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Development of Heavy Minerals and Trace Heavy Elements Database of Soils from Japan by Using SR X-ray Analysis.

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Since Heavy minerals are an important component of soils, mineralogical examination is essential in forensic soil identification. Besides, trace elements of soils will usually reflect their geographical origin and has long been used as provenance indicator in forensic investigation. In this study, we aim to develop heavy minerals and trace elements database of soils that collected from 3024 sampling points across Japan by using Synchrotron Radiation (SR) X-ray analysis. The soil samples were supplied by the Geological Survey of Japan (AIST) and they were the same samples used to produce the geochemistry map of Japan. In our experiment, the heavy mineral composition of the soils were determined by using SR X-Ray Diffraction (SR-XRD) analysis while the trace heavy elements data were obtained by using High-Energy SR X-Ray Fluorescence (HE-SR-XRF) analysis. Both analyses were carried out at SPring-8. Since a very large number of soil samples need to be analyzed before creating the database, a high-through put measurement system with enough measurement speed and experiment efficiency is thus required. Recently, a highly automated system that allows us to analyze 30 samples continuously in one measurement run with the large Debye-Scherrer camera was installed at the SR-XRD experiment hutch of BL19B2 beam line by Osaka. Besides, an automated Sample X-Y stage that allows us to analyze 25 samples continuously in one measurement run was also installed to the HE-XRF experiment hutch of BL08W beam line. These newly automated systems were applied to our measuremenst and we have successfully analyzed a very large quantity of samples within a limited beam time (approximately 130 samples in 24 hours). This indeed, is the world first attempt of creating a large forensic database by using SR X-ray analytical techniques.

For the SR-XRD experiment, heavy mineral components of the soil samples were separated by using Tetrabromoethane with the specific gravity of 2.82. The heavy mineral fractions were then ground and filled into a glass capillary (0.3 mm). Measurements were carried out by using Debye-Scherrer method with a synchrotron X-ray beam of 1angstrom in wavelength at BL19B2 beam line. The exposure time was 10 minutes per sample. As for the HE-XRF experiment, the soil samples were ground into fine powder to obtain a homogeneous composition. Measurements were carried out by using a high energy X-ray beam with an excitation energy of 116keV at BL08W beam line. The measurement time was 10 minutes per sample.

As an example of the results, the interpretation of the heavy minerals and trace heavy element distribution in Kanto region is shown as below. The Kanto plain is covered with thick quaternary sediments including a large amount of volcanic ash derived from volcanoes to the west of the plain, while the Kanto Mountains is dominated by a complex mixture of metamorphic, granitoid and volcanic rocks. The XRD patterns of the heavy minerals of soils from Kanto plain show that they were dominated by clinoypyroxene, orthopyroxene and amphiboles, minerals of volcanic origin while the heavy minerals of the soils from Kanto mountain were dominated by a great variation of heavy mineral components which include epidote, amphibole, clinopyroxene and others. As for the HE-XRF data, the XRF spectra of the samples of Kanto plain also exhibited large differences in the heavy element compositions from those of the samples collected from Kanto mountain. Besides, there were also such cases that the SR-XRD patterns of the soils within an area were similar while the HE-XRF spectra differ. An example was given in Fig.1, which shows the XRF spectra of four different sampling points in Chiba area yielding similar XRD patterns. The sampling points of Chiba1 and Chiba2 have higher concentration of Hf and W than Chiba3 and Chiba4. This infers that in certain cases the HE-XRF data can provide further provenance information that SR-XRD data cannot provide, and vice versa.

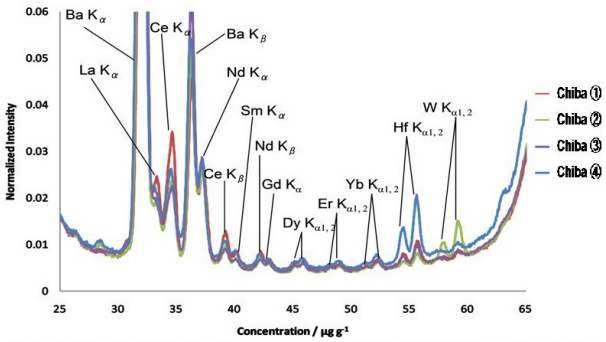

Fig.1 XRF spectra of four different sampling points (Chiba area)

Keywords: Synchrotron Radiation, soil, heavy minerals, heavy elements, XRF, XRD