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

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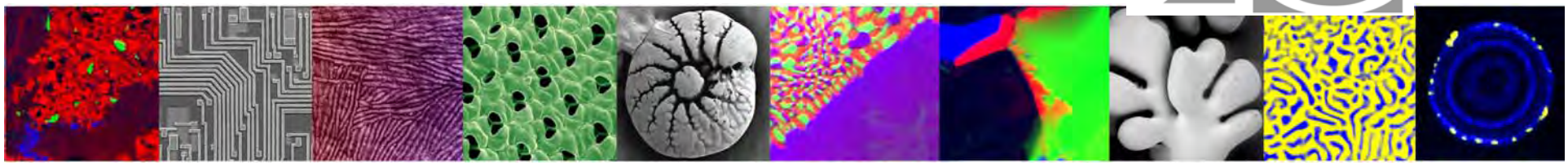
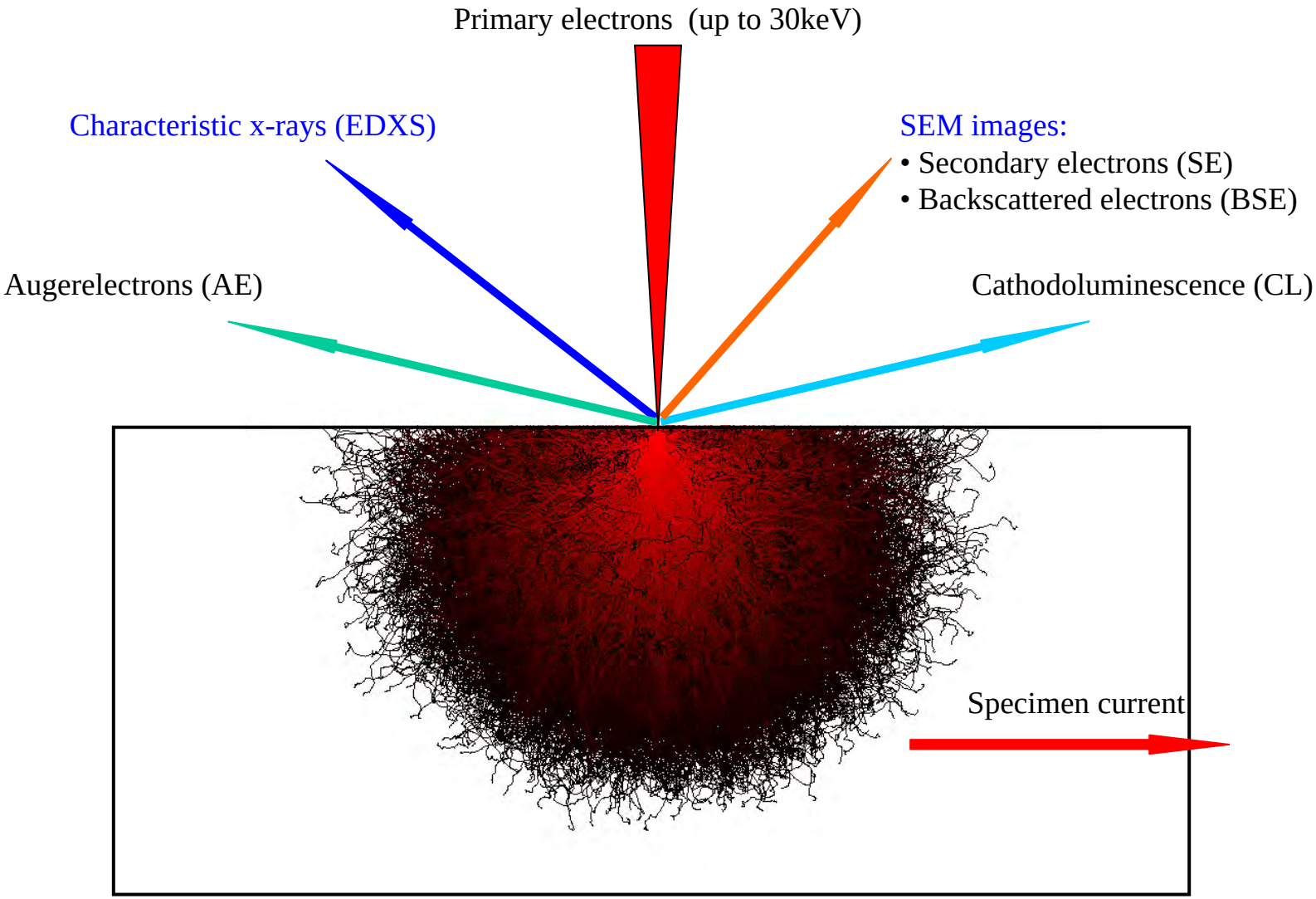


Overview

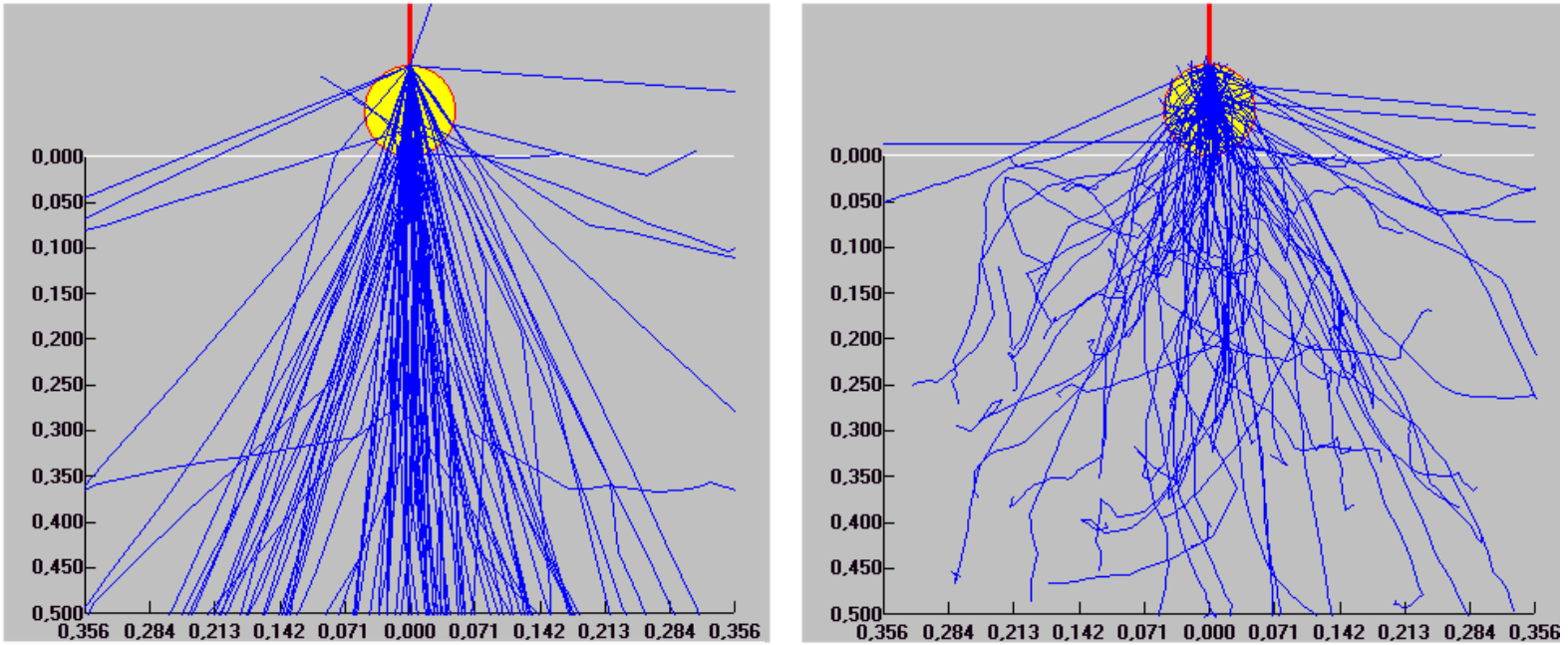
- General information about SEM
- Fundamentals of particle analysis
- Principles of the automated particle analysis (APA)
- APA of aerosols from biomass plants

