Chemical Speciation of Trace Elements by X-ray Absorption Fine Structure Analysis.

Makoto Kobayashi, Central Research Institute of Electric Power Industry

Background

- Behavior of trace elements in coal and related materials is important in their use and environmental protection.
- What is the chemical form of such trace elements?
- We have to determine their chemical species by a nondestructive analysis.
- The trace analysis was quite difficult because the target species as well as matrix components are mixture of various compounds.

Objective

- Apply X-ray absorption fine structure (XAFS) analysis on speciation of As, Cr, and Se in the standard samples of coal and fly ash.
- Clarify the capability of the analytical procedure on the speciation of trace elements in coal and related materials.

Experiment

- Specimen

  standard bituminous coal, NBS1632c and standard fly ash, NBS1633b,

  both supplied from NIST.

- XAFS measurements, fluorescence XAFS performed at BL16B2 and BL16XU.

Table 1. Verified concentration of the trace elements in the standard sample of coal and fly ash.

Specimen	As	Se	Cr
NBS1632c	6ppm	1ppm	13ppm
NBS1633b	136ppm	10ppm	198ppm

Results and Discussion

- Valence of arsenic compounds were determined by XANES spectra of the specimens by comparing with pure arsenic compounds. (Figure 1)
- Proportion of the arsenic compounds with two different valences could be estimated by curve fit of the XANES spectra. (Figure 2)

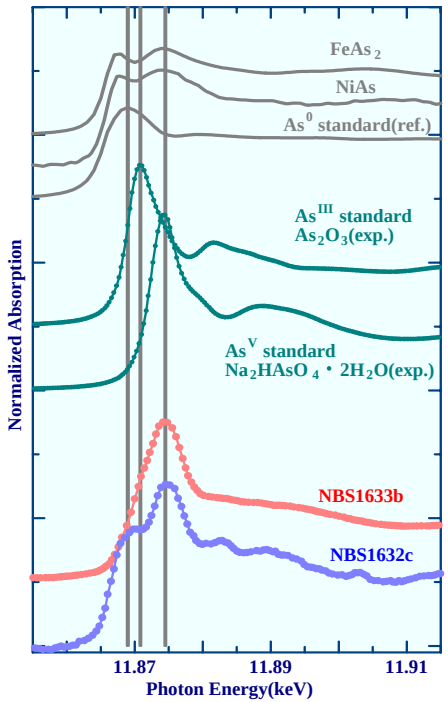

Figure 1. XANES spectra of arsenic contained in coal and fly ash.

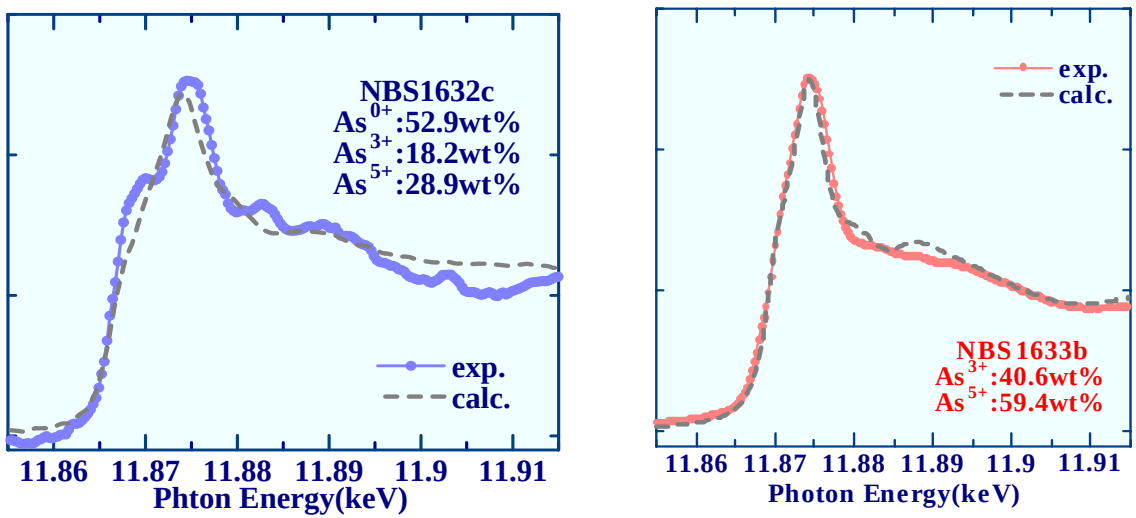

Figure 2. XANES spectra of arsenic contained in coal and fly ash and their curve fits assuming chemical form of the components.

- EXAFS analysis of the arsenic was able to determine atomic distance with neighboring oxygen atoms. (Figure 3)
- Trivalent chromium compounds was identified by XANES spectra. Chemical forms of the compounds were not clear because of weakness of the spectra. (Figure 4)

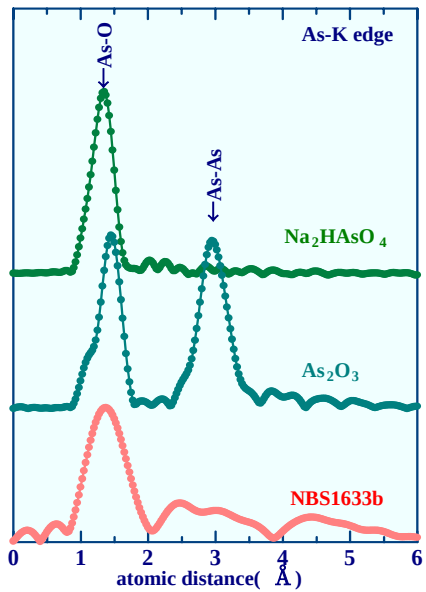

Figure 3. EXAFS spectra of arsenic contained in fly ash specimens.

