coated) Isopore poly-carbonate filter

1 µm

top: in-lens image (SEI);
bottom: BSE image (BSE)
$\text{PbSO}_4$-particles
The Scanning electron microscope

Instrumentation:
- Zeiss DSM 982 Gemini
- Noran Voyager EDX-detector, 30 mm² Si(Li), ultrathin window
Size measurement

Influence of the coating of the particles on the measured particle size
Size measurement

yellow: SEI-signal
green: BSE-signal
particle: PbCl₂
no coating
acc. voltage: 7 keV

yellow: SI-signal
green: BSE-signal
particle: PbCl₂
carbon coating
acc. voltage: 7 keV
Size measurement

Influence of mean atomic number of the particles on the measured particle size
Size measurement

![Graph showing size measurement with average diameter in microns and frequency as y-axis. Lines represent SEI-uncoated, average SEI-BSE C-coated, and SEI-C-coated.]
Chemical composition

X-ray analysis of biomass fly ash particle ($E_0 = 7$ keV, $I_p = 0.7$ nA);

a) image of particles with the analysis spot marked by a cross (SE – image)
b) x-ray spectra
K$_2$SO$_4$ particles: measured ratio of K (at%) / S (at%), in dependence on both the type of the substrate and the coating (spot analysis, $E_0 = 7$ keV, $I_p = 0.7$ nA; spectra at 10s and 60s stored without any interruption; mean particle diameter: 400 nm)
Chemical composition

Automated analysis of standard particles: $\text{K}_2\text{SO}_4$, $\text{PbSO}_4$ and $\text{PbCl}_2$
Principles of Automated Particle Analysis

SEI-image

BSE image

binary images

image width 3.8 µm
Scheme auf the automated particle analysis procedure
Automated Particle Analysis of aerosols

waste wood  
bark

Size distribution of aerosols (fuel: waste wood, bark)
Automated Particle Analysis of aerosols

Concentrations of Pb and K in dependence on the diameter of the aerosols (fuel: waste wood, bark)
Automated Particle Analysis of aerosols

Correlations between various elements for aerosols (fuel: waste wood, bark)
Automated Particle Analysis of aerosols

Correlations between various elements for aerosols (fuel: waste wood, bark)
Automated Particle Analysis of aerosols

SE-image (image width: 0.29 µm) of an aerosol particle (left) and the EDXS – spectra (right) at the 2 spots marked in the image.
If only the particle size is of interest, uncoated particles and the use of the in-lens detector is recommended.

In case of carbon coated particles the average of the sizes obtained from images of the in-lens and the BSE-detector have to be used.

Automated analysis of aerosols with SEM/EDXS is possible down to particle diameters of about 50 -100 nm depending on the chemical composition.
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