The Scanning electron microscope



Electron-specimen interaction and generated signals

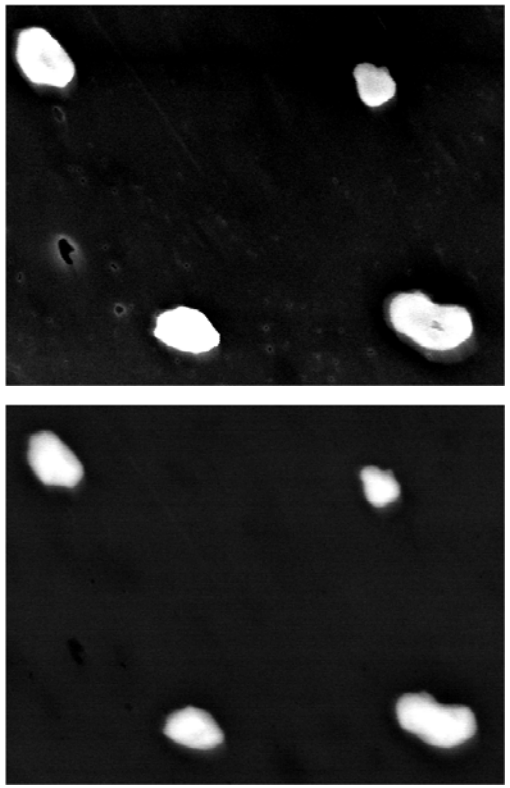
The Scanning electron microscope



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

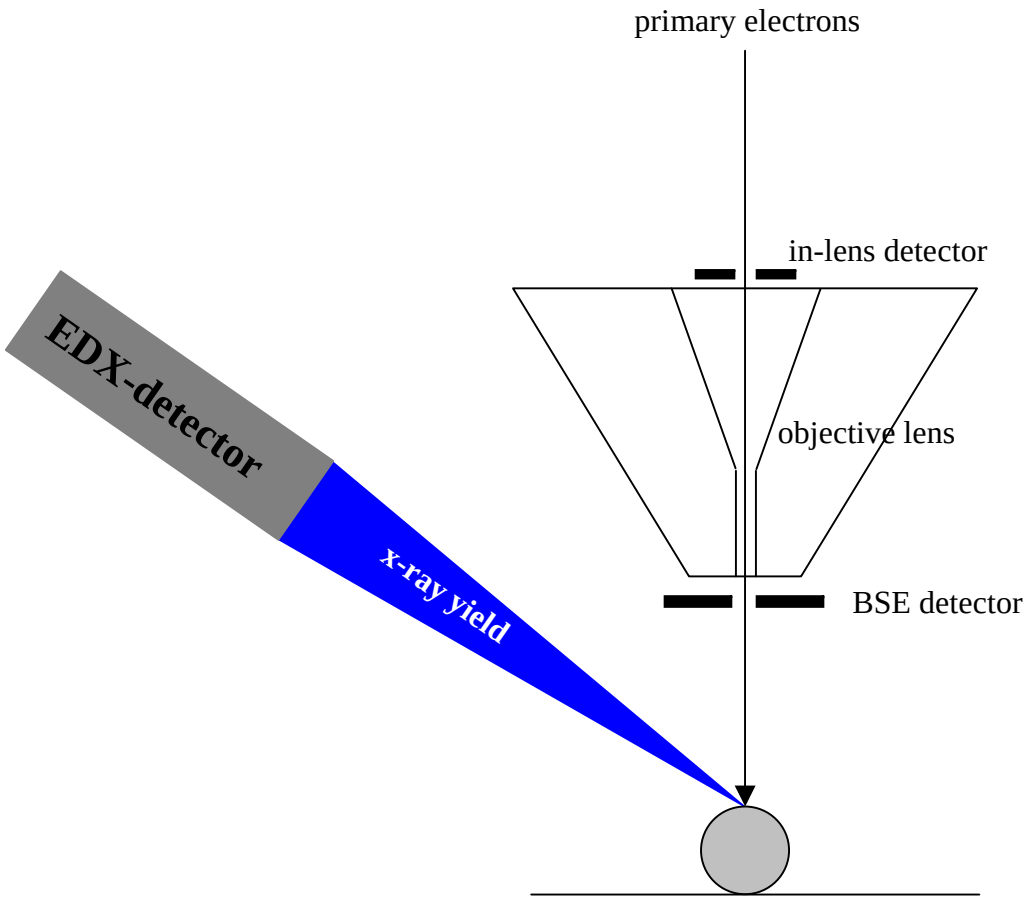
Experimental

- pure chemicals (K_2SO_4, $PbSO_4$ and $PbCl_2$) 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



