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Investigation of Fly Ash Particulates Using SEM, TEM and Synchrotron Microprobe Techniques

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When coal and coal-based fuels are burnt to produce power, ash is generated as a by-product. Escaped fly ash (EFA) is that ash which vents up the smokestack, transits any mechanical and electrostatic filters and escapes to the atmosphere. EFA particles can remain suspended in the atmosphere for long periods (up to 18 months), interact with atmospheric moisture and so affect atmospheric processes. The Purdue University power plant did an experiment wherein two different fuels (pure coal, and a mixture of 95% coal and 5% shredded tires, TDF) were combusted under identical conditions (at ~1500°C). Detailed studies are being undertaken to ascertain the chemistry of the fuels, combustion products, atmospheric emissions and bioavailability of metals in the ash. This paper reports examination, by SEM, TEM and Synchrotron Microprobe (SMP) techniques, of EFA that was collected on filter paper inserted into the top part of the smokestack above all the filters.

SEM specimens were prepared by carbon coating small pieces of the EFA-exposed filter paper. Images and quantitative SEM EDX analyses were performed using a JEOL 6400 SEM fitted with a Noran Voyager energy dispersive spectrometer [1]. Specimens for TEM and SMP analysis were fabricated by scraping EFA off the filter papers, suspending the scrapings in ethanol and pipetting the mixture onto holey carbon coated Cu TEM specimen grids. TEM CCD images were taken to map the positions of particles of interest for SMP and TEM EDX analysis. SMP data were collected using the high resolution microprobe at the APS at ANL with an excitation beam energy of 17.5 keV, which above the U-L edge. Particles of interest were first located using the previously collected CCD images. Then elements of likely interest were specified and data cubes were collected using a Ge EDX detector. Areas scanned ranged in size from ~5 x 5μm to ~25 x 25 μm and collection times for individual spectra ranged from ~1-4 secs. The IDL based program "GUI_maps" written by S. Vogt was then applied a) to correct raw data (for beam current variations and signal efficiency, using standards NBS 1832 &NBS 1833) and b) to specify and sum the spectra in regions of interest (ROIs). The summed spectra from ROIs were then exported and processed using PIXAN fitting routines [2] to give peak areas. TEM EDX data were collected using JEOL 2000 FXII operated at 200 keV fitted with a Link ISIS energy dispersive spectrometer [3].

Bulk chemistry of the two fuels used showed TDF contains 5 times as much Zn and 1.5 times as much S as pure coal. SEM and TEM show both EFAs contain amorphous and crystalline material. Al-Si-O glass (including glass spheres) constitutes most of the amorphous material. The crystalline phases found include lime, mullite and a variety of euhedral S and O-bearing phases (inferred to be sulfates). Both EFAs contain sulfates of Ca, Pb and Fe-Zn; and TDF EFA contains Zn-sulfate. The Zn and Fe-Zn sulfates rnage in size from a few nm to >100 μm. The larger crystals must have condensed after the flue gas had passed through the filters in the smokestack. Figures 1 and 2 show a TEM image and SMP maps of a single poly-phase EFA particle. Full analysis of the microanalytical

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data we have collected associated with many such images will reveal which toxic and radioactive minor constituents in the starting fuel sequester into which EFA phases. Beyond information provided by TEM, preliminary analysis of our SMP data shows the presence of V, Ni, Cu, Pb, Sr in Al-silicate phases and the presence of V and Cu in sulfate phases. All these metals are potentially toxic to humans [4]. [5]

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[5] The authors thank 2-id-d station staff at APS for assistance. Use of the APS was supported by the U.S. DOE, BES, Office of Science under Contract No. W-31-109-Eng-38. Support for travel was provided by the Australian Government funded Access to Major Research Facilities Program.

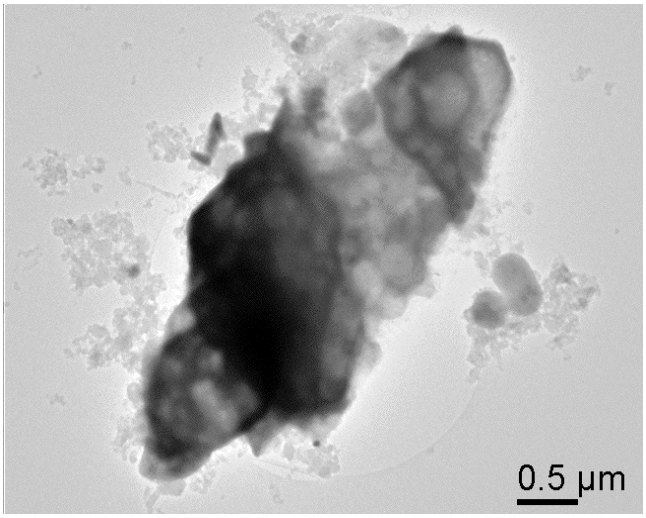
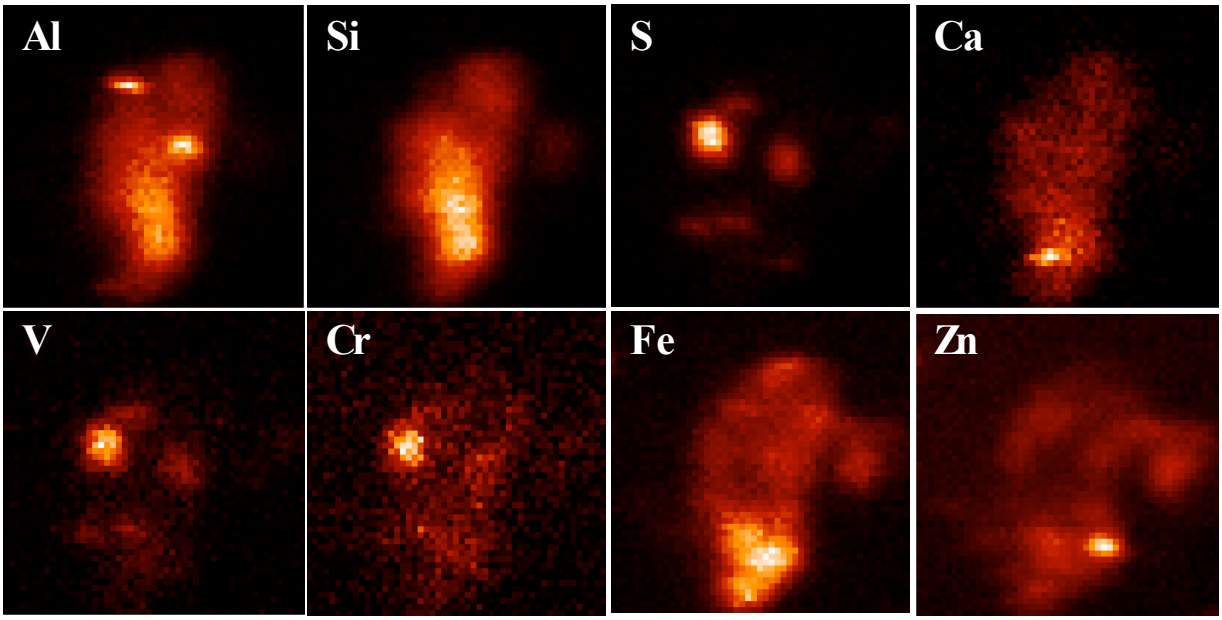


Figure 1 (left). Bright field TEM image of many phase particle.

Figure 2 (below). SMP elemental maps of the same particle shown in figure 1.



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