GRAPHICAL REPRESENTATION OF CCSEM DATA FOR COAL MINERALS AND ASH PARTICLES.

Naresh Shah, G.P. Huffman, F.E. Huggins, Anup Shah
233 Mining and Mineral Resources Building
University of Kentucky, Lexington, KY 40506-0107.
W.J. Mershon , G. Casuccio
R.J. Lee Group, Inc.,
350 Hochberg Road, Monroeville, PA 15146.

Introduction:

Since its inception, Computer Controlled Scanning Electron Microscopy (CCSEM) has proved to be a powerful tool for materials science (1-4). In coal research, CCSEM has been used to obtain important physical mineralogical information about coal (5,6). Several factors make CCSEM the method of choice in this area. Bulk mineralogical and chemical methods are time consuming and limited. Furthermore, such methods are incapable of providing physical characteristics like size and shape of coal mineral particles which clearly play important roles in ash formation during combustion (7) and catalysis during liquefaction (8). Besides coal mineral analysis, CCSEM has been used to identify and characterize ash particles to gain insight into deposition and fouling characteristics of coals during the coal combustion phenomena. Recent research emphasis in the CCSEM field has been on investigating mineral-maceral associations (9).

CCSEM is basically a statistical analysis method. Thousands of individual particles are analyzed in the course of a single sample analysis and for every one of them chemical composition from X-ray EDS spectra and dimensional (size) parameters from back scattered electron imaging are obtained. These data are then statistically analyzed to infer mineralogical characteristics of the sample. Traditionally, all the statistical data thus obtained is listed in a tabular format. Although such information is very useful and can not be easily obtained by other means, some rather important information and relationships may be obscured in such typical statistical analysis. Graphical representation of CCSEM data provides the means for revealing this hidden complementary information. Though such information may not be easily quantifiable, it can provide the user with a completely different perspective about the sample. Graphical representation of CCSEM data often unveils trends and correlations in a very user friendly way and the information, which usually is difficult to extract from tables, can be easily displayed and understood. In this paper, we show some of the graphical representations our group has developed and used to analyze various problems in coal research.

Size distribution:

Determination of particle size distributions is one of the oldest applications of CCSEM. Bar and line charts make data presentation very user friendly. Stacked charts help in understanding size distribution trends of different minerals in the same sample or the same mineral in different samples. Commercial CCSEM instruments invariably provide software to display such trends. Figure 1 shows pyrite size distributions for some of the Argonne premium sample bank coals (6).

Binary diagrams:

To study the interaction between any two components, we have developed a binary diagram representation. Two component CCSEM data are plotted as a number or volume percentage frequency as a function of concentration of one of the components. Since the data is normalized to 100 percent to represent only two components, it also displays the same information with decreasing concentration of the second component. Figure 2 shows Fe-S binary diagrams for the Argonne Beulah coal. Figure 2a is a plot of number percentage distribution as a function of Fe and S concentrations for all the particles with more than 80% combined counts for Fe and S from EDS spectrum. Since the particles can have different sizes, number percentage representation alone is not quite adequate. We have also incorporated the size information in binary diagram (Figure 2b). If we know density as a function of the concentration, such plots can be easily converted to actual weight distribution.

In some cases, representing just two components is not sufficient and interactions of a single component with a combination of phases or a mineral with several elements (e.g. illite, kaolinite) is important. To display such interactions graphically, we have modified binary diagrams so that one of the ends can be a combination of components. Figure 3 shows several such diagrams for a series of Beulah ash samples collected by University of Arizona's combustor on a size segregating impactors. Graphical representation of such data clearly shows the trends in reaction mechanism. With increasing impactor number (smaller size ash particles), there is more interaction between the aluminosilicate phase with CaO forming a glassy phase.

