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Adsorption Sequence of Toxic Inorganic Anions on a Soil

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Abstract The adsorption of six anions (arsenate(As(V)), chromate(Cr(VI)), fluoride(F(−I)), molybdate(Mo(VI)), selenate(Se(VI)), and selenite(Se(IV))) on an andosol and the concomitant H^+ consumptions together with the sorption were investigated to understand the behaviors of toxic inorganic anions in environment. Based on the estimated adsorption maximum by the simple Langmuir equation, the order of adsorption affinity to the andosol at a suspension pH of 5.0 was $F(-I) >> phosphate(P(V)) > Se(IV) > Mo(VI) \geq As(V) >> Se(VI) \geq Cr(VI) \geq nitrate(N(V))$. The order of the concomitant H^+ consumptions together with the adsorption was $F(-I) >> P(V) > Se(IV) > Mo(VI) \geq As(V)$, while they were not observed in the adsorption of Se(VI), Cr(VI) and N(V).

Keywords Anion adsorption sequence · Selenite · Chromate · Fluoride

The problem of high concentrations of arsenic, selenium, chromate and fluorine contained in ground waters and soils have often occurred in various locations of worldwide (e.g. Losi et al. 1994; Chen et al. 1994; Corwin et al. 1999; Smedley and Kinniburgh 2002). Elements such as arsenic and fluorine exist mainly as anions under ordinary environmental oxidation conditions. The roles of soil in influencing the behaviors of pollutants are divided into (1) adsorption on a variety of soil constituents and (2) decompositions by soil microorganisms. In particular, it is essential to consider the adsorption or fixation of the anions by soil constituents to understand their behaviors in soil. The selectivity sequence type studies are useful to help understand aqueous metal–mineral sorption behavior (Puls and Bohn 1988). Many researchers have reported adsorptive sequences of heavy metal cations by soil specific minerals such as phyllosilicates (Saeki 2005) and amorphous Fe hydroxides (Kinniburge et al. 1976). However, there are few studies on the adsorptive affinity sequences of anions by soil or soil constituents (Parfitt 1978; Neal 1990).

Some anions are adsorbed by soil particle surfaces by ligand exchange, forming inner-sphere complexes, and some anions are attracted as an ion pair, forming outer-sphere surface complexes with water molecules between the anions and the soil surface (Sposito 1989). In the former case, one can, to some extent, observe the extend to which the ligand exchange reactions occur in the anion adsorptions by determining the concomitant hydrogen ion (H^+) consumption in the suspensions upon reaction. To understand the anion adsorption on soil for brief periods, our study reports on the adsorptions of As(V), Cr(VI), F(−I), Mo(VI), Se(VI), and Se(IV) by an allophanic andosol from Japan and the concomitant H^+ consumption that occurs as the adsorption process proceeds. The investigations of N(V) and P(V) adsorptions were also carried out as comparative data.

Materials and Methods

This study used a representative allophanic andosol taken from the Tama farm of the University of Tokyo in Nishi-Tokyo city. This soil was classified as Typic Meranudands (Saeki and Matsumoto 1992). The relevant physicochemical data for this soil is listed in Table 1. The soil sample was air-dried and sieved (<0.5 mm). The positive charge

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Table 1 Chemical and physical properties of the soil

Location	Tokyo, Japan
Taxonomy	Typic Meranudands
Particle size distribution	
Clay	23.9%
Silt	27.1%
Sand	49.0%
pH (H_2O)	5.3
pH (KCl)	4.5
EC	0.098 mS cm^{-1}
Total carbon content	8.3%
Specific surface area	215 m^2 g^{-1}
Acid oxalate extractable	
Al	4.9%
Fe	3.3%
Si	1.3%

of the soil determined with Cl^- was 32.2 molc kg^{-1} at pH 5.0 in the suspension. This soil was used as a sample to evaluate the anion adsorption behaviors because the soil can sorb a larger amount of selenite than other soils (Saeki and Matsumoto 1994).