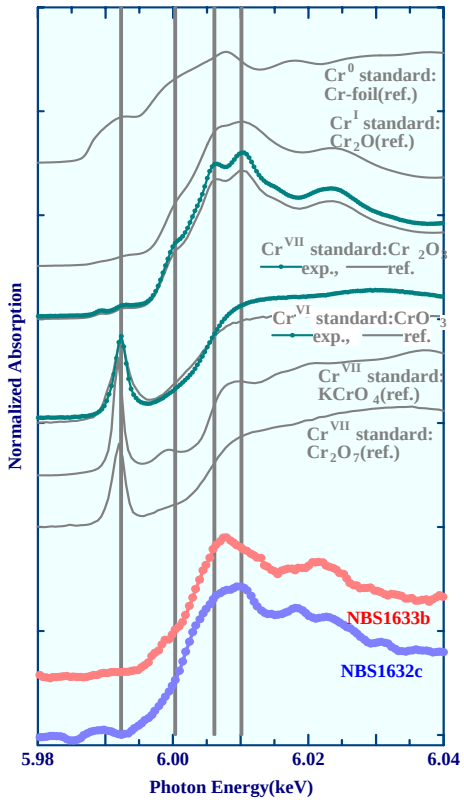

Figure 4. XANES spectra of chromium contained in the specimens.

- Selenium in NBS1633b might be mixture of tetravalent and hexavalent oxides. Selenium in NBS1632c exhibits compounds with low oxidation number. (Figure 5)

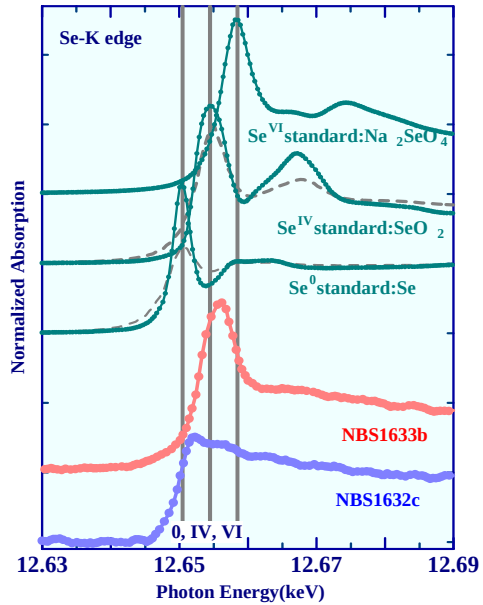

Figure 5. XANES spectra of selenium contained in the specimens.

Conclusion

- XAFS analyses using synchrotron X-ray from the bending magnet and the insertion device has capability to determine oxidative state of arsenic and selenium whose content were as low as a few ppm. (Figure 6)
- Estimation of chemical formulae of such compounds was also possible in some cases.

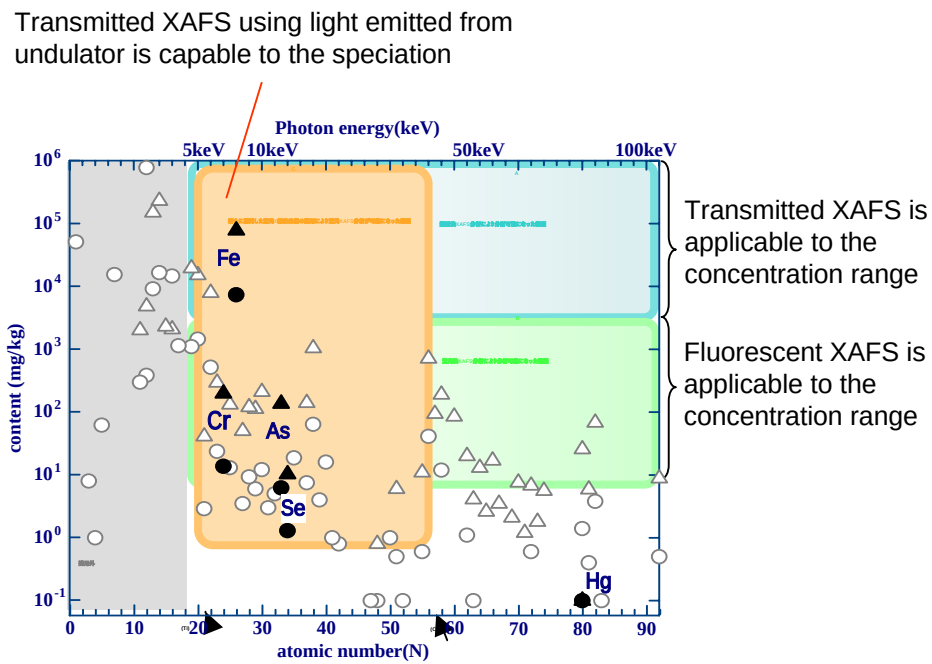

Figure 6. Capability of XAFS analysis on the speciation of various elements in typical bituminous coal and fly ash.