

Anion Sorption on a Calcareous, Montmorillonitic Soil—Selenium

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ABSTRACT

Selenite and selenate sorption on a calcareous, montmorillonitic soil was investigated as a function of solution pH (2–11) at two initial Se concentrations (1.9 mmol m^{-3} and 19 mmol m^{-3}). Selenate sorption was not observed at any pH value for either initial Se concentration. Selenite sorption exhibited a maximum near pH 3, a sharp decline to pH 6, and a sorption plateau above pH 7. Selenite sorption as a function of pH was studied on reference minerals representative of the dominant mineral constituents of the soil: montmorillonite, kaolinite, and calcite. Selenite sorption on the clay minerals increased at low pH, exhibited a peak near pH 5, and decreased at higher pH. Selenite sorption on calcite increased from pH 6 to 8, peaked between pH 8 and 9, and decreased above pH 9. The constant capacitance model was able to describe selenite sorption on montmorillonite and kaolinite over