top: in-lens image (SEI);
bottom: BSE image (BSE)
$PbSO_4$-particles

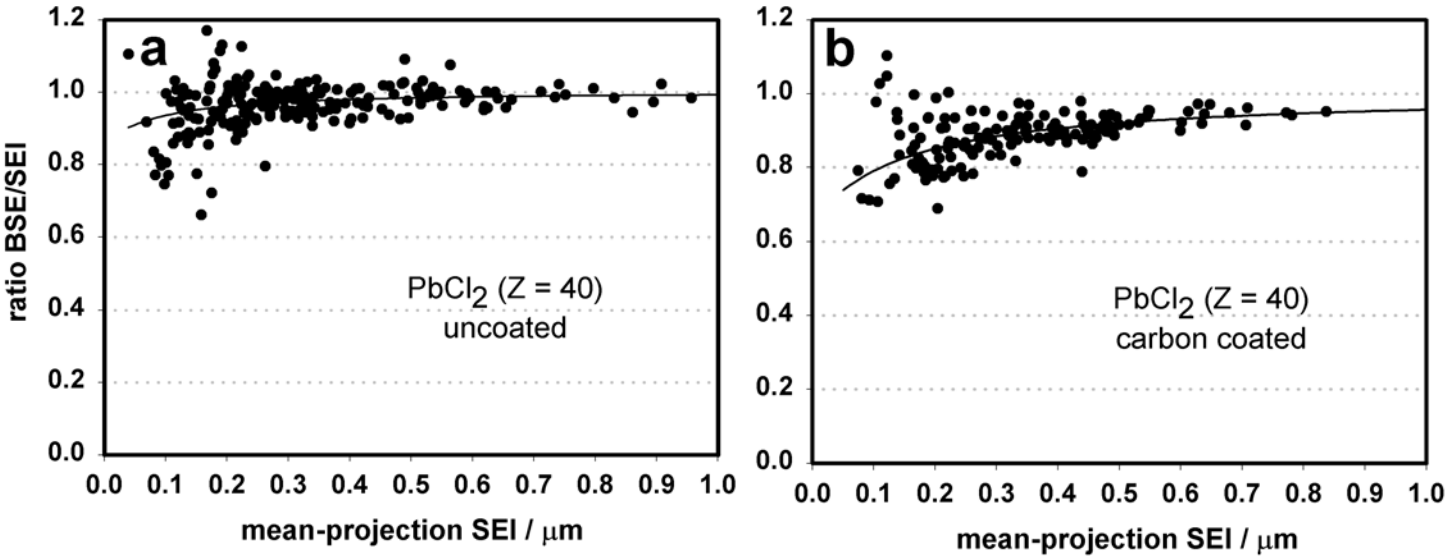
The Scanning electron microscope



Instrumentation:

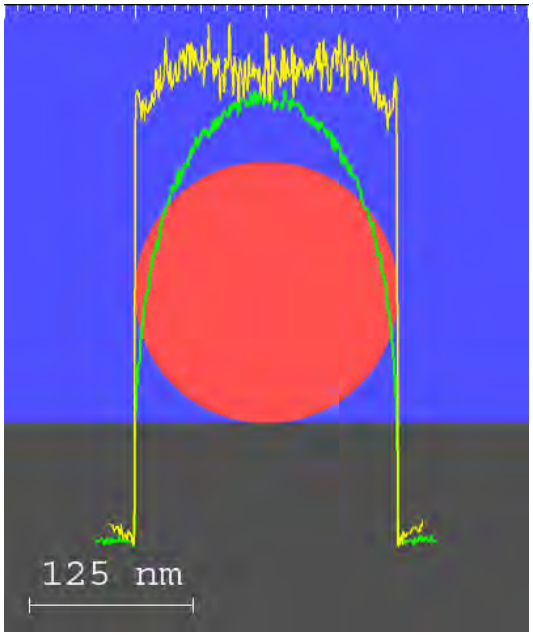
- Zeiss DSM 982 Gemini
- Noran Voyager EDX-detector, 30 mm^2 Si(Li), ultrathin window

Size measurement

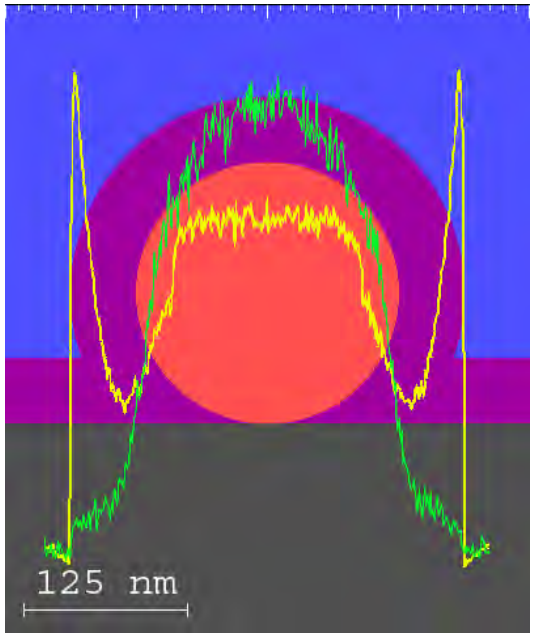


Influence of the coating of the particles on the measured particle size

Size measurement

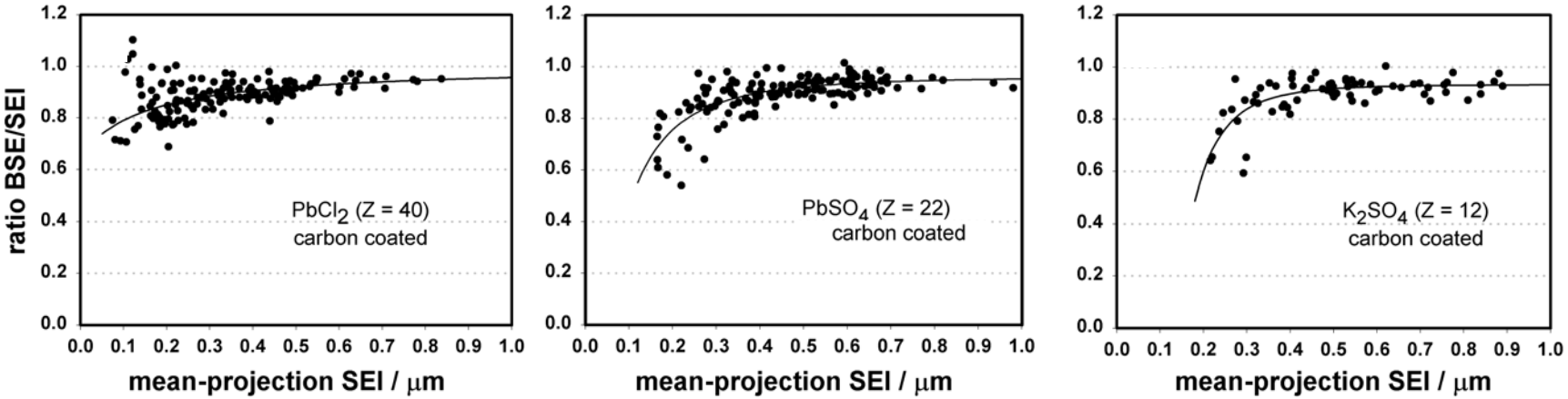


yellow: SEI-signal
green: BSE-signal
particle: $PbCl_2$
no coating
acc. voltage: 7 keV



