An Evaluation of Fly Ash in Sediment by Microscopy

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Introduction

Since the occurrence of the fly ash spill at the Kingston facility the majority of material in the main channel of the Emory River has been, or is currently being recovered. The next step in the cleanup process requires the identification and removal of ribbons and pockets of material along the river's edges.

In order to facilitate this cleanup one must know which areas have been impacted. Classical approaches such as metals analysis have been unsuccessful due to sample invariability as well as the absence of unique chemical tracers associated with fly ash particles that can be reliably used to map location and determine concentration.

The purpose of this investigation is to develop an analytical technique that can be used to identify and reasonably quantitate fly ash in sediment samples; thus providing the necessary information to conduct targeted material removal efforts. Optical Microscopy techniques offer material identification based on characteristic morphology as well as transmitted polarized light properties of the particles. This analytical approach was applied to core samples taken from various locations along the banks of the Emory River.

Characterization

The purpose of evaluating the reference Fly Ash sample was to identify unique characteristics of the material in order to identify the presence and amount of fly ash in environmental samples. Polarized Light Microscopy (PLM) and Scanning Electron Microscopy/Energy Dispersive Spectroscopy (SEM/EDS) techniques were utilized to evaluate the sample.

The PLM analyses identified that the majority of the fly ash particles were isotropic to opaque spheres along with particulate consistent with more bottom ash type morphologies. The spheres ranged in opacity and they occurred both as distinct particles or agglomerations.

The SEM results indicate that the reference material consisted primarily of spherical aluminum silicates (fly ash) and bottom ash particles. Single particles ranged from approximately 50 micrometers (µm) to submicron in diameter, although ash particles were often agglomerated. Fly ash and bottom ash particles had similarities in composition consisting primarily of silicon, aluminum, and oxygen; with lesser amounts of iron, potassium, titanium, and calcium. The remainder of the sample consisted of minor amounts of iron-rich spherical particles and trace concentrations of porous carbonaceous material.

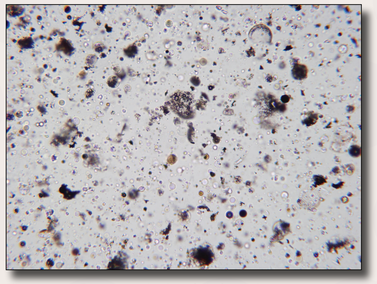

Figure 1: Plane polarized light microscope image illustrating fly ash particles of various sizes and opacity for fly ash reference sample (3068675). Field of view = 0.5 mm.

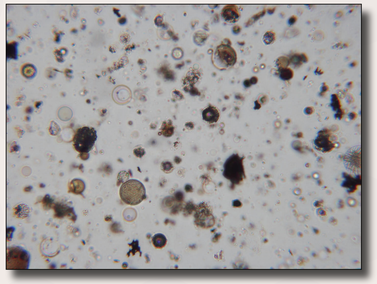

Figure 2: Plane polarized light microscope image illustrating fly ash particles of various sizes and opacity for fly ash reference sample (3068675). Field of view = 0.25 mm.

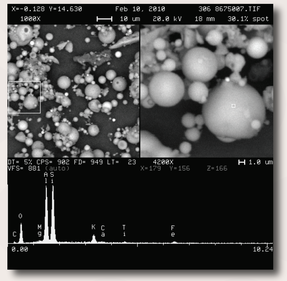

Figure 3: Back scattered electron images and elemental spectra collected from the Reference Fly Ash.

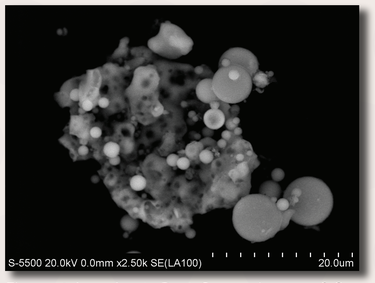

Figure 4: Low Angle Back Scatter image of fly ash agglomerate illustrating differences in elemental composition.

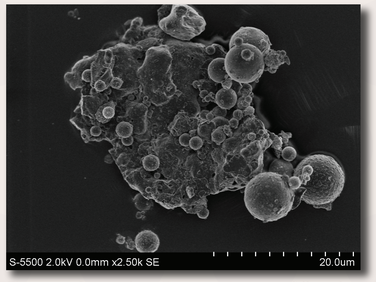

Figure 5: Secondary Electron image of fly ash agglomerate illustrating surface morphology.

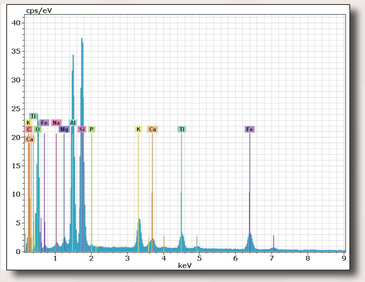

Figure 6: Energy Dispersive Spectra illustrating elements and relative abundance associated with fly ash agglomerate.