Ternary diagram:

Pseudobinary diagrams like the one mentioned above (where two components are lumped together), do not provide the interactions between all the components in the ash chemistry. A true ternary diagram, as shown in Figure 4, displays individual particles with sum of x-ray counts for the three components > 80%. Figure 4 shows the same sample as in Figure 3a. Using such a ternary diagram representation, we were able to confirm the presence of a nepheline (NaAlSiO4) phase in ashes of low rank coals with large alkali content. Such a phase can play an important role in the fouling and deposition characteristics of the coal during combustion (10).

As a logical extension to ternary diagrams, we can also draw pseudoternary and higher order diagrams in one of two ways. Just like pseudobinary diagrams, we can represent several phases lumped together on one of the vertices of the ternary diagram. Such alumina-silica-base ternary diagrams, where base is a combination of several basic elements (viz. Na, Mg, K, Fe etc.) have been traditionally used by refractory engineers (11). We are currently working on extending such diagrams to include melting point and viscosity information for the ash particles. Such information should provide further insight into stickiness of the ash particles and thereby the fouling and deposition characteristics of such ashes. Alternatively, we can use a color display to represent a fourth dimension in the ternary diagrams. By assigning different colors or different symbols to different concentrations we can display the concentration of the fourth phase on the ternary diagram by different colors (or symbols).

To make this tool more user friendly, we have developed an interactive computer program. In this program, the user can display data in any combinations of components at the three vertices. The user can also select to display a fourth component by different color or a combination of up to five components on one of the vertices. Using a mouse, the user can further select a subset of the data displayed by positioning a smaller variable size triangle on this ternary diagram. The size distribution information of the interactively selected data subset can then be displayed.

One of the major drawbacks of such a ternary diagram is its inability to show number distribution within the ternary diagram. If two particles have the same composition, they overlap giving only one point on the ternary diagram which can be quite misleading. To overcome this problem, we have added the fourth dimension in the ternary diagram by displaying it in a perspective view. Figure 5 is a number density distribution of Ca-Si-Al ternary diagram for Beulah ash collected on an impactor filter (#1) of University of Arizona combustor (Note the correspondence to Figures 3a and 4). As is clearly evident, the points at both the Ca and Si vertices on the ternary diagram actually represent a large number of particles. Such concentration information is not readily available on the simple ternary diagrams.

Often the physical size information provided by CCSEM is not enough as such information does not reveal anything about the morphology or the association of the particles. To resolve this problem, we have improved CCSEM data acquisition program by including real-time microimaging (12). Once the particle is identified and its x-ray spectrum is obtained, the SEM beam is controlled by the computer to raster the area bounded by a minimum rectangle totally surrounding the particle. The video image of the particle is then captured and stored on the computer with the EDS spectrum as well as the standard physical dimensions and X-ray counts. Since the raster is narrowed down from the entire frame to just the particle dimensions, we can also get a virtual magnification of the particle. These video images can be called up by a mouse driven selection of the particles on a ternary diagram. Figure 6 shows such a digitally stored backscattered electron image of a single coal particle sitting on a beryllium substrate. An experienced user can then deduce something about the morphology and juxtaposition of these particles. We are using such digital images to investigate mineral-maceral association.

Conclusions:

The amount of information generated by CCSEM is difficult to assimilate just by tabular presentations and some important trends have been obscured in conventional analysis. We are developing graphical tools to represent the standard information in a more friendly representation as well as to depict information previously hidden. Further progress in such CCSEM analysis will be carried out by including n-dimensional analyses and video image processing.

REFERENCES

1. R.J. Lee, F.E. Huggins, G.P. Huffman, Scanning Electron Microscopy, 1, (1978), 561-568.
2. F.E. Huggins, D.A. Kosmack, G.P. Huffman, R.J. Lee, Scanning Electron Microscopy, 1, (1980), 531-540.
3. R.J. Lee, J.F. Kelly, Scanning Electron Microscopy, 1, (1980), 303-310.
4. G.P. Huffman, F.E. Huggins, R.W. Shoenberger, J.S. Walker, F.W. Lytle, R.B. Greegor, Fuel, 65, (1986), 621-632.