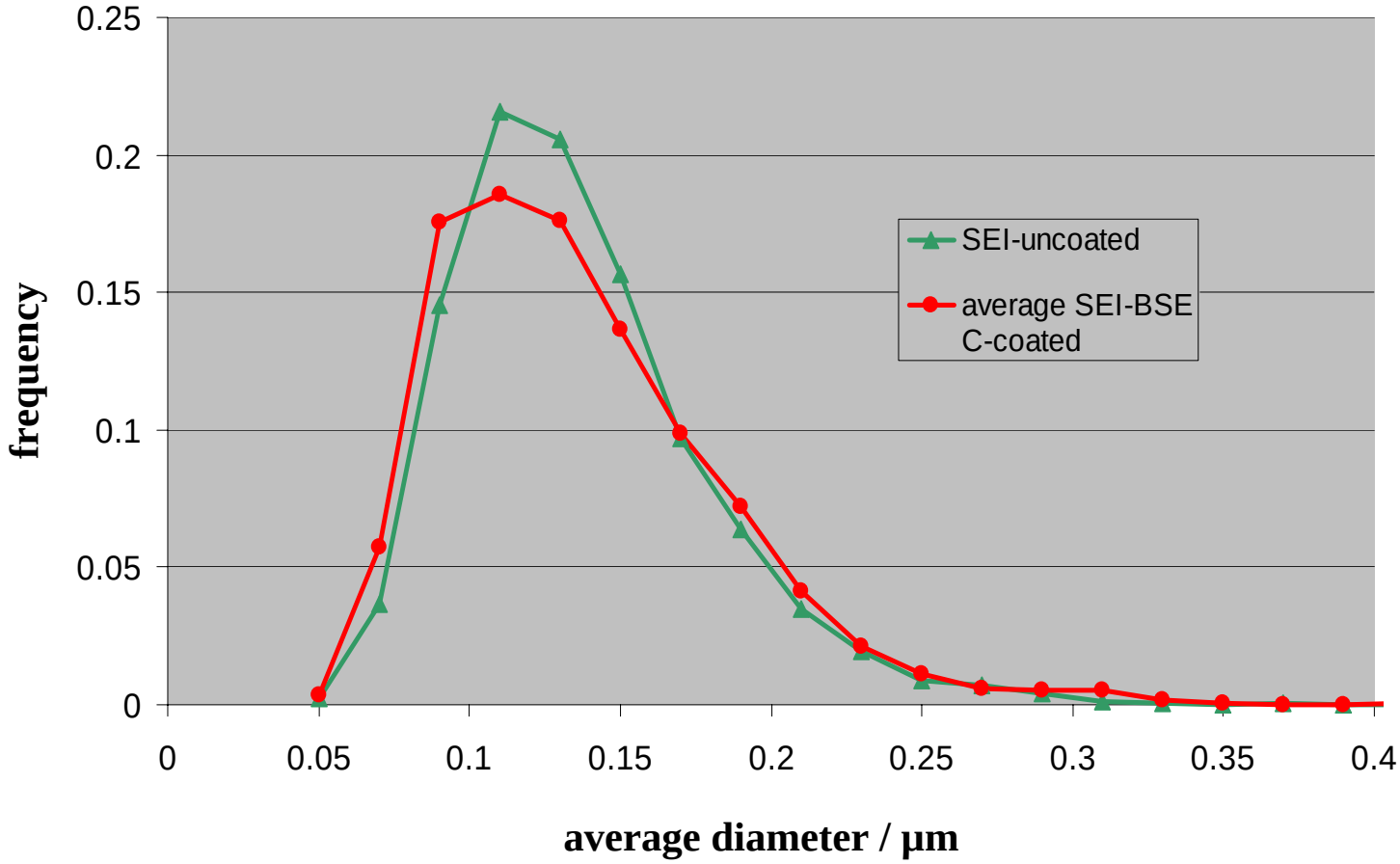
yellow: SI-signal
green: BSE-signal
particle: $PbCl_2$
carbon coating
acc. voltage: 7 keV

Size measurement

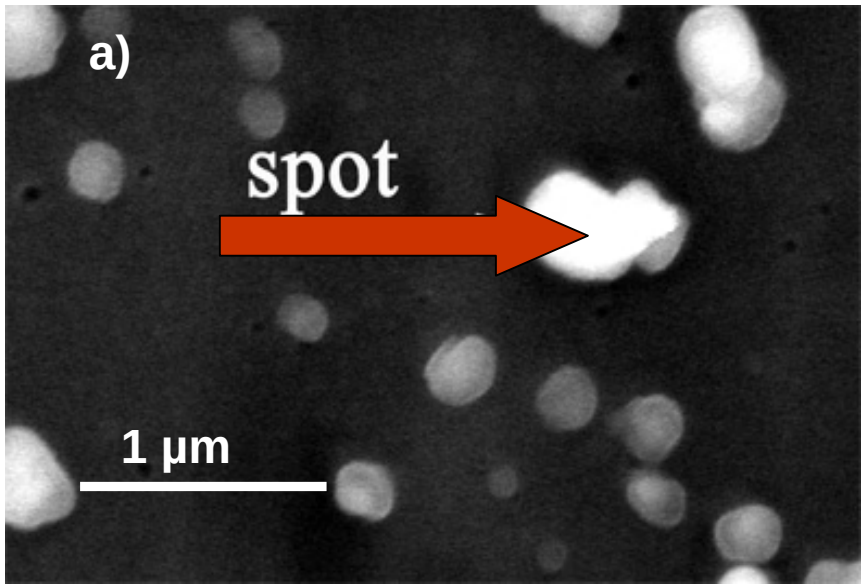


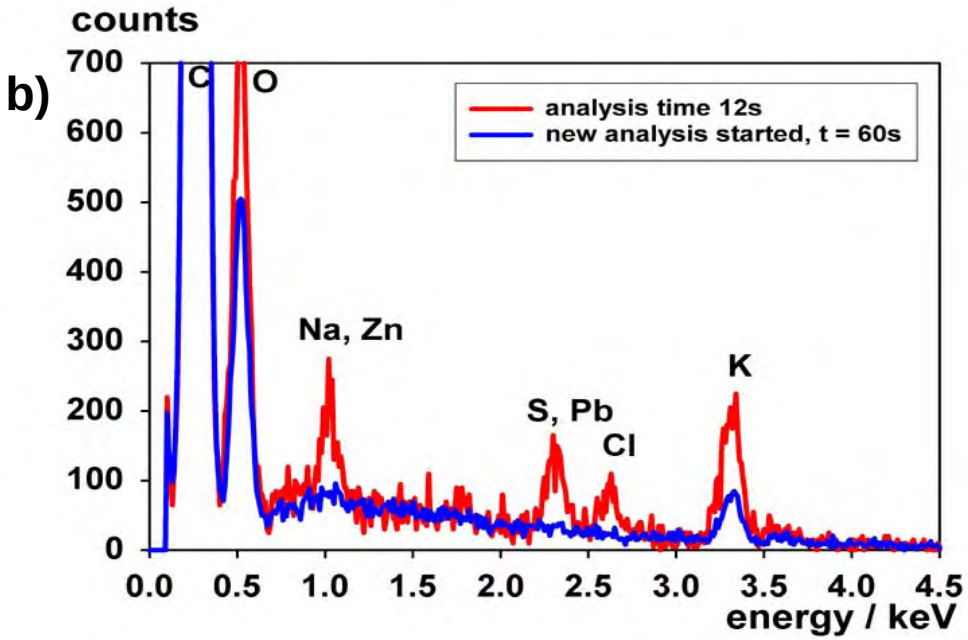
Influence of mean atomic number of the particles on the measured particle size