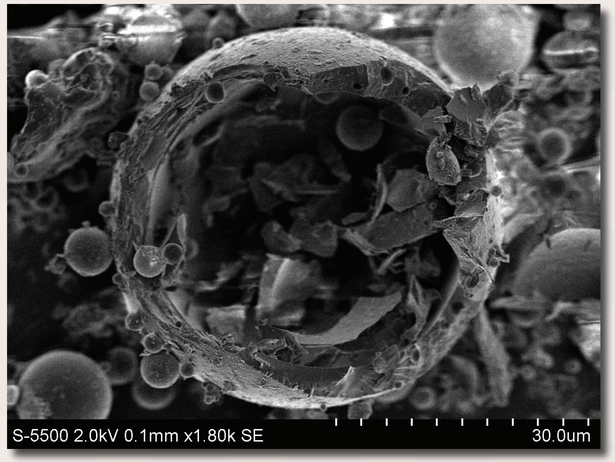

Figure 7: Secondary Electron image of fly ash particle illustrating particle structure.

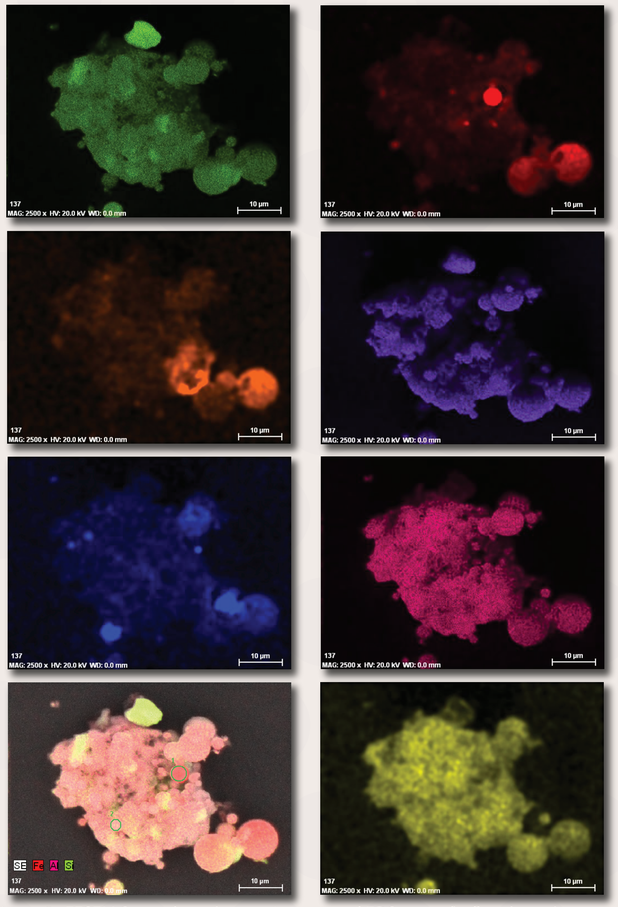

Figure 8: FESEM Secondary electron images and EDS map of a fly ash agglomerate.

Analysis

Once the fly ash material was sufficiently characterized, reference samples were prepared. A field sample found to be free of fly ash was selected as a project specific reference matrix. Known amounts of traceable fly ash material were added to the matrix to achieve specific weight percent concentrations. Melt mount slides were prepared from the mixed standards to serve as quality control and training specimens.

All analysts familiarized themselves with the sediment matrix, fly ash, and concentrations associated with various loadings. Accuracy and precision were then evaluated and field samples were subsequently analyzed by PLM.

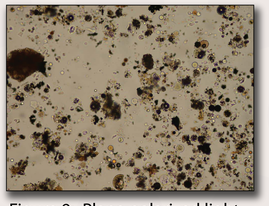

Figure 9: Plane polarized light microscope image illustrating a field sample with heavy loading (75-80%) of fly ash.
Field of view = 1.2 mm

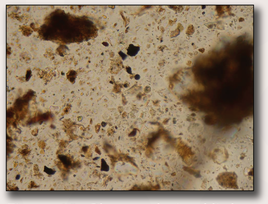

Figure 10: Plane polarized light microscope image illustrating a field sample with nominal fly ash loading.
Field of view = 0.6 mm

Results

The results indicate that analysis by optical microscopy can easily and reliably detect fly ash in field samples.

RJLG Sample ID	Date Analyzed	Date Sampled	Particulate Components	
3068676	2/11/2010	1/07/2010	Quartz	45-50
			Clay	25-30
			Opaques	10-15
			Feldspar	1-5
			Organic Material	1-5
			Fly Ash	<1
			Amphibole	<1
3068677	2/11/2010	1/07/2010	Fly Ash	75-80
			Quartz	15-20
			Clay	5-10
			Feldspar	1-2
			Organic	1-2
3068678	2/11/2010	1/13/2010	Quartz	60-70
			Clay	15-20
			Feldspar	1-5
			Opaque	1-5
			Organic	1-5
			Fly Ash	<1
3068679	2/11/2010	1/13/2010	Quartz	58-65
			Clay	10-15
			Opaques	10-15
			Feldspar	1-5
			Organic	1-5
			Fly Ash	1-2
3068680	2/11/2010	1/18/2010	Fly Ash	60-70
			Quartz	20-25
			Clay	1-5
			Opaques	1-5
			Organic	1-5