Twenty mL of 12.5 mM $NaClO_4$ was added to a 1 g aliquot of soil. The suspension was adjusted to pH 5.0 by the addition of either NaOH or $HClO_4$ after 1 day of standing. To the suspension was added 5 mL of a known concentration of each anion solution adjusted to pH 5.0 by $HC1O_4$. The anion solutions were prepared from analytical grade $NaAsO_2$, Na_2HAsO_4 $7H_2O$, Na_2CrO_4 $4H_2O$, Na_2MoO_4, NaH_2PO_4, Na_2SeO_3, Na_2SeO_4, $NaNO_3$, and NaF supplied by Wako Chemicals. The soil solution ratio was 1:25. The reaction time was 6 h with continuous stirring and the suspension pH was maintained at 5.00 ± 0.02 by automatically adding $HClO_4$ as required. The experiment was conducted in an air-conditioned room at 298 ± 1 K. The suspension was centrifuged at 2100*g* and the supernatant was filtrated through a 0.22 μm Millipore filter. The filtrate was analyzed for each anion as follows: As(V), Se(VI), and Se(IV) by atomic absorption spectrophotometry: Cr(VI), Mo(VI), and P(V) by the vanadomolybdate method and F(−I) and N(V) by ion chromatography. The amount of H^+ consumed was estimated from the amount of $HClO_4$ required to maintain the pH of the suspension at 5.00 ± 0.02. The amounts of anions sorbed were calculated from the differences between the amounts of the added anions and those remaining in the solutions.

The speciation of each anion in the method condition of the present study was estimated by the thermodynamic equilibrium code SOILCHEM (Sposito and Coves 1988).

The adsorptive affinity of each anion to the soil was determined with the maximum adsorption (A_{max}) from the Langmuir equation, shown below,

$$A = \frac{k \cdot A_{max} \cdot C}{1 + k \cdot C} \tag{1}$$

where A is each anion's adsorption, k is an adsorptive equilibrium constant, and C is each anion's equilibrium concentration. From Eq. 1, we then obtain,

$$\frac{C}{A} = \frac{1}{k \cdot A_{max}} + \frac{1}{A_{max}} \cdot C \tag{2}$$

The maximum adsorptions can be approximately calculated by the application of the empirical adsorptive data to Eq. 2.

Results and Discussion

SOILCHEM estimated that most of As(V), Cr(VI), F(−I), Mo(VI), N(V), P(V), Se(VI) and Se(IV) exist with a concentration range of 0.0001–0.1 M at pH 5.0 in 0.01 M $NaClO_4$ background solution as a free ion or a hydrogen bond ion, F^- (>99%), $H_2PO_4^-$ (>94%), $HSeO_4^-$ (>99%), and $H_2AsO_4^-$ (>95%). Se(IV) is dominated by $HSeO_3^-$ (56–89%) and $NaHSeO_3$ (10–40%), and Mo(VI) by MoO_4^{2-} (87–89%) and $HMoO_4^-$ (11–13%). In aerial aquatic and soil environments, inorganic chromium is mainly dominated by Cr(III) and Cr(VI) (Losi et al. 1994), and inorganic arsenic by As(III) and As(V) (O'Neill 1990). Cr(III) and As(III) exist as $Cr(OH)^{2+}$ and $HAsO_2^0$, respectively, at pH 5.0 in 0.01 M $NaClO_4$ background solution, as estimated by SOILCHEM. Therefore, Cr(III) and As (III) were not investigated in this study.

The anion adsorptions by the soil are shown in Fig. 1. The apparent adsorption of N(V) was negative. This is caused by no adsorption of N(V) due to the competition with ClO_4^- added as the background salt and the increased N(V) concentration in the suspension due to the desorption of N(V) existing naturally in the soil by the added ClO_4^-. The amount of N(V) extracted by 25 mL of 0.01 M $NaClO_4$ solution from the soil was 25.5 mmol kg^{-1}. The N(V) desorption in the experiment nearly coincided with this extractable N(V) amount. The amounts (mmol kg^{-1}) of the other elements extracted only by the $NaClO_4$ solution from the soil were negligible, As < 0.003, Cr < 0.015, F < 0.03, Mo < 0.03, and Se < 0.003.

