SPECIATION OF LEAD ADSORBENTS IN WEATHERED GRANITE USING FIB-TEM TECHNIQUE

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Contamination of soil with toxic heavy metals (Pb, Cd, As, etc.) is one of serious worldwide environmental problems. However, identification of adsorbents of the heavy metals in actual soil is not easy because the density of the adsorbents is generally low and/or the adsorbents are very fine. Here we report application of the focused ion beam (FIB) sample preparation technique and subsequent TEM analyses for comprehensive analyses of Pb adsorbents in actual contaminated soil.

We selected soils originated from weathering of granite, which are common in Japan. X-ray diffraction showed the peaks ascribed to smectite, illite, kaolinite, quartz and feldspar. TG-DTA analysis indicated no organic substance detectable. The soil was dipped in a $Pb(NO3)_2$ solution, to make the concentration of Pb^{2+} in the soil c.a. 0.2 wt%. After drying, the soil was moulded into a pellet by pressing. Elemental mapping on the surface of the pellet by SEM showed that Pb exists discretely and frequently associated with Mn or Ti. Thin sections (˜10 mm wide by ˜5 mm deep) for TEM observation were fabricated from the Pb-concentrated areas, using Hitachi FB-2100 FIB with micro-sampling system. Up to now, three types of Pb adsorbents have been identified in the present study.

(1)Mn-Fe (hydr)oxide. The mineral phase could not be identified because the material is too beam-sensitive. Fe/Mn ratio is close to unity. This material often is associated with foliated fine smectite or intercalated with vermiculite-like phyllosilicates. Efficiency of Pb adsorption is very high, Pb/(Mn+Fe) atomic ratio is several %.

(2)Aggregates of anatase (TiO2) fine particles. Probably they are weathering product of the TiO2 component in the original granite. Anatase particles of ˜10 nm in diameter are attached with the same orientation. A considerable amount of Fe is also detected from the aggregate. Pb/Ti atomic ratio is ˜2%.

(3)Goethite (FeOOH) or ferrihydrite. Pb/Fe atomic ratio is ˜1%.

Considering the bulk composition of the soil and the above results, it is suggested that Pb2+ adsorbs mainly to these transition metal (hydr)oxides, not to silicate clay minerals.