FIB-SEM-EDS Analysis of Aerosols: Implications to Particle Optics

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Abstract: In retrieval techniques, aerosol generally considered as chemically homogeneous spheres which results in erroneous results. There is a severe need for multi-dimensional analysis of aerosol. Therefore, mineral dust particles were analyzed using focused ion beam (FIB) coupled with energy dispersive X-ray spectroscopy (EDS) and scanning electron microscopy (SEM). In present study, particle was observed in core-shell type structure, where shell was rich in Fe, Ca, C, Al, and Mg and core were rich in Si. In conventional SEM-EDS, the particle was observed as homogeneous mixture of Ca, Si, Cu, S, K, Mg, C, Fe and Al. Single scattering albedo (SSA) was calculated using FIB-SEM-EDS results and conventional SEM-EDS, difference between two results was observed to increase with increasing wavelength and the maximum difference was observed at 860 nm i.e. 19.38\%. This shows that homogeneous spheres assumption is putting 19.38\% uncertainty in results, which could have a tremendous effect on radiative forcing estimations.

Keywords: Radiation budget, FIB-SEM-EDS, Core-shell, SSA.

1. Introduction

Aerosol particles are major constituent of atmosphere, they affect earths’ radiation budget. This effect can be better understood with assessment of their optical property (Single Scattering Albedo; SSA). The optical property of aerosols depends upon their chemical composition [1], shape [2, 3], size [4] and internal structure [5]. In satellite observations the shape of aerosols are generally assumed as spherical and their composition is assumed to be homogeneous throughout the particle [6]. The conventional techniques like scanning electron microscopy [7] (SEM), Energy dispersive X-ray spectroscopy (EDS) and transmission electron microscopy [8] (TEM) used for the analysis are not capable to reveal the internal structure of the aerosol particle. Therefore, there is a severe need for detailed multi-dimensional analysis of aerosol particles which is capable of imaging embedded phases and voids hidden from the conventional SEM or TEM-EDS.

In the present work, dust particles were analyzed using Focused Ion Beam (FIB) coupled with SEM and EDS. The gallium (Ga) was used to cut the particle which was followed by taking EDS spectra of exposed the surface. This procedure was repeated may time (2–3) on a single particle, that reveals its internal structure.
2. Experimental Section

In the current study, the aerosol particles were collected from Jaisalmer (26.9157° N, 70.9083° E), Rajasthan, India using Envirotech® APM801 sampler. The particles were collected on tin substrate. After collection the particles were stored in desiccator and brought to the CSIR-National Physical Laboratory, New Delhi for individual particle analysis using Auriga Cross beam FIB-SEM-EDS.

3. Result and Discussion

Mineral dust dominated the overall loading of aerosol in atmosphere [9]. Conventional SEM-EDS and FIB-SEM-EDS analysis of dust particle is shown in fig (1). The results of conventional SEM-EDS show that the particle is rich in Ca, Si, Cu, S, K, Mg, Fe, O and Al (fig 1a). After taking the conventional SEM-EDS spectra the particle was milled with high energy focused Ga gun. The milling cuts the particle and exposes its internal structure. EDS spectra of exposed internal surface shows the particle is rich in Si and O only (fig 1b). The atomic percentage of Si and O confirms the presence of SiO$_2$ inside the particle. The same particle was milled second time at the same position to check the elemental composition of the core of the particle. The EDS spectra recorded after second milling shows the presence of SiO$_2$ rich core. The advanced FIB-SEM-EDS analysis shows that the particle is having core-shell type structure, where the core is rich in SiO$_2$ and shell is rich in Fe, Ca, C, Al, and Mg.

![Figure 1. FIB-SEM-EDS analysis of dust particle](image)

Optical property (SSA) of dust particle shown in fig (1) was simulated using T-matrix and core-shell-shell code. The spectral variation of SSA is shown in fig (2). Here, SSA was simulated for five different structures of particles i.e. Quarts homogeneous sphere (S1), Quartz homogeneous spheroid (SPH1), multiple species homogeneously mixed sphere (S2), multiple species homogeneously mixed spheroid (SPH2) and core-shell structured particle (CS). The SSA of S1 and SPH1 was observed to be near to 1 due to completely scattering characteristic of quartz. Chemical composition of S2 and SPH2 was determined by conventional SEM-EDS, SSA for S2 and SPH2 was observed to be very low near to 0.56. The low value of SSA shows the highly absorbing character which was attributed to the presence of hematite and carbon in particle. The effect of shape between SSA of S1, SPH1 and S2, SPH2 was observed to be negligible. Chemical composition of CS particle was determined by advanced FIB-SEM-EDS, SSA for CS was observed to be ranging from 0.54-0.66. The increasing character if SSA in CS was observed due to spectral variation of imaginary part of refractive index of hematite. The SSA of CS was found to be between S1, SPH1 and S2, SPH2. The S1 and SPH1
calculation overestimating the real value, whereas, the S2 and SPH2 were underestimating the real value. The difference between the SSA calculated using conventional SEM-EDS results (S2, SPH2) and advanced FIB-SEM-EDS results (CS) was observed to increase with wavelength and maximum difference was observed at 860 nm. The difference between S1 and CS at 860nm was observed to be 19.38%. Therefore, the conventional results are putting high degree of error in optical property simulations.

Figure 2. Single Scattering Albedo of dust particle shown in fig.1.

4. References