The adsorption of all the anions except for N(V) increased with the equilibrium concentrations. Nearly all of the fluoride (>90%) added to the suspensions was removed from the solution by the soil particles. The sorption of Se(IV) was larger than those of As(V) and Mo(VI), and smaller than that for either P(V) or F(−I). The adsorptions of Se(VI) and Cr(VI) were much smaller than those of Se(IV) and P(V). Between two oxyanions of the same element, Se, the amounts of Se(VI) adsorbed were clearly less than those of Se(IV). A similar result was observed

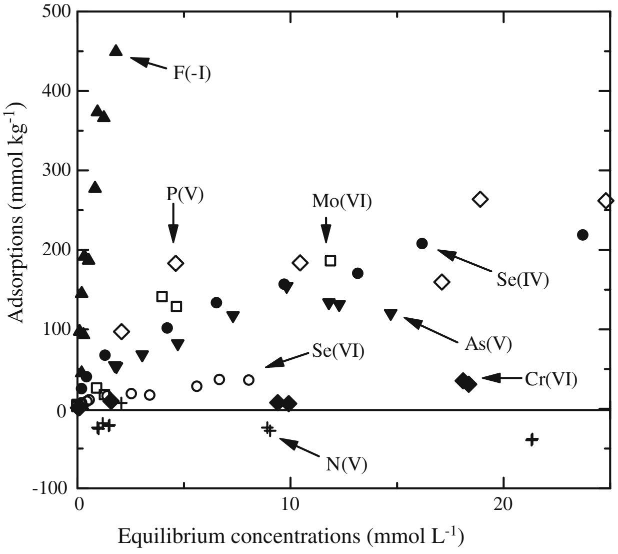

Fig. 1 Adsorption of anions by the andisol pH = 5.00 ± 0.02; soil sample = 1.0 g; solution = 25 mL of 10 mM NaClO4; ▲: F(−I), ◇: P(V), ●: Se(IV), □: Mo(VI), ▼: As(V), ○: Se(VI), ◆: Cr(VI), +: N(V)

even in fluvisol, cambisol and acrisol (Saeki and Matsumoto 1992). Neal and Sposito (1989) were unable to detect any significant Se(VI) adsorption in the alluvial soils obtained from California. Barrow and Whelan (1989) revealed that Se(IV) was adsorbed to a greater extent than Se(VI) in the soil collected from Western Australia.

The maximum adsorptions were approximately calculated with statistical significance by the application of the empirical adsorptive data to the Langmuir equation shown in Table 2. Only the estimation of Mo(VI) maximum adsorption was accepted with no statistical significance. The positive charge (32.2 mmolc kg^{-1}) determined with Cl$^-$ at pH 5.0 was similar to the estimated adsorption maximum of Se(VI), and it was higher than the adsorption maximum of Cr(VI). Judging from the estimated adsorption maximum, the order of adsorption affinity to the andosol was F(−I) > > P(V) > Se(IV) > Mo(VI) ≥ As(V) > > Se(VI) ≥ Cr(VI) ≥ N(V). The adsorption of inorganic anions by hydrous ferric oxide gel from 0.1 M NaClO$_4$ at pH 6.5 decreased in the order P(V) > As(V)=Se(IV) > silicate (Si(IV)) > Mo(VI) > sulfate (S(VI)) > Se(VI) > chloride (Cl(−I))=N(V) (Ryden et al. 1987). The orders of inorganic anion adsorptions were as follows: As(V) > P(V) > As(III) > Si(IV) > HCO$_3^-$ for Fe hydroxides (Meng et al. 2002) and P(V)=As(V) > Mo(VI) for goethite and gibbsite (Manning and Goldberg 1996). Parfitt (1978) reported the order of anion sorption for soil as P(V) > As(V) > Se(IV)=Mo(VI) > S(VI) > Cl(−I) > N(V). Roy et al. (1989) characterized the adsorption on the three soils in the order, P(V) > As(V) > Mo(VI). Neal (1990) summarized the order of adsorption affinities of anions to soil and soil components from the various data as follows: P(V) > As(V) ≥ Se(IV) ≥ Si(IV) > > S(VI) ≥ Se(VI) > N(V) > Cl(−I). Both these results implied that these anions could be divided into two groups according to their adsorptive affinities. The former group with high adsorption affinity included F(−I), P(V), As(V) and Se(IV), whereas the latter group with low affinity included Se(VI), Cr(VI) and N(V).

