

Distinguishing Coal, Coke and Other Black Particles

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KEYWORDS

Opaque particles, coal, anthracite, bituminous, lignite, petroleum coke, peat

ABSTRACT

Particles of coal (anthracite, bituminous, and lignite) and coke can be distinguished from other black particles based on light microscopy examination. It is possible to distinguish between coal and petroleum coke particles based on their elemental composition as determined by electron microscopy x-ray analysis. This paper provides some background information about each substance and augments the information in *The Particle Atlas* (published by McCrone Research Institute) about the particle characteristics as determined by light and electron microscopy. Example micrographs and x-ray elemental spectra of particles of coal and petroleum coke are included.

INTRODUCTION

Millette et al. 2007 (1) have described the microscopic characteristics of many of the particles that may be encountered when using the ASTM Standard Practice D6602 (2) in the investigation of darkening agents. This paper focuses on two types of black particles: coal and petroleum coke.

Coal is a term for a brown and black, brittle, compact, amorphous combustible sedimentary rock containing more than 50% by weight and more than 70%

by volume carbonaceous material (3). All types of coal are formed naturally by the application of heat and pressure to swamp deposits that contain abundant plant matter. Anthracite coal, bituminous coal and lignite coal are commercial classifications (or ranks) of coal based on thermal considerations. Anthracite produces more heat per ton when burned than either bituminous coal or lignite.

Peat is the unconsolidated deposit of semicarbonized plant remains that acts as the precursor material in the coalification process. Coalification begins with the burial of peat deposits by overlying sediment. The associated increase in heat and pressure results in conversion of peat to lignite, which is the lowest rank of coal. Further changes in rank occur as thickness of the overlying sediments increases. Next to be formed is bituminous, or "soft" coal. Anthracite, "hard" coal, represents the highest known rank of coal.

Commercial coke is the residue left after the volatile matter is driven out of bituminous coal or petroleum (4). Coke has also been described in *The Particle Atlas* as "the residue after the volatiles have been driven off from any carbonaceous fuel." (5) Commercial coke is produced from coal and heavy oils (petroleum coke) by driving off the volatile gases in an oven. Commercial coke can also be generated from a wood source and is called charcoal.

A considerable amount of work involving petrographic examination has been performed on coal. Coal petrology involves the examination of thin sections (6, 7) and deals with identifying mineral inclusions and

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determining coal maceral (source organic unit) types such as vitrinite (humus), sporinite (spores/pollen) and alginite (algae). Much less microscopical work has been devoted to the general characterization of coal particles. A considerable literature search found *The Particle Atlas* to be the only published reference that contains light and electron microscope images of coal and coke particles.

This article augments the information in *The Particle Atlas* about the particle characteristics of coal and coke as determined by light and electron microscopy.

MATERIALS

Reference samples of coal (anthracite, bituminous, lignite) and peat were obtained from the American Coal Foundation (ACF), Washington, DC. Samples of reference bituminous coal (NIST - SRM 2693) and petroleum coke (NIST - SRM 2718) were obtained from the National Institute of Standards and Technology. A sample of petroleum coke from a commercial supplier was also obtained for study.

METHODS

The reference samples were first examined by stereomicroscopy utilizing a Zeiss Stemi 2000 stereomicroscope having a magnification range of 6.5X to 47X. The samples were then examined by polarized light microscopy (PLM) augmented with top light illumination (reflected light). PLM examination was conducted utilizing an aus Jena, Jenapol microscope having a magnification range of 32X to 500X. Representative portions of each material were examined and analyzed by scanning electron microscopy (SEM) using a JEOL model JSM-6400 or JSM-6490 microscope coupled with a Noran or Oxford x-ray energy dispersive spectrometry (EDS) system. The fine fractions were analyzed by analytical electron microscopy (AEM) using a Philips CM120 transmission electron microscope (TEM) equipped with an Oxford EDS x-ray analysis system.

RESULTS

Anthracite coal particles (ACF)

In transmitted light, the material is opaque (Figure 1). In reflected light a high degree of reflectivity, and conchoidal and hackly fracture is commonly observed (Figure 2). SEM-EDS shows the presence of Al, Si, and S (Figure 3).

Bituminous coal particles (ACF and NIST 2693)

In transmitted light, thicker sections appear opaque while a red to brown color is transmitted at thin edges and in very fine particles (Figure 4). Occasional points of amber or deep red colors are observed in reflected light (Figure 5). The material exhibits high specular reflectivity. Conchoidal fracture is observed together with irregular to rough fracture. SEM-EDS shows Al, Si, and S (Figure 6).