5. W.E. Straszheim, J.G. Yousling, R. Markuszewski, in Mineral Matter and Ash in Coal (Ed.: K. Vorres), American Chemical Society, (1986), 449-461.
6. N. Shah, R.A. Keogh, F.E. Huggins, G.P. Huffman, A. Shah, B. Ganguly, S. Mitra, ACS Div. of Fuel Chem. Preprints, 35(3), (1990), 784-792.
7. G.P. Huffman, F.E. Huggins, N. Shah, A. Shah, Prog. Energy Combust. Sci., 16, (1990), 243-251.
8. F.V. Stohl, ACS Div. of Fuel Preprints, 34(3), 1989, 814-818.
9. W.E. Straszheim, R. Markuszewski, Energy & Fuels, 4, (1990), 748-754.
10. J. J. Helble, S. Srinivasachar, A. A. Boni, S. G. Kang, A. F. Sarofim, J. M. Beer, N. Gallagher, L. Bool, T. W. Peterson, J. O. L. Wendt, N. Shah, F. E. Huggins, and G. P. Huffman, Proc. Sixth Ann. Pitt. Coal Conf., 1, (1989), 81-89.
11. "Phase Diagrams for Ceramist", Volumes 1-5, American Ceramic Society, (1964).
12. B.C. Henderson, I.M. Stewart, G.S. Casuccio, American Lab., (Nov. 1989).

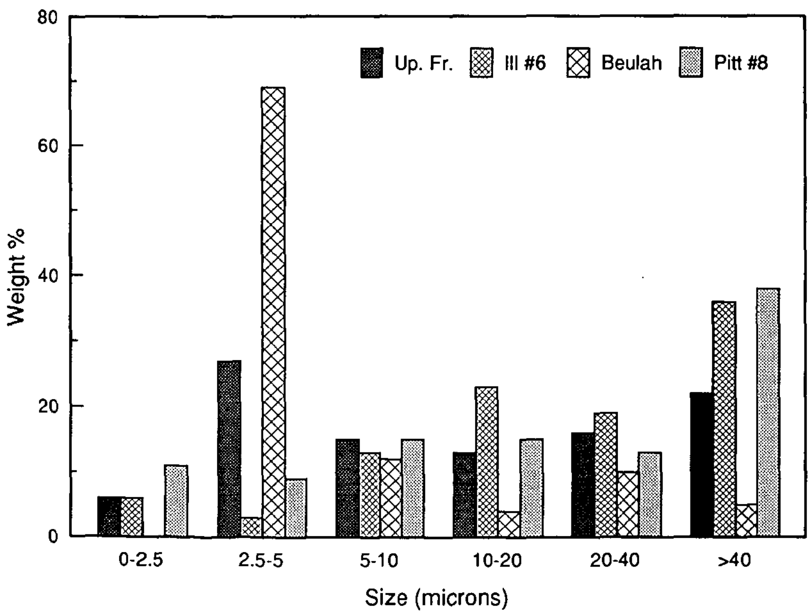

Fig. 1. Example of size distribution obtained by CCSEM for pyrite in several Argonne coals.

