

Analysis of clay fraction in surface soils by laser induced breakdown spectroscopy

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[Introduction]

Laser induced breakdown spectroscopy (LIBS) is a technique for quantitative and qualitative elemental analyses by ablation of laser on sample surface. This technique can achieve simultaneous multi-element analysis under air environment. In forensic science, it is considered as an easy and rapid method, because a small amount of sample can be analyzed without complicated pretreatment.

For the purpose of application of LIBS to forensic geological examination, we studied analytical conditions for clay analysis. Samples were prepared by filter-membrane peel technique. The specimens should be analyzed without contamination of glass supporting samples. This technique enables elemental analysis after X-ray diffraction of oriented clay specimens and can be applied to analyze a small amount of soil and suspended materials of rivers and lakes. In addition, the principal component analysis (PCA) was applied to the results obtained by LIBS for evaluation of homogeneity of the clay films.

[Materials and methods]

Surface soils collected from farm land and residential area in Japan, and subsoil to substratum collected from Nokonoshima island in Fukuoka Prefecture. Samples were treated by hydrogen peroxide for decomposition of organic materials and clay fraction were separated by wet sieving using stainless steel mesh (53 μm). The fraction was treated by sodium dithionite for removal of iron. Suspension including 40 mg of clay was vacuumed on a piece of membrane filter (Merck-millipore, 0.45 μm pore size), and wet film of clay was moved onto a piece of a slide glass for analysis.

The specimens were analyzed by J200LIBS (Applied Spectra Inc.) for the data collection with Nd:YAG laser (wavelength 266 nm), laser power of 60 %, laser repetition rate of 10 Hz and the detection range from 185 to 1048 nm. First, spot size and gate delay were examined by spot analysis from 30 to 200 μm and from 0.25 to 1 μsec , respectively. And then, line analysis was conducted on three different lines of each sample. The scan speed was also examined from 0.1 to 10 mm/s.

Data analysis and PCA were conducted by data analysis software Aurora (Applied Spectra Inc.) on accumulated spectra except 266 nm (± 0.2 nm) of 20 points by 1 line.

[Result]

In this study, spot size of 100 μm , gate delay of 1 μsec and scan speed of 1 mm/s were suitable for the analysis of clay film without ablating supporting glass. In consequence, PCA plots showed only a small variation among 3 lines of the same samples, so that homogeneity of the samples for LIBS was suggested about line analysis. Further, analysis of soils from various geological background is required to determine the discriminability.

Keywords: laser induced breakdown spectroscopy