Lignite coal particles (ACF)

In transmitted light, coarser fragments appear opaque. Thin edges and very fine particles transmit a red to brown color (Figure 7). It is generally less reflective than bituminous coal and shows some reddish brown coloration in top light (Figure 8). Occasional conchoidal fracture is observed in reflected light, though the more common fracture observed is narrow, consisting of ragged points and lines. Relict plant structure may be preserved but may be more easily recognized in larger fragments (Figure 9). SEM-EDS shows Al, Si, S and Ca (Figure 10).

Peat particles (ACF)

In transmitted light, the material is transparent to translucent clear to brown (Figure 11). The material disaggregates easily and bio-film, predominately fungus, is common. Relict plant structure is common. In reflected light, the biological material/plant structure is very evident (Figure 12). SEM-EDS shows Al, Si, and Ca (Figure 13).

Petroleum Coke particles (NIST 2718 and commercial coke)

In transmitted light the coarser fragments appear opaque. Thin edges and very fine particles transmit a reddish-brown coloration (Figure 14). In reflected light conchoidal fracture is observed with high reflectivity (Figure 15). SEM-EDS shows only S (Figure 16).

DISCUSSION

The characterization of coal particles presented in this study are similar to those described in *The Particle Atlas*. The petroleum coke particles characterized in the current study are more varied in morphology than those described in *The Particle Atlas*, which depicts petroleum coke as "rounded tending toward spherical." The reference petroleum coke particles examined in this study also exhibit irregular shapes and morphologies similar to bituminous coal particles.

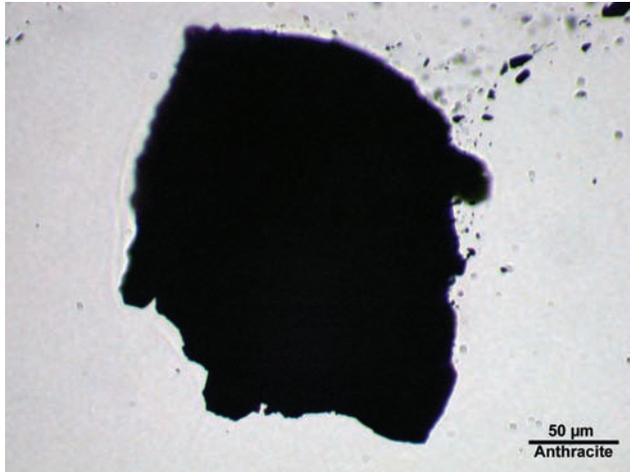


Figure 1. Anthracite coal, transmitted light.

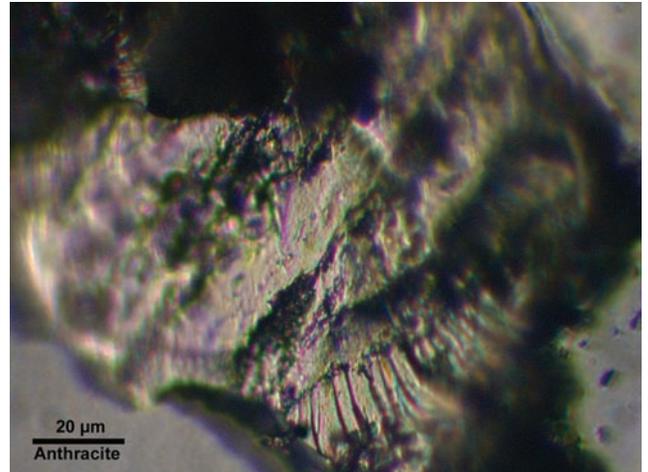


Figure 2. Anthracite coal, reflected light.

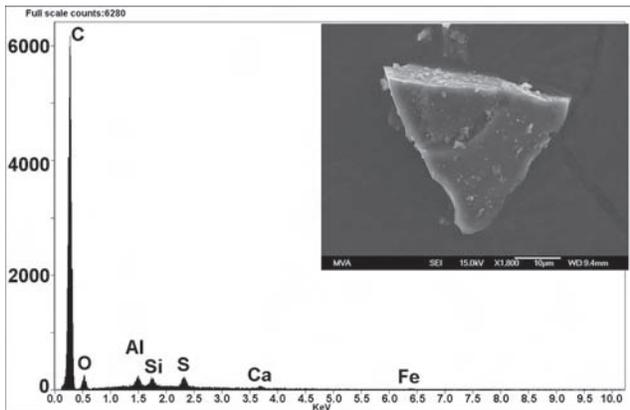


Figure 3. Anthracite coal, SEM.

