Goldschmidt 2000
September 3rd–8th, 2000
Oxford, UK.

Journal of Conference Abstracts
Volume 5(2), 902

Determination of Soil Selenium Speciation Using a New Extraction Methodology and Chemometric Data Analysis

Kevin Seed (k.j.seed@reading.ac.uk)[1], **Mark Cave** (m.cave@bgs.ac.uk)[2], **Joy Carter** (j.carter@derby.ac.uk)[3] & **Andrew Parker** (a.parker@reading.ac.uk)[1]

[1] Postgraduate Research Institute for Sedimentology, University of Reading, Whiteknights, Reading, RG6 6AB, UK
[2] Analytical Geochemistry Group, British Geological Survey, Keyworth, Nottingham NG12 5GG, UK
[3] School of Environmental & Applied Science, University of Derby, DE22 1GB, UK

Selenium (Se) is both an essential nutrient and a potentially toxic trace element. In the natural environment Se exists in +6, +4, 0 and -2 oxidation states. The Se^{+6} and Se^{+4} valence states form the oxyanions selenate (SeO_4^{2-}) and selenite (SeO_3^{2-}) / biselenite ($HSeO_3^-$), respectively. Se oxyanions are highly soluble and therefore bio-available and potentially toxic (e.g. White et al., 1991). The reduced forms, selenide (Se^{-2}) and elemental Se (Se^0), are insoluble under normal soil conditions and therefore have a reduced potential for bio-availability. Determining the chemical speciation of Se in soils/sediments and its association with various solid-phase constituents is therefore essential for understanding the potential for mobilisation, bio-availability, and toxicity of the element in natural systems.

Sequential chemical extraction is a commonly used method for determining the distribution of Se in soils and sediments (e.g. Fio & Fujii, 1990; Martens & Suarez, 1997). These methods are often modifications of more traditional procedures (e.g. Tessier et al., 1979) which involve the selective extraction of elements through the use of a specific reagent for each phase association. This type of methodology suffers from a number of limitations e.g. the methodological definition of the partitioning of the trace elements between solid phases, and the fact that reagents are not specific to one mineral phase so that the associated analysis is not a true representation of the amounts of an element extracted from a single phase (Cave & Wragg, 1997).

A new extraction scheme was developed for determining Se speciation and solid-phase partitioning in soils/sediments. Extractions were carried out in polypropylene centrifuge tubes with 0.45 μm filter inserts. 2 g of sample was accurately weighed into the filter tube insert. The extractants used were deionised water (extracts 1 & 2), 0.01 (extracts 3 & 4), 0.1 (extracts 5 & 6), 1 M HCl (extracts 7 & 8) and a 4 M HCl/HNO_3 mix (extracts 9 & 10). The samples were leached sequentially with two 10 ml aliquots of each extractant. Each sample was centrifuged for 10 minutes at 3000 rpm. A total ($HF/HClO_4/HNO_3$) digestion was then carried out to dissolve any residual material (extract 11). The whole extraction procedure was carried out in duplicate.

The 10 extractions and a total digest for each sample were analysed for major and trace elements (As, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, S, Si, Sr, V, Zn) by inductively coupled plasma- atomic emission spectrometry. Se analysis was carried out using hydride generation- atomic fluorescence spectrometry (HG-AFS). The selectivity of HG-AFS for Se^{+4} was used in combination with oxidative and reductive chemical reactions to determine Se^{+4} and the sum of Se^{+4} and Se^{+6} concentrations. Organic Se was determined by the subtraction of Se^{+4} and Se^{+6} concentrations from a total Se concentration determined using a total digestion. Factor Analysis (e.g. Malinowski, 1991) was applied to the leachate data. Figure 1 shows the three resolved components identified in the test sample using this method. The Se was found to be associated with component 1 which occurred in the residual fraction (extraction 11). Component 1 was composed predominantly of Al (54%) and Fe (26%) indicating that the Se was largely associated with insoluble Fe/Al oxyhydroxides The composition of the identified components (component 1 = Fe/Al phase, components 2 & 3 = Ca rich phases) agreed well with distinct mineral phases identified by XRD and SEM analysis of the same sample. The results of this procedure and its application to a number of different soil types will be presented.

