



Effects of incubation time and filtration method on K_d of indigenous selenium and iodine in temperate soils



T. Almahayni ^{a,*}, E. Bailey ^b, N.M.J. Crout ^b, G. Shaw ^b

^a Biosphere Impact Studies, SCK-CEN, Belgian Nuclear Research Centre, Boeretang 200, B-2400 Mol, Belgium

^b School of Biosciences, University of Nottingham, Sutton Bonington, LE12 5RD, UK

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ABSTRACT

In this study, the effects of incubation time and the method of soil solution extraction and filtration on the empirical distribution coefficient (K_d) obtained by de-sorbing indigenous selenium (Se) and iodine (I) from arable and woodland soils under temperate conditions were investigated.

Incubation time had a significant soil- and element-dependent effect on the K_d values, which tended to decrease with the incubation time. Generally, a four-week period was sufficient for the desorption K_d value to stabilise. Concurrent solubilisation of soil organic matter (OM) and release of organically-bound Se and I was probably responsible for the observed decrease in K_d with time. This contrasts with the conventional view of OM as a sink for Se and I in soils. Selenium and I K_d values were not significantly affected by the method of soil solution extraction and filtration.

The results suggest that incubation time is a key criterion when selecting Se and I K_d values from the literature for risk assessments. Values derived from desorption of indigenous soil Se and I might be most appropriate for long-term assessments since they reflect the quasi-equilibrium state of their partitioning in soils.

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1. Introduction

Sorption is a generic term encompassing processes such as adsorption, ion exchange and precipitation. It controls, to a large extent, the transport and accumulation of substances in the environment. The empirical distribution coefficient (K_d) is often used in transport and risk assessment models (e.g. Almahayni, 2014) to aggregate many poorly understood or multi-parameter sorption processes.

K_d values are periodically published and updated (e.g. IAEA, 2009; IAEA, 2010). However, published K_d data vary by orders of magnitude between soils. This large variation has been partially attributed to the lack of a consistent experimental methodology. Degryse et al. (2009) noted that, depending on the methods used to characterise the solid and liquid phases, different K_d values may be obtained. Additionally, the lack of a consistent methodology complicates the comparison of K_d data and may cause confusion when interpreting and applying K_d values in risk assessment models. Factors such as solid-to-liquid ratio, contact time and the extraction

and filtration of soil solution are among the main sources of methodological variation between studies determining K_d values.

The solid-to-liquid ratio varies considerably between K_d determination methods. For example, in their review of the sorption coefficients of a suite of elements on iron hydroxides, Li and Kaplan (2012) reported solid-to-liquid ratios that varied over two orders of magnitude. This variation could affect K_d as indicated by Sheppard et al. (2007) who noted that K_d values from batch experiments with a 1:10 soil:water ratio were higher than those from soils incubated at field capacity. As K_d is typically used to estimate retention against leaching, Sheppard et al. (2007) suggested that it is preferable to measure K_d at moisture contents close to, or slightly above, field capacity because that is when leaching occurs. In any case, K_d values should be determined using a solid-to-liquid ratio that is as representative as possible of natural conditions (Limousin et al., 2007).

Contact time between the solid and liquid phases during a K_d experiment often varies between hours and weeks (e.g. Vandenhove and Van Hees, 2007; Sheppard et al., 2009). Short contact times may be ineffectual for measuring the effects of slow (de)sorption mechanisms on K_d . For instance, Ashworth and Shaw (2006a) observed a two order of magnitude increase in ^{125}I K_d

* Corresponding author.

E-mail address: tamahain@sckcen.be (T. Almahayni).

during a 7-week mini-column experiment with spiked soils. Recent studies (e.g. Shetaya et al., 2012; Tolu et al., 2014b) have revealed ongoing changes in fractionation and speciation of soil trace elements over periods longer than typical incubation times in short-term K_d experiments.

Of the available methods to extract soil solution, centrifugation is probably the most widely used in K_d experiments (e.g. Xu et al., 2015; Tolu et al., 2014a). 'Rhizon' soil moisture samplers have also been used (e.g. Ashworth and Shaw, 2006c; Ashworth et al., 2008). The dissolved phase is operationally defined by the pore size of the membrane used to filter soil solution. Filters of 0.45 μm or 0.22 μm are commonly used to discriminate the dissolved phase from suspended particles.

This study focused on selenium and iodine, both of which have important stable and radio-isotopes. From a non-radiological perspective, deficient or excessive intake of either Se or I can result in serious human or animal health problems (Chen, 2012; Lei et al., 2011; Zimmermann et al., 2008). From a radiological perspective, the radioactive isotopes of Se (^{79}Se , $t_{1/2}$ of 3.27×10^5 years) and I (^{129}I , $t_{1/2}$ of 1.61×10^7 years) could give rise to additional radiological exposure to humans and non-human biota following their release into the environment (e.g. from waste repositories).

The objectives of this study were to:

- obtain K_d values for Se and I by de-sorbing indigenous Se and I from soils under realistic moisture conditions;
- evaluate the effects of incubation time and the method of soil solution extraction and filtration on K_d values of indigenous Se and I.