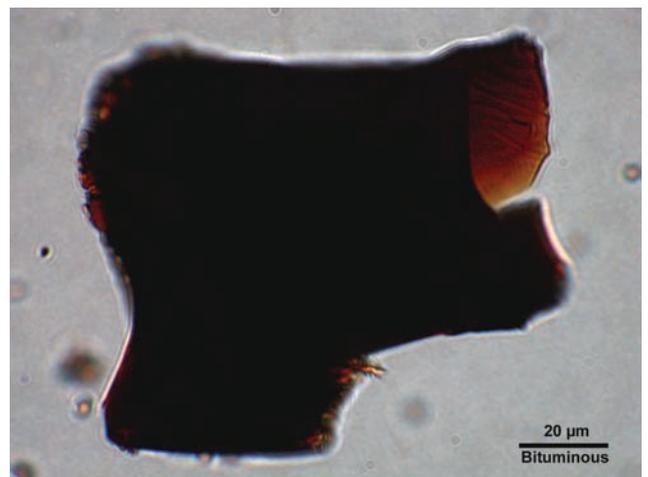


Figure 4. Bituminous coal, transmitted light.

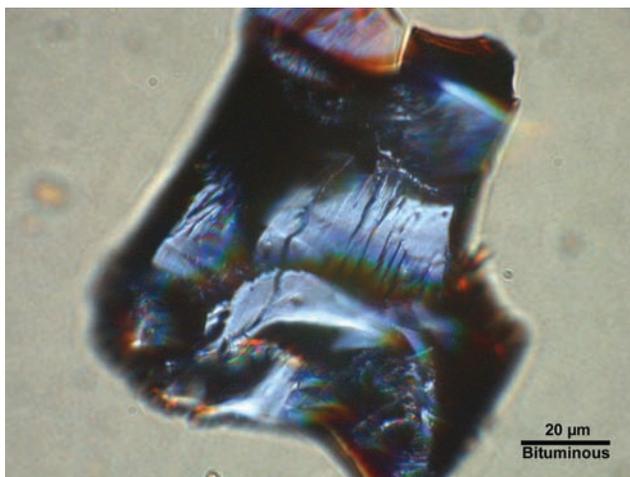


Figure 5. Bituminous coal, reflected light.

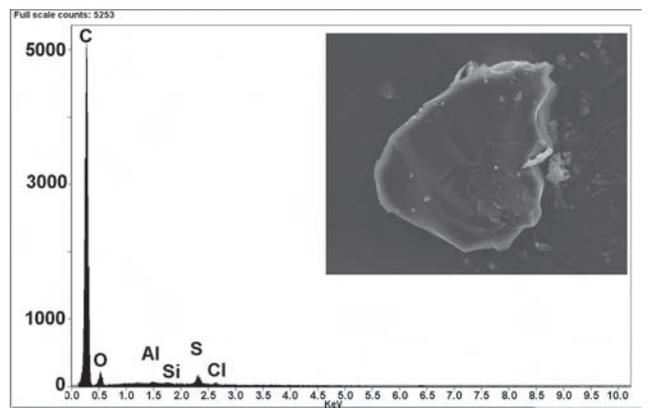


Figure 6. Bituminous coal, SEM.

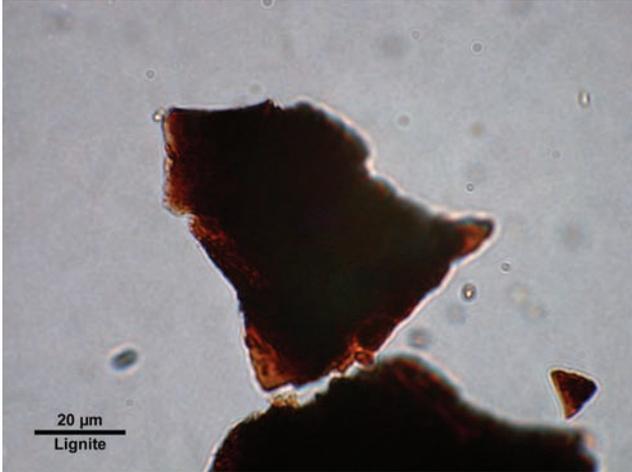


Figure 7. Lignite coal, transmitted light.