Size measurement



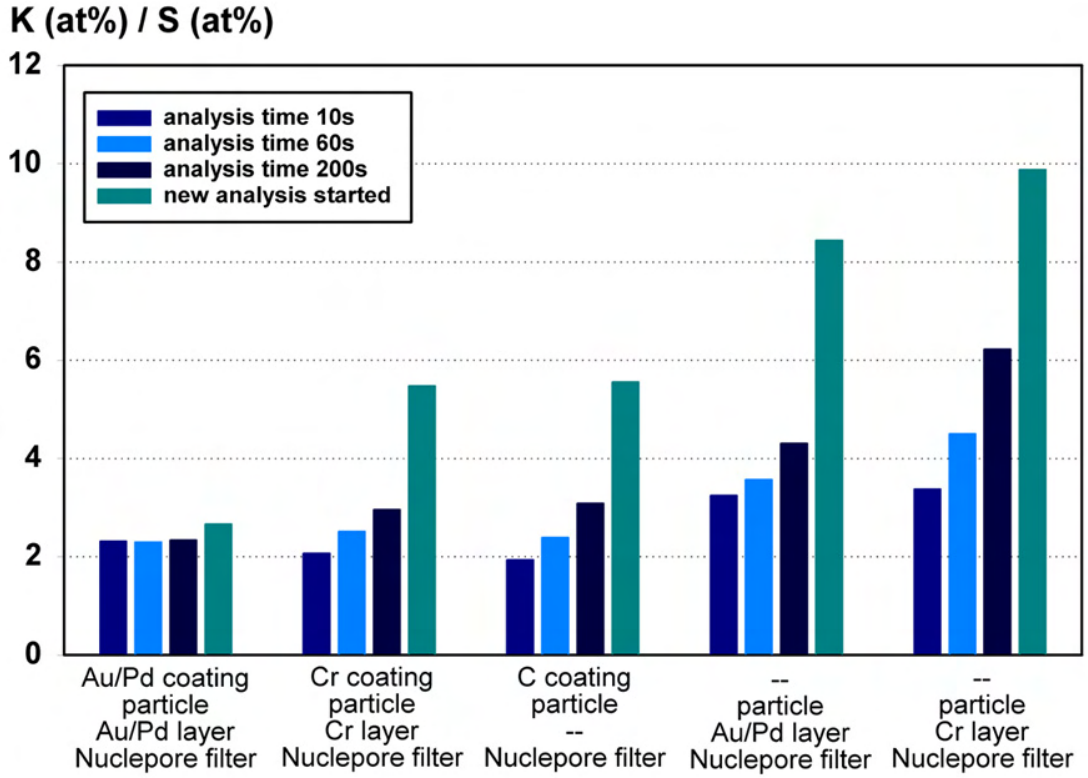
Chemical composition





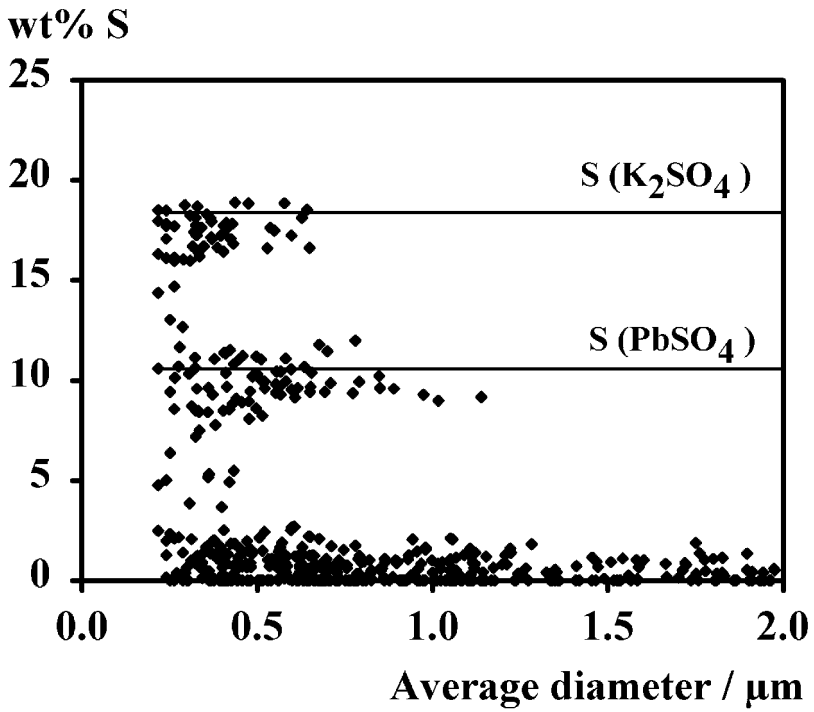
X-ray analysis of biomass fly ash particle (E_0 = 7 keV, I_p = 0.7 nA);
a) image of particles with the analysis spot marked by a cross (SE – image)
b) x-ray spectra

Chemical composition



K_2SO_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)

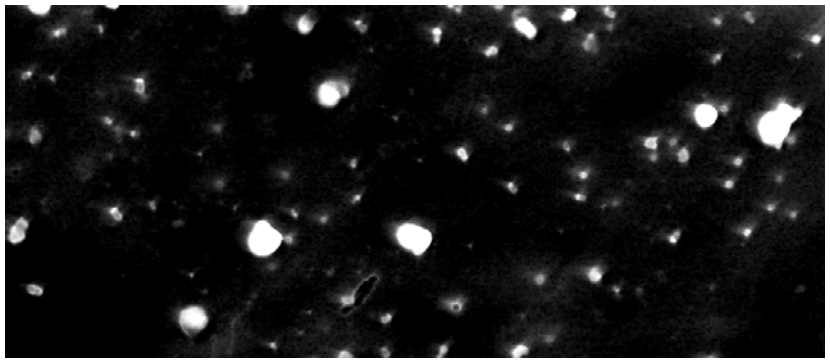
Chemical composition

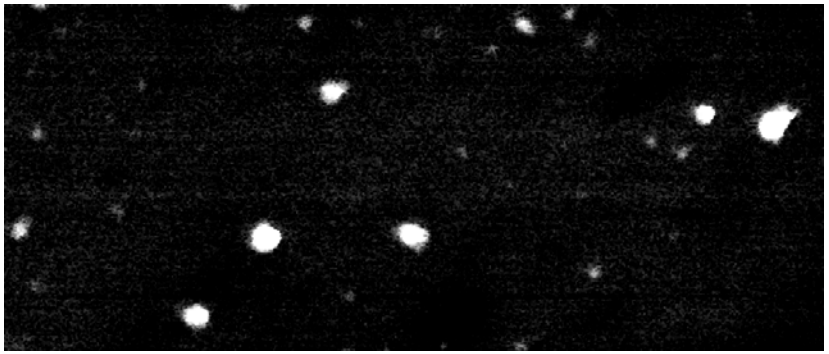


Automated analysis of standard particles: K_2SO_4 , $PbSO_4$ and $PbCl_2$

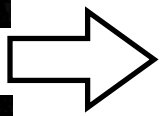
Principles of Automated Particle Analysis