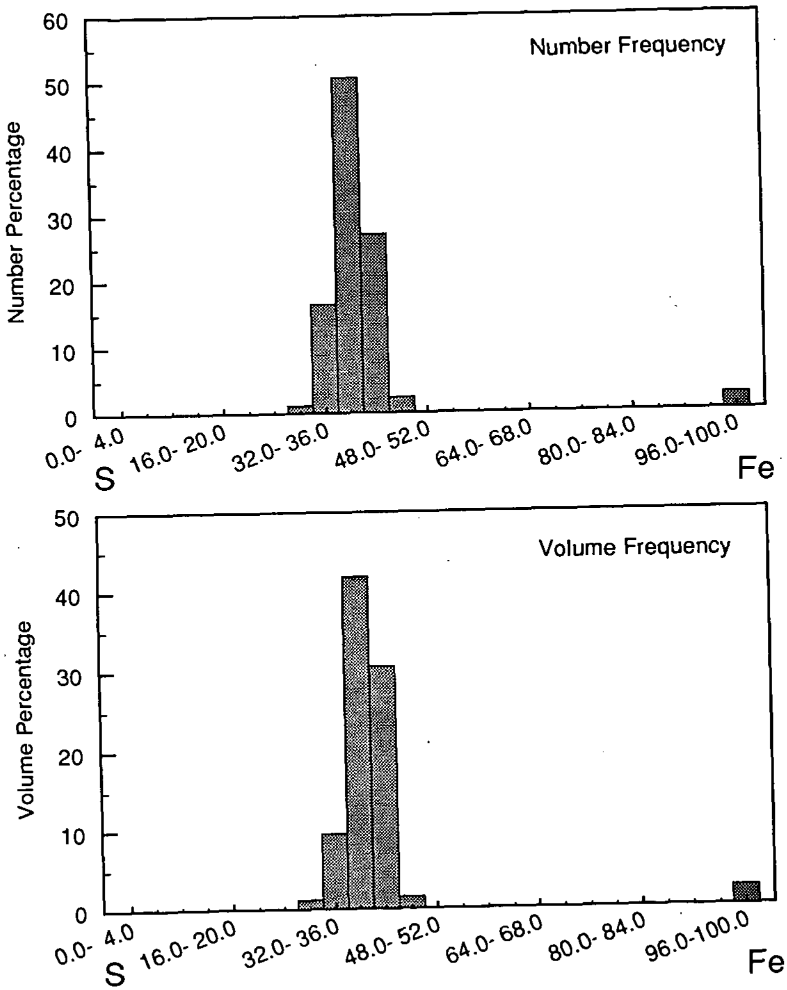

Fig. 2. Examples of Fe-S binary composition diagrams for Beulah coal.