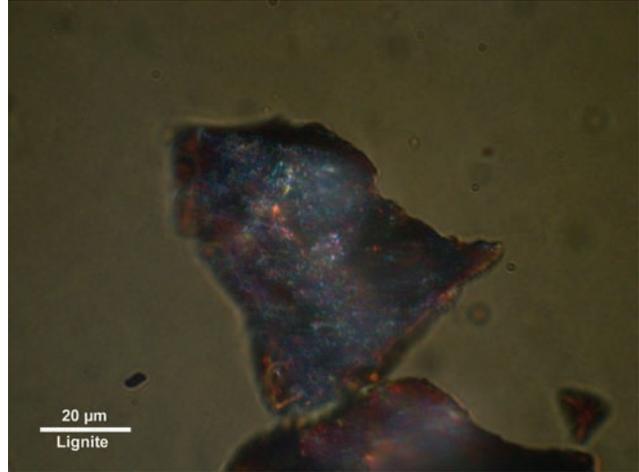


Figure 8. Lignite coal, reflected light.



Figure 9. Lignite coal, reflected light (stereomicroscope image).

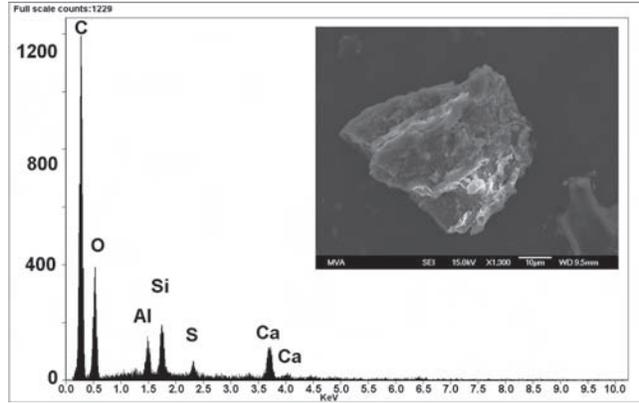


Figure 10. Lignite coal, SEM.



Figure 11. Peat, transmitted light.

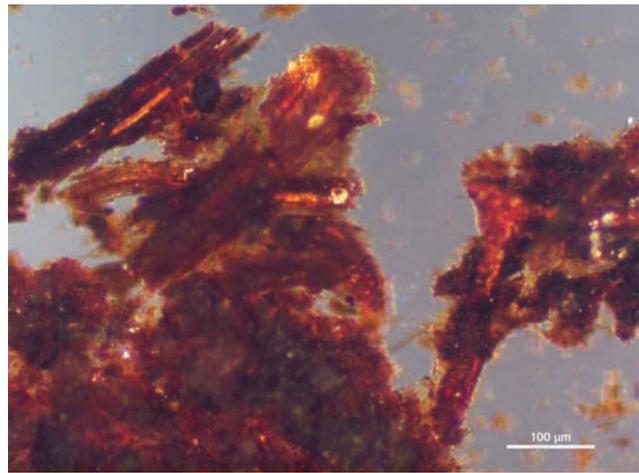


Figure 12. Peat, reflected light.

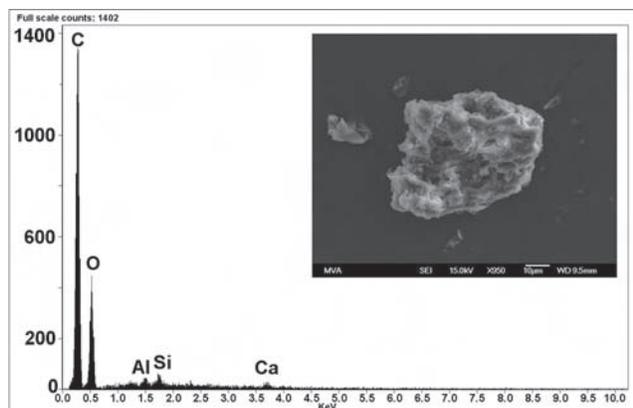


Figure 13. Peat, SEM.