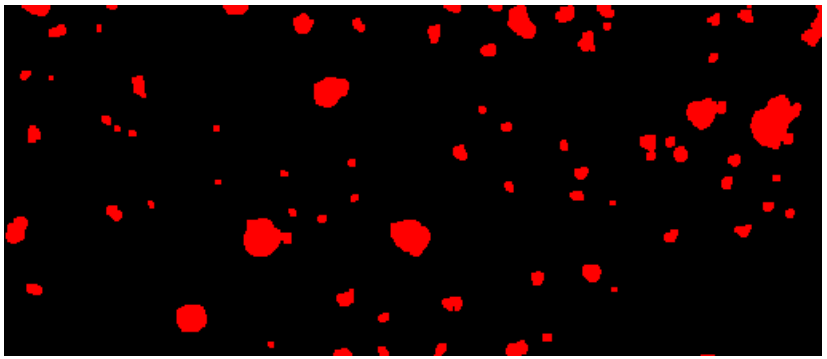
SEI-image

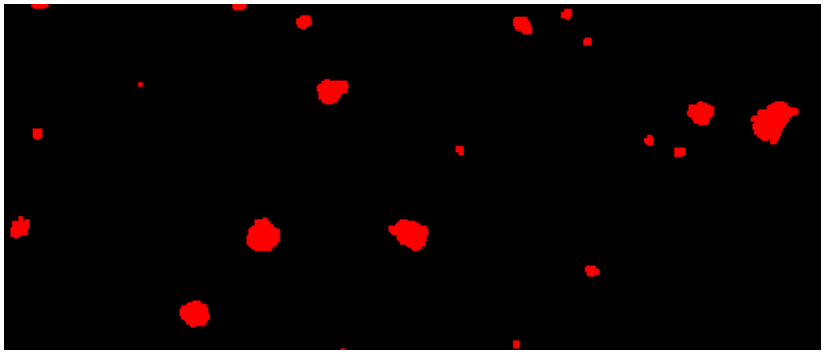




BSE image



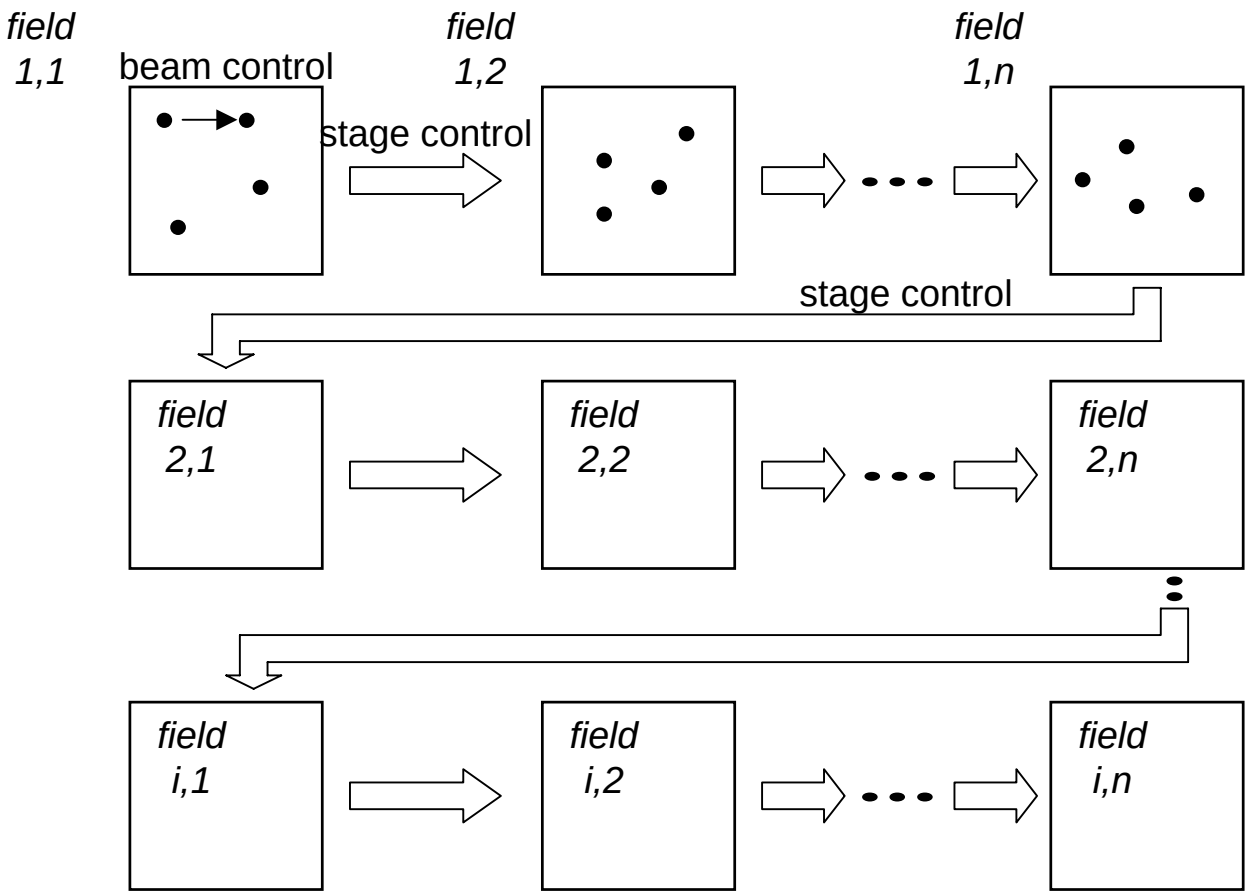




binary images

image width 3.8 μm

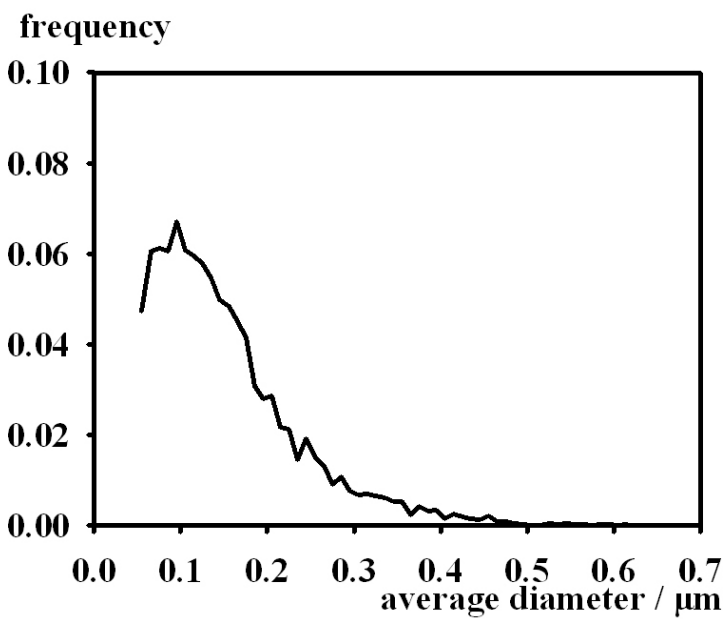
Principles of Automated Particle Analysis