Journal of Conference Abstracts
Volume 5(2), 903

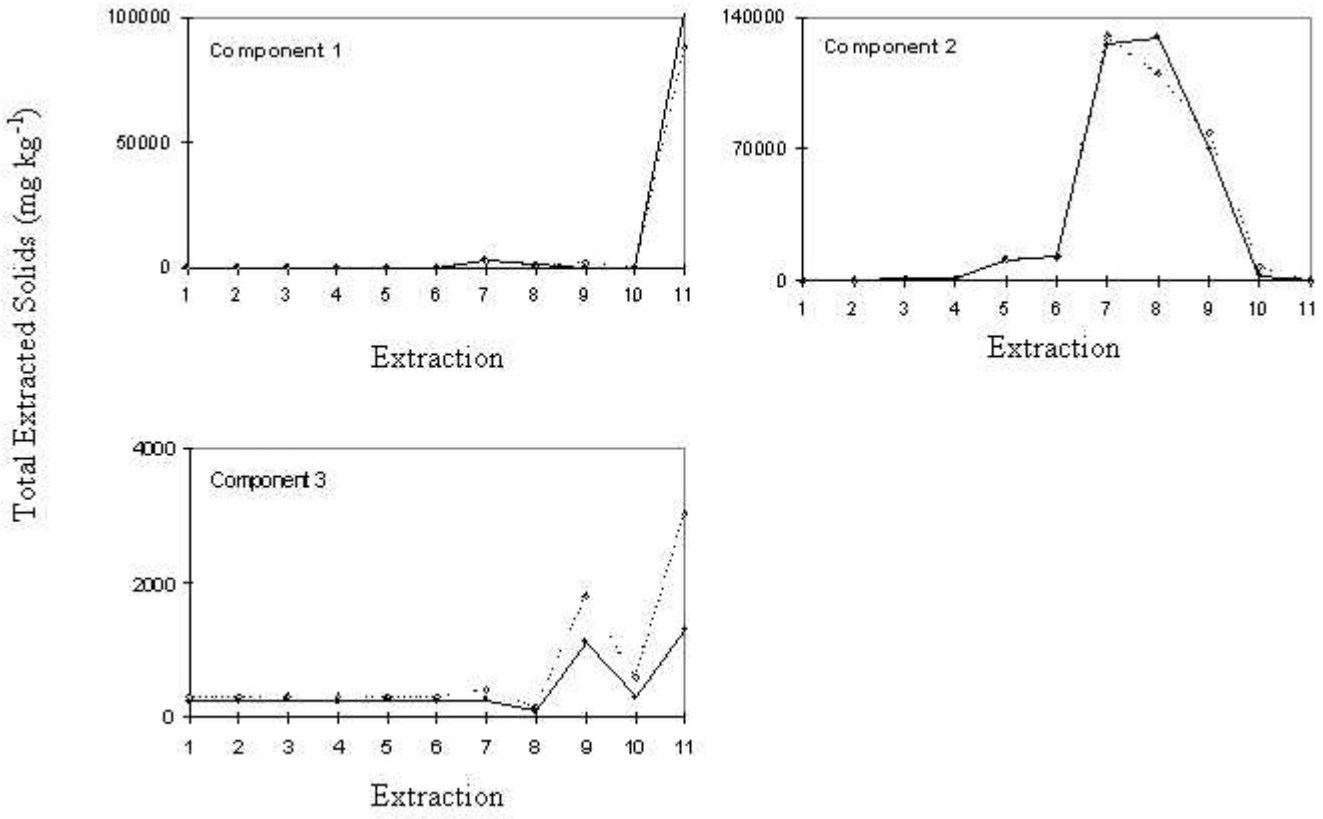

Figure 1. Resolved components in test soil (solid and dotted lines represent the results from duplicate extractions).

Cave MR & Wragg J, *Analyst*, **122**, 1211-1221, (1997).

Fio JL & Fujii R, *Soil Science Society of America Journal*, **54**, 363-369, (1990).

Malinowski ER, *Factor Analysis in Chemistry. John Wiley & Sons, Inc*, **2nd Edition**, (1991).

Martins DA & Suarez DL, *Environmental Science & Technology*, **31**, 133-139, (1997).

Tessier A, Cambell PGC & Bisson M, *Analytical Chemistry*, **51**, 844-850, (1979).

White AF, Benson SM, Yee AW, Wollenberg HA & Flexser S, *Water Resources Research*, **27**, 1085-1098, (1991).