2. Materials and methods

2.1. Soil sampling and soil characteristics

Soil samples were collected from an arable field and an adjacent mature deciduous woodland strip in the Sutton Bonington area, Leicestershire, UK (52°49'48"N, 1°14'23"W). The soils belong to the Wick Soil Series (based on fluvio-glacial sand and gravel overlying Triassic Keuper Marl) with a sandy loam texture. Further details about the study area can be found in Ahmed et al. (2014) and in Shetaya et al. (2012). Soil samples were taken from surface (0–10 cm) and subsurface (40–50 cm) layers of the soil column. All samples were air-dried and sieved (<2 mm) prior to analysis. Selected characteristics of the soils are shown in Table 1.

2.2. Soil incubation experiments

Soil microcosms were prepared as described by Sheppard et al. (2009). Approximately 60 g of air-dried, sieved soil were weighed into a plastic syringe body. Deionised water was slowly added to the syringe to bring the soil to field capacity. The final moisture content at field capacity was approximately 30% for the arable topsoil, subsoil and the woodland subsoil and 45% for the woodland topsoil. All syringes were sealed with a parafilm membrane, to reduce moisture evaporation, and incubated at 10 °C in a temperature-controlled room awaiting sampling.

At the end of the incubation time, the microcosm contents were transferred to a polyoxymethylene (Acetal) centrifuge tube fitted with 20 μm stainless steel mesh filter (Di Bonito et al., 2008) to extract soil solution. These tubes were specially manufactured to fit a Beckman J2-21 refrigerated high speed centrifuge with AJ-10 rotor ($r_{\text{max}} = 158$ mm). The centrifuge buckets were spun at a relative centrifugal field value of $600 \times g$ for 30 min at 4 °C.

Centrifugal force pushed the liquid through the stainless steel mesh down to a collection cup. The supernatant was then removed from the collection cups and passed through the appropriate filter.

Selenium and I K_d values determined after 1, 2, 3 and 4 weeks of incubation were compared to investigate the effect of incubation time on K_d . For this experiment, duplicate microcosms (32 in total) were prepared for each combination of soil type and incubation time. Each week, 8 microcosms (2 per soil type) were sampled and their soil solution was extracted by centrifugation then passed through a 0.45 μm filter (Millex[®] Millipore, Watford, UK) and preserved ready for analyses as described in section 2.3.

Selenium and I K_d values derived from dissolved Se and I concentrations measured in 0.45 μm , 0.22 μm and 0.15 μm soil solution filtrates were compared to investigate the effect of filtration on the K_d of these elements. For this experiment, duplicate microcosms (16 in total) were prepared for each combination of soil type and the 0.22 μm and 0.15 μm filtration levels. The microcosms were incubated for 1 week only, after which the soil solution from the 0.22 μm treatment microcosms was extracted by centrifugation and passed through 0.22 μm Millex[®] Millipore filters. Soil solution from the 0.15 μm treatment microcosms was extracted and filtered using Rhizon soil moisture samplers (Eijkelkamp, Netherlands). The K_d data derived from this experiment were combined with K_d data obtained from the 0.45 μm treatment after 1 week of incubation.

Eh was measured in all microcosms immediately prior to soil solution extraction using a calibrated combination electrode (Thermo Scientific ORP glass/platinum electrode) and a high impedance pH/Eh meter. The calibrated electrode was inserted half-way through the wet soil and the reading allowed to stabilise before recording. All values were corrected by adding the standard potential (185 mV) of the combination electrode to the recorded raw potentials.

2.3. Chemical analyses

For the determination of total elemental composition, samples of the sieved soils (<2 mm) were ground in an agate planetary ball mill before acid digestion. Approximately 250 mg of finely ground soil was digested in PFA Teflon vessels with 2.5 mL hydrofluoric acid (HF; 40% Analytical Reagent), 2 mL nitric acid (HNO₃; 70% Trace Analysis Grade), 1 mL perchloric acid (HClO₄; 70% AR) and 2.5 mL H₂O on a 48-place Teflon-coated graphite block digester.

Total soil I was extracted with tetra methyl ammonium hydroxide (TMAH) from finely ground soil samples following the method of Watts and Mitchell (2009).

Soil and soil solution Se, I and multi-elements were determined by ICP-MS (Model X-Series^{II}, Thermo-Fisher Scientific, UK) on the digests after dilution in MQ water. The ICP-MS was operated in collision cell mode (to reduce polyatomic interferences) with hydrogen as the cell gas for Se analyses and 7% hydrogen in helium for the multi-element analyses. For Se analyses, ⁶⁹Ga, ¹¹⁵In and ¹⁹³Ir, in 2% HNO₃ and 4% methanol, were used as internal standards (methanol was added to improve the ionisation efficiency, stability and sensitivity of the instrument). For the multi-element analyses,

Table 1

Major characteristics of the arable and woodland soils. Iron and Se were determined in acid extracts, whereas I was determined in TMAH extracts.

	pH	OC %	Fe mg kg ⁻¹	Se mg kg ⁻¹	I mg kg ⁻¹
Arable topsoil	7.3	2.24	10382	0.27	4.87
Arable subsoil	6.8	0.79	8517	0.20	2.35
Wood topsoil	4.3	10.1	7475	0.33	4.41
Wood subsoil	3.8	1.66	7121	0.10	1.98

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