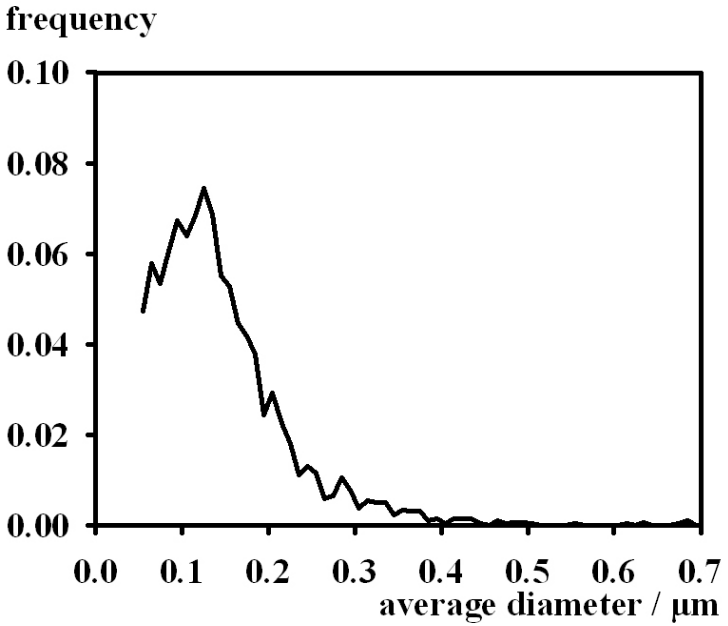
Scheme auf the automated particle analysis procedure

Automated Particle Analysis of aerosols

waste wood



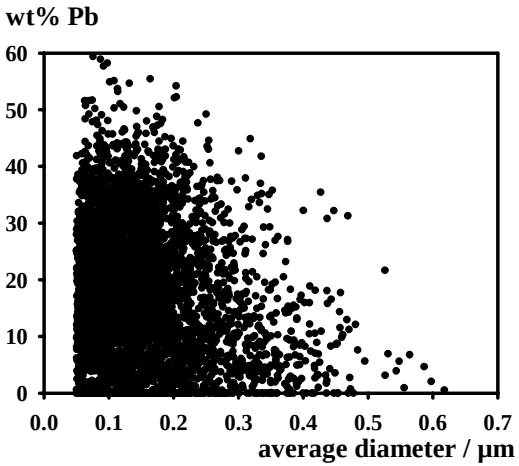
bark

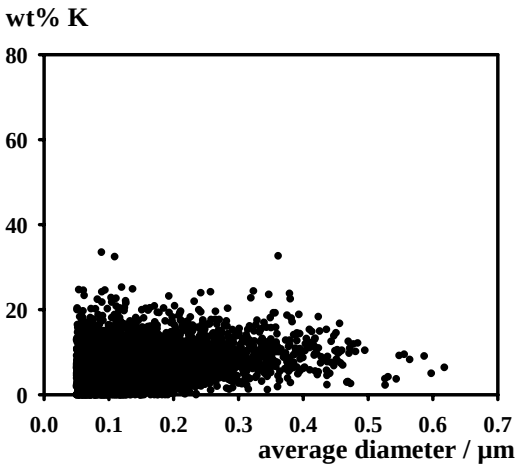


Size distribution of aerosols (fuel: waste wood, bark)

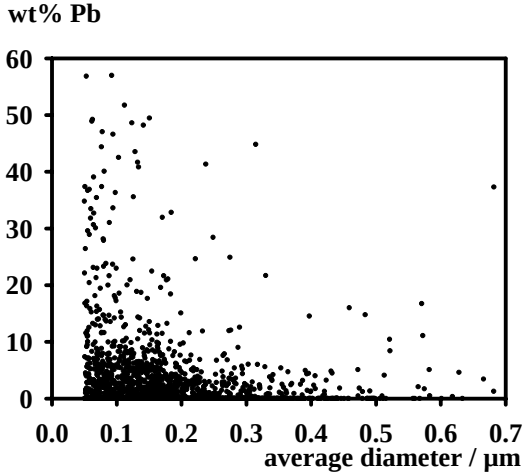
Automated Particle Analysis of aerosols

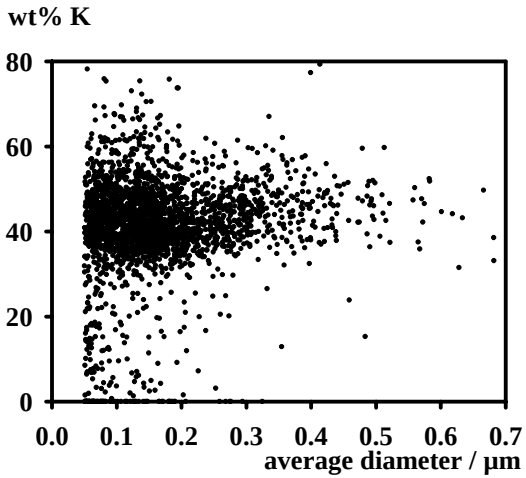
waste wood





bark

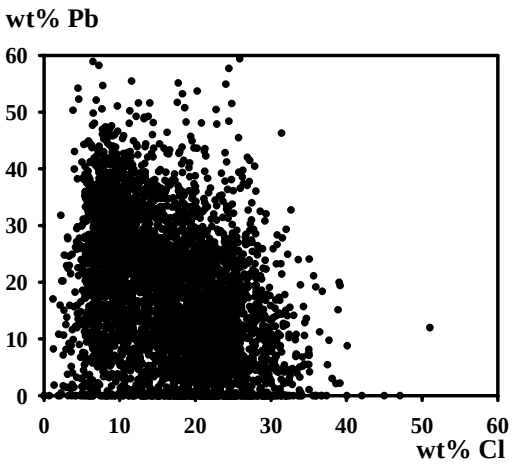


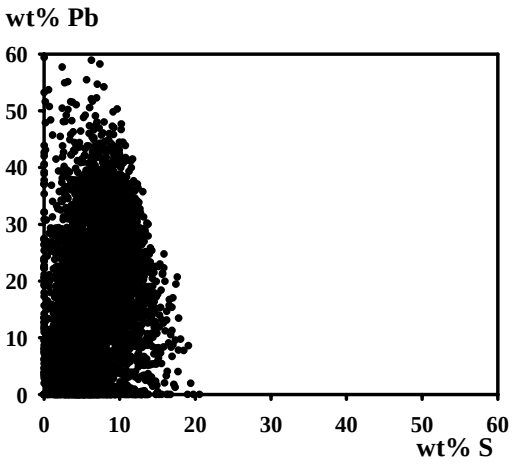


Concentrations of Pb and K in dependence on the diameter of the aerosols (fuel: waste wood, bark)