The consumption of' H$^+$ as a function of the anion adsorptions is shown in Fig. 2. The H$^+$ consumptions depend on the anion species. Any H$^+$ consumptions in the suspensions were not observed for N(V), Cr(VI) and Se(VI) adsorptions. The H$^+$ consumptions increased with the increments in the adsorptions of F(−I), P(V), As(V), and Se(IV) on the soil. In particular, H$^+$ consumption increased linearly with the amount of F(−I) sorbed on the soil and was obviously larger than the others. The ratios of H$^+$ consumed to the sorption of F(−I) ranged from 0.68 to 0.78. The H$^+$ consumptions in the sorptions of Se(IV), As(V), and Mo(VI)

Table 2 Each anion's adsorption maximum (*A*max) estimated with the simple Langmuir equation

Anions	Estimated Amax	Correlation coefficient *r*	Significance	Data No
As(V)	171	0.9336	$p < 0.001$	n = 9
Cr(VI)	18	0.6599	$p < 0.05$	n = 10
F(−I)	1080	0.8974	$p < 0.01$	n = 11
Mo(VI)	158	0.4597	$p < 0.3$	n = 9
P(V)	277	0.8974	$p < 0.01$	n = 7
Se(IV)	221	0.9737	$p < 0.001$	n = 9
Se(VI)	38	0.9047	$p < 0.001$	n = 10

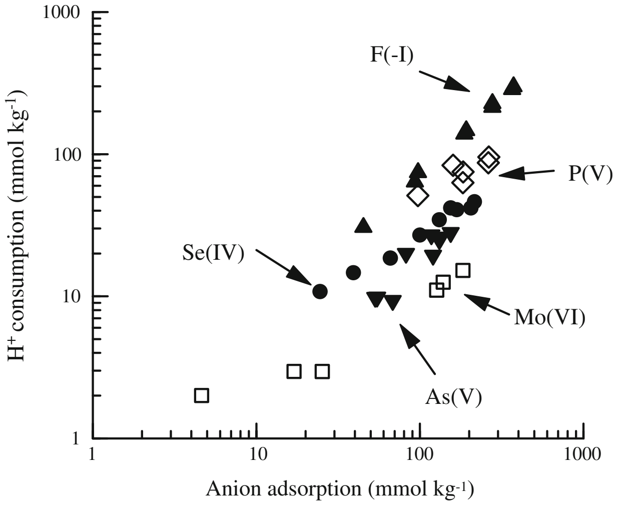

Fig. 2 Concomitant H$^+$ consumption with the anion sorptions at pH 5.00 ± 0.02. ▲: F(−I), ◇: P(V), ●: Se(IV), □: Mo(VI), ▼: As(V)

were considerably smaller than those in either P(V) or F(−I) sorption. The ratios of H^+ consumed to the Se(IV) and Mo(VI) sorptions decreased with the increases in the sorptions from 0.429 to 0.20 and from 0.43 to 0.08, respectively. In the case of As(V), the concomitant H^+ consumptions increased proportionally with the sorptions, with constant ratios ranging from 0.15 to 0.24. The H^+ consumption was also observed in the As(V) adsorption on ferrihydrite at pH 4.6 (Jain et al. 1999). The order of the concomitant H^+ consumptions together with the adsorptions was F(−I) > > P(V) > Se(IV) > As(V) ≥ Mo(VI). This order was the same as the former part of the adsorption affinity shown above, wherein the anions could be sorted to two groups, with or without H^+ consumption in their adsorptions.

