

Speciation study in sediments using BCR sequential extraction method: a long-term stability of chemical forms

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The mobility, bioavailability and toxicity of metals in environmental samples such as stream sediments and soil substances change according to their chemical forms. Therefore, elemental speciation is informative for appropriate risk assessment in an environment. For reliable speciation analysis of various environmental samples, we have identified chemical species in Japanese geochemical reference materials using a sequential extraction procedure (SEP). However, it has not been elucidated yet whether stability of metal speciation in collected samples is preserved through drying and storage procedures. This work aimed to test a long-term stability of chemical forms in the materials and the effect of sample pretreatment on the result of the extraction fraction.

The various kinds of sediments such as stream sediment, coastal sea sediment, and soil were collected in 2010 for metal speciation study. Collected samples were treated with different procedures. One is immediately stored at 4°C after sampling, freeze-dried on the same day, and finally stored in a refrigerator. The other is air-dried for 14-21 days, and stored at ambient temperature away from direct sunlight. As a speciation analysis, we used BCR scheme that the three-step extraction procedure developed by the Community Bureau of Reference (Ure et al., 1993). The BCR protocol is designed to extract elements of the intended phases: acid soluble phase (carbonates) in step 1, reducible phase (Fe and Mn oxides) in step 2, and oxidizable phase (sulfides and organics) in step 3. The residue was digested using a mixture of HNO₃-HClO₄-HF solution and termed 'step 4' here.

The systematic changes of speciation of Cd, Cr, Cu, Ni, Pb, and Zn in sediments have been examined over the three years. The test demonstrated that the extracted results of Cd, Cr, Cu, Ni, Pb, and Zn in most samples are unchanged for three years. Exceptionally, metals extracted at the step 3 procedure systematically and significantly decreased for polluted samples and organic-rich samples. There was no significant difference in the results obtained among air-dried and freeze-dried samples. However, significant differences in the extraction results of Cd are found in these samples.