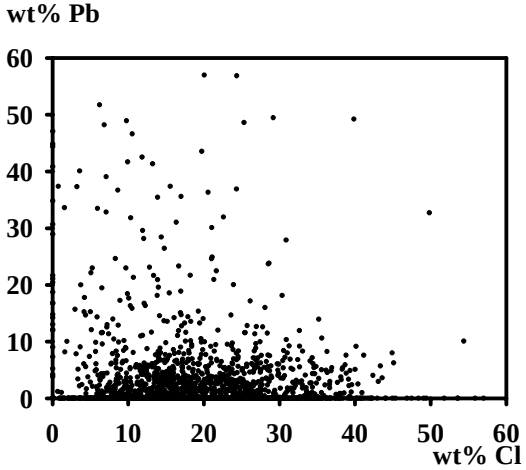
Automated Particle Analysis of aerosols

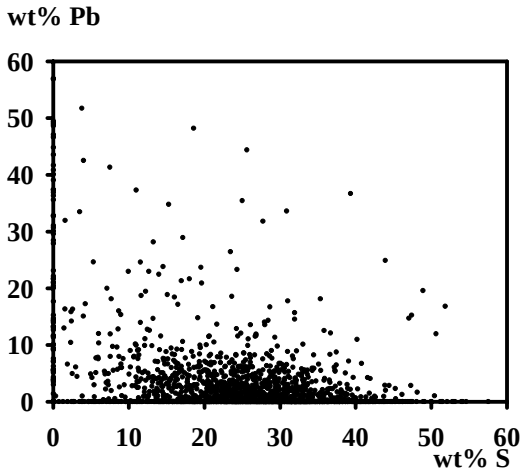
waste wood





bark

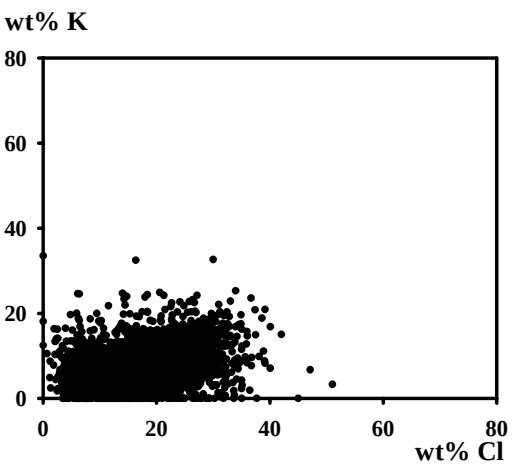


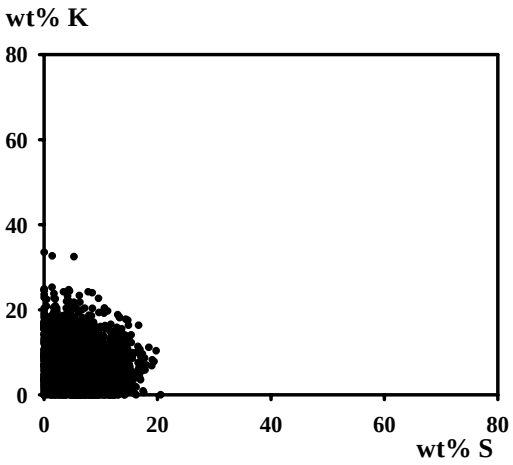


Correlations between various elements for aerosols (fuel: waste wood, bark)

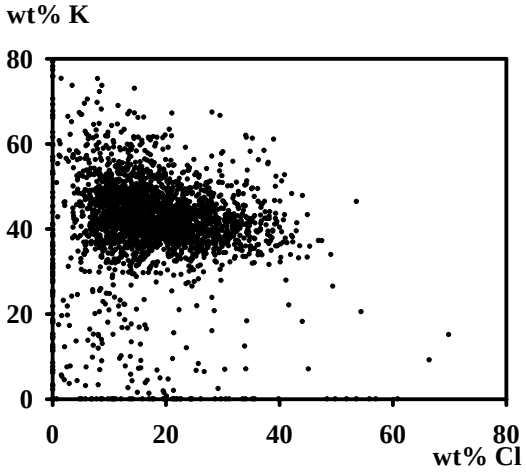
Automated Particle Analysis of aerosols

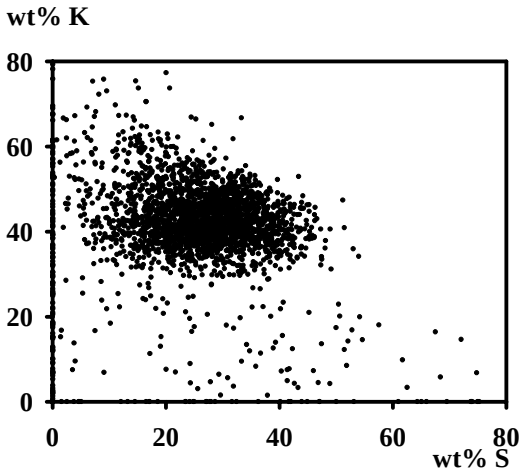
waste wood





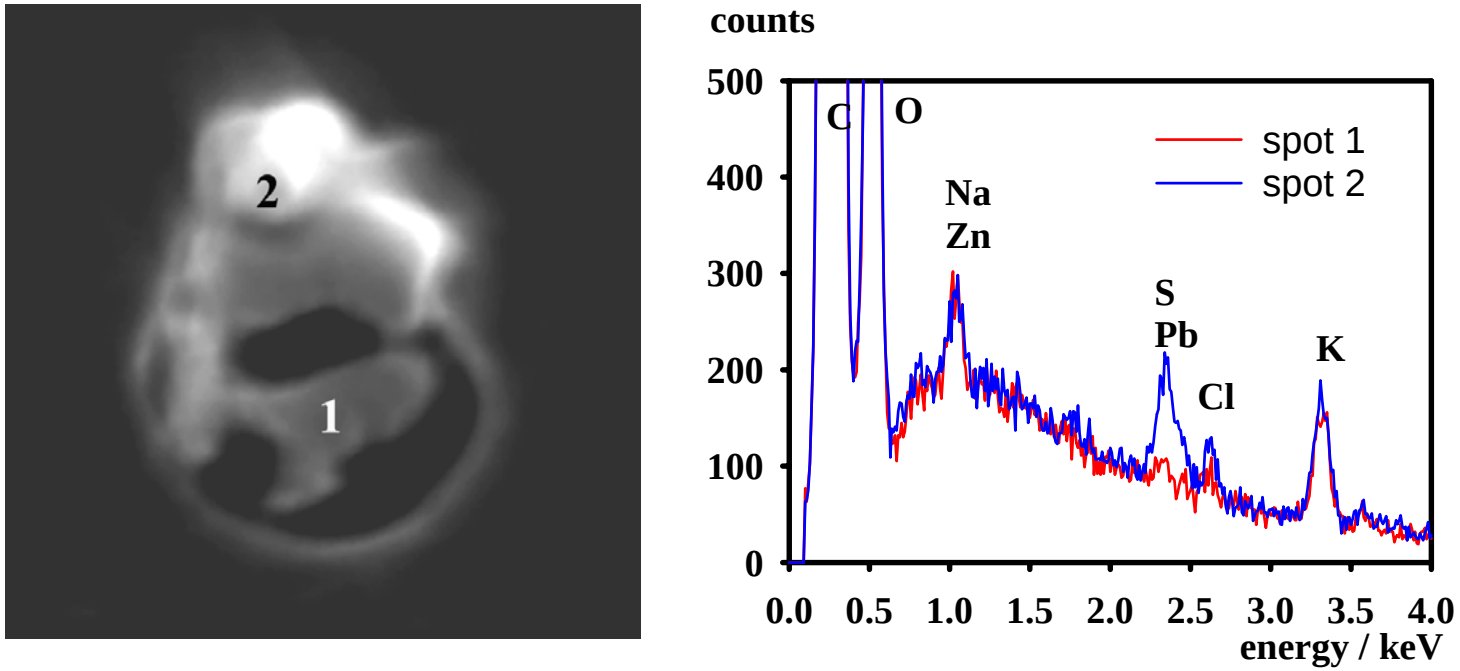
bark





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

Summary

- 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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