In the present study, for anion adsorption by the soils, the following four reactions including ligand exchanges would be conceptually suggested:

$$X{-}OH_2^+ + L^- = X{-}OH_2^+ \blacklozenge L^- \quad (3)$$

$$X{-}OH_2^+ + L^- = X{-}L + H_2O \quad (4)$$

$$X{-}OH + H^+ + L^- = X{-}L + H_2O \quad (5)$$

$$X{-}O^- + 2H^+ + L^- = X{-}L + H_2O \quad (6)$$

where X is a metal atom such as Al or Fe in a skeleton of oxide particles, L^- is an anion such as $HSeO_3{}^-$, $H_2PO_4{}^-$, and F^- based on the dissociation constants, and ◆ is a water molecule. The adsorptions of Se(VI) and Cr(VI) on the soil sample were much smaller than the other anions such as P(V) and Se(IV) and did not induce any concomitant H^+ consumption in the solutions. These results suggested that much of the Se(VI) and Cr(VI) is retained only by the coulomb force on the positive charge sites of the soil particles shown in Eq. 3, because the adsorption maxima of Se(VI) and Cr(VI) were similar or smaller than the positive charge of the soil at pH 5.0. The adsorptive strength and affinity of Se(VI) to soil particle surfaces would be very weak in comparison to those of Se(IV). The findings of Hayes et al. (1987) indicate that selenate retains a hydration shell when adsorbed by goethite and forms a weakly bonded, outer-sphere surface complex. Zhang and Sparks (1990) concluded from the equilibrium and kinetics studies that selenate adsorption by goethite occurs at the layer via electrostatic attraction to form outer-sphere surface complexes. Some macroscopic studies have also demonstrated that Cr(VI) is fairly mobile in such an environments (Stollenwerk and Grove 1985; Selim et al. 1989; Zachara et al. 1989), although advanced spectroscopic evidences have shown that Cr(VI) forms inner-sphere complexes on goethite (Hsia et al. 1993; Fendorf et al. 1997).

In the present study, H^+ consumptions with Se(IV), As(V) and Mo(VI) sorptions were obviously smaller than those with P(V) and F(−I) sorptions. The release of OH^- accompanied with Se(IV) sorption on allophane clay was larger than that by P(V) sorption (Rajan and Watkinson 1976). Although most researchers have concluded that the increase in the solution pH with a certain anion adsorption on particles indicates the release of OH^- from the particle surface, our results are based on the reaction (5) described by Zhang and Sparks (1990). The H^+ consumption was related not only to the reaction (5) but also to the reaction (6). However one cannot differentiate these reactions. The proportion of the reaction (5) for Se(IV), As(V), and Mo(VI) was smaller than those for P(V) and F(−I), suggesting that the surface sites with OH groups for the inner-sphere complexes with Se(IV), As(V), and Mo(VI) on soil particles were restricted in comparison to those for P(V) and F(−I). Soil differs from pure systems in that the sites for ligand exchange reactions are not restricted to OH and water molecules (Pardo and Guadalix 1990). For P(V) sorption on soil, the concomitant release of OH, Si, organic C, and sulfate have been observed (Obihara and Russell 1972; Rajan and Fox 1975; Ryden et al. 1977; Parfitt 1989; Pardo and Guadalix 1990). The concomitant consumption of H^+ and release of organic-C, Si, and $SO_4{}^{2-}$ also took place when Se(IV) was sorbed by the allophanic soils (Saeki and Matsumoto 1994). Some of the Se(IV), As(V), and Mo(VI) are considered to be sorbed by ligand exchange, such as P(V) (e.g., Arai et al. 2005), and to form inner-sphere surface complexes on soil particles and specimen minerals such as goethite (Hayes et al. 1987; Bibak and Borggaard 1994; Sun and Doner 1996; Goldberg et al. 1996). The present results support this concept.

The ligand exchanges with F(−I) sorption were qualitatively and quantitatively different from those for other anion sorptions, especially for that shown in reaction (5), which is commonly regarded as the displacement of OH groups by F ions; this displacement is large and constitutes a major proportion of the ligand exchanges in this case. The tendency of an F ion to displace OH ions may indicate that the average plane of F(−I) adsorption is closer to the surface than that of P(V) (Barrow and Ellis 1986). It is assumed that F(−I) ions can penetrate into areas where neither P(V) nor Se(IV) ions can enter because of their ion size, and F(−I) can react with pure OH groups.

The results confirm that F(−I), P(V), Se(IV), Mo(VI), and As(V) are adsorbed by soil particle surfaces by ligand exchange, thus forming the inner-sphere complexes, and that Se(VI), Cr(VI), and N(V) are attracted as an ion pair, forming outer-sphere surface complex with water molecules between the anions and the soil surface.

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