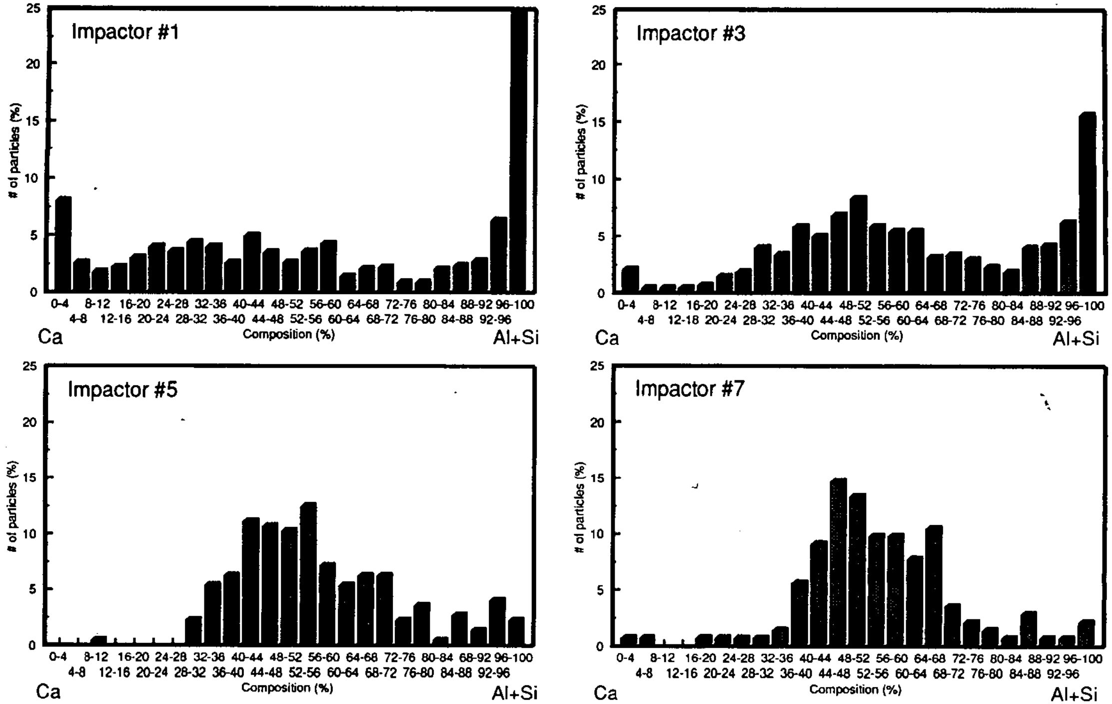

Fig. 3. Ca-Al+Si pseudo binary diagram for Beulah ash collected on size segregating impactor filters. Note the decrease in number of pure Ca and Al+Si particles and increase in glassy Ca+Al+Si mixture particles with increasing impactor number (decreasing size).

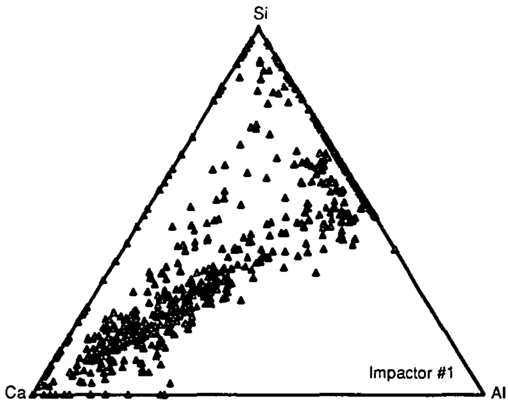

Fig. 4. Ca-Al-Si ternary diagram for Beulah ash

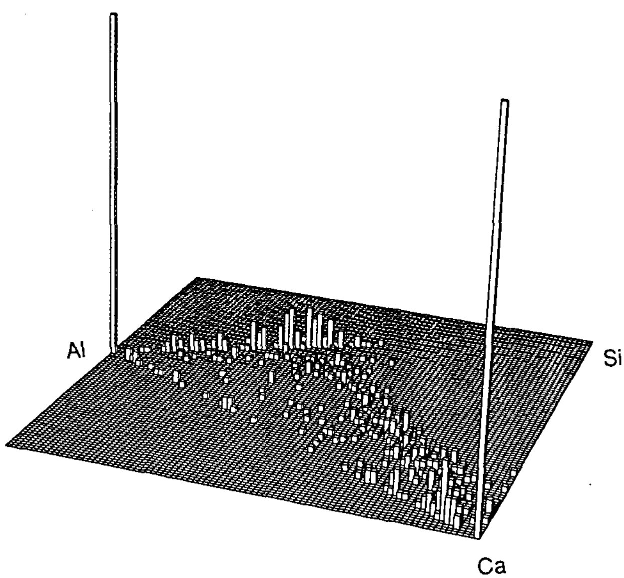

Fig. 5. Ca-Al-Si ternary diagram for Beulah ash showing number frequency.

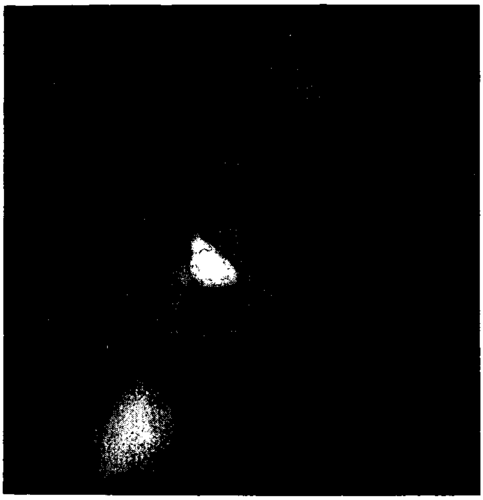

Fig. 6. Digitally stored backscattered electron image of a coal particle on a beryllium substrate. Note a bright mineral particle included within the dark coal matrix.