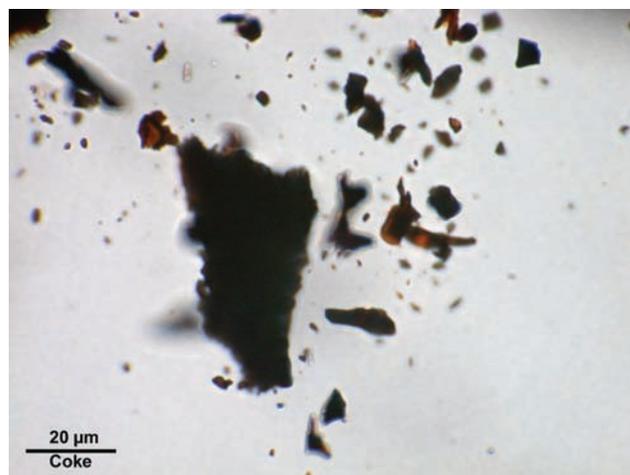


Figure 14. Petroleum coke, transmitted light.

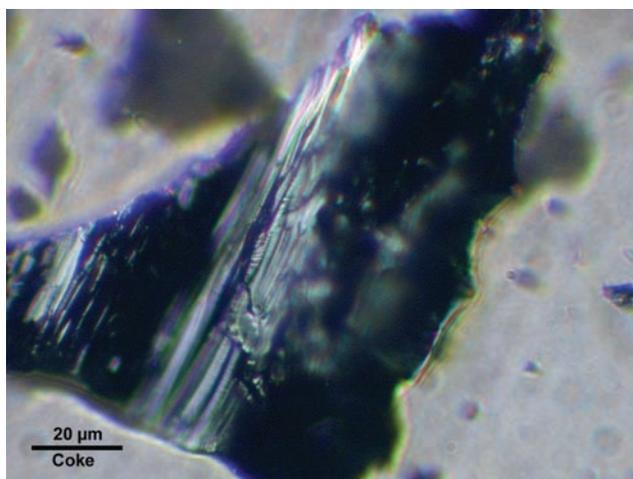


Figure 15. Petroleum coke, reflected light.

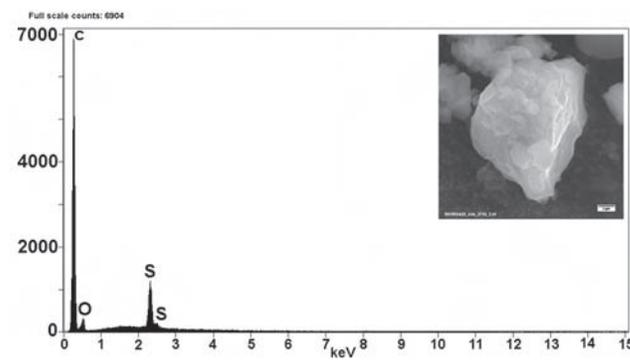


Figure 16. Petroleum coke, SEM.

This study adds additional images of reference coal and petroleum coke particles obtained by PLM, SEM and TEM as well as additional x-ray spectra (EDS). At the TEM level, the particles of coal show a wide range of morphologies and elemental compositions (Figures 17-20). The particles are generally solid, irregular shapes with thinning edges. The TEM-EDS spectra showed various combinations of Mg, Al, Si, S, Cl, K, Ca, Ti and/or Fe probably related to localized mineral inclusions in the small coal particles. The TEM-EDS spectra of petroleum coke showed primarily S as the main peak (Figure 21).

The Particle Atlas describes anthracite coal particles (*Atlas* no. 559) as “opaque, black to brownish black, smooth surface, conchoidal fracture, highly reflective, differentiated from bituminous by greater opacity.” The SEM-EDS elemental spectrum shows Al, Si, S, Mn

and Fe. This is generally consistent with our findings and the statement by Murray (8) that “coal does not have a fixed chemical composition.” The minor elements other than carbon found in coal arise primarily from minerals found as inclusions in the coal particles. The most prominent of those minerals and the elements they contribute are quartz (Si), calcite (Ca), pyrite (Fe, S), and clay minerals (Al, Si) (9).

The Particle Atlas describes bituminous coal particles (*Atlas* no. 560) as “translucent, reddish-brown if thin enough, brownish-black with dull to moderately high reflectivity. Surfaces slightly rough with occasional indications of origin in fibrous structure. Irregular chips have sharp edges and may show conchoidal fracture. Most thin edges show strain birefringence as well as occasional mineral birefringence.” The SEM-EDS elemental spectrum shows Al, Si, S, and Fe.

The Particle Atlas describes lignite coal particles (*Atlas* no. 561) as “showing woody structure. Rough surfaces with fibrous striations. Few sharp edges. Low reflectivity. Thin edges translucent reddish-brown by transmitted light, show strain birefringence. Brownish-black in reflected light. Little indication of conchoidal fracture.” SEM-EDS elemental spectrum shows S, Ca, and Fe.

The Particle Atlas describes petroleum coke particles (*Atlas* no. 492) as “highly reflective, smooth, black globules, tending to be spherical, comparatively smooth and rounded.” No SEM-EDS spectrum is provided.

The Particle Atlas describes coal coke particles (*Atlas* no. 496) as “irregular shape, porous, rough, some sharp edges, gray black in reflected light, may show fibrous structure if from bituminous coal (also vesicular, hackly).” No SEM-EDS spectrum is provided. It is assumed that the spectra for coal coke would be similar to that of coal, because only the volatile organics are driven off during the coking process and the elements detected by EDS would remain.

CONCLUSIONS

This study provides additional information for identifying coal and coke particles by microscopy that may be used to distinguish them from other particles found in dust. Bituminous coal particles and petroleum coke particles that exhibit similar morphology and reddish edge coloration with light microscopy can be differentiated on the basis of their elemental composition by SEM-EDS (Figures 22-24).

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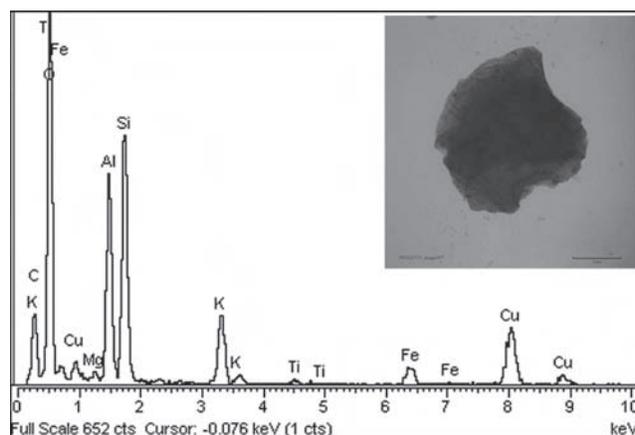


Figure 17. Anthracite coal, TEM.

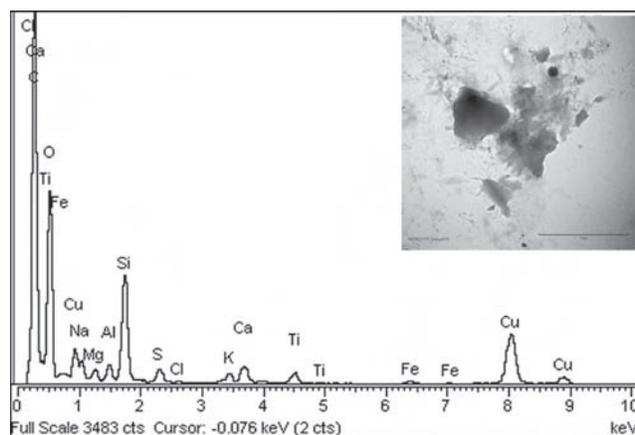


Figure 18. Bituminous coal, TEM.

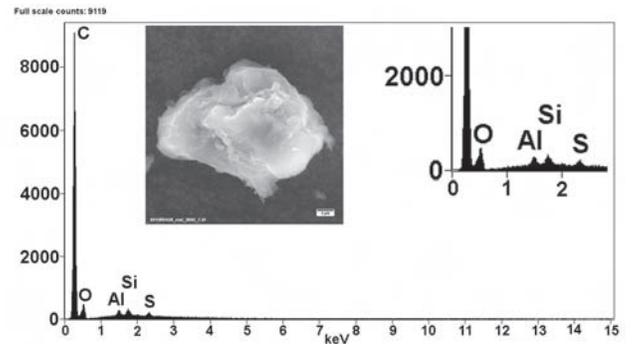
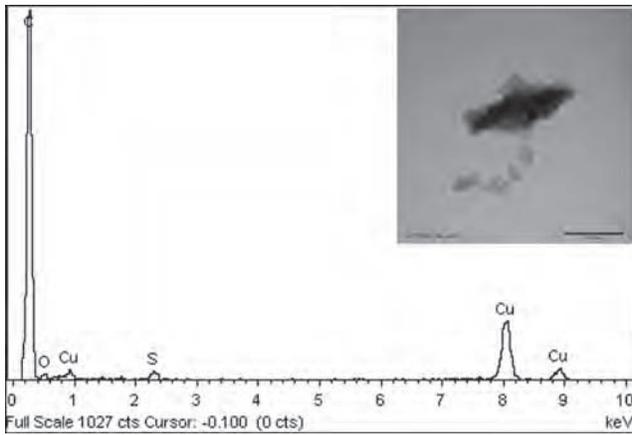
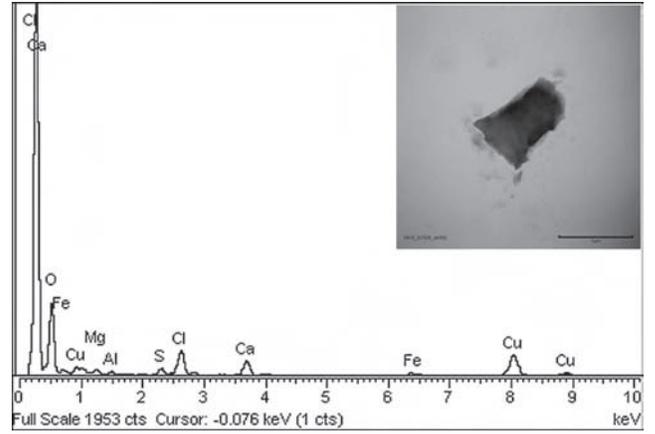
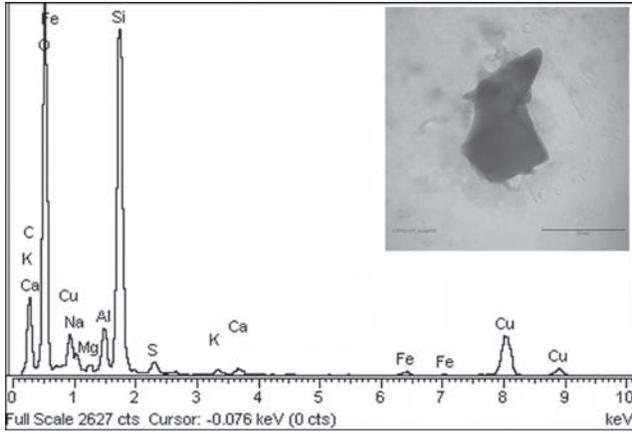


Figure 21. Petroleum coke, TEM.

Figure 22. NIST bituminous coal, SEM.

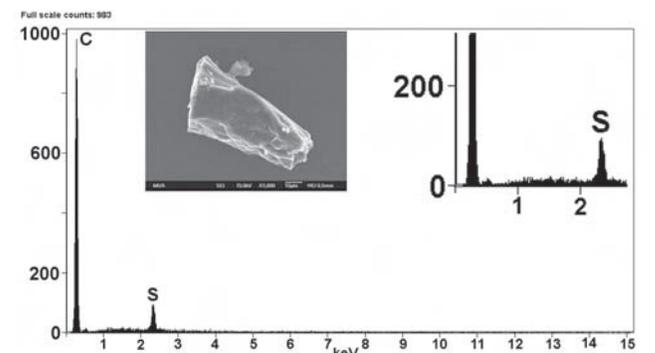
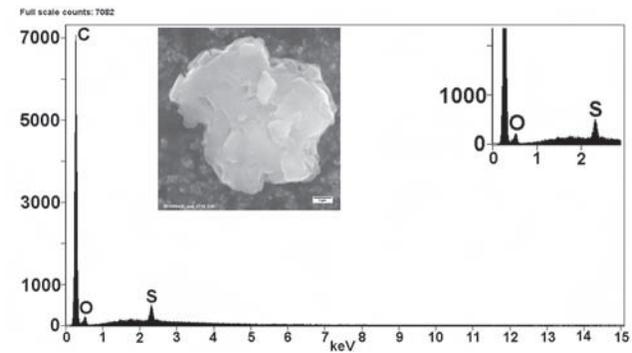


Figure 23. NIST petroleum coke, SEM.

Figure 24. Commercial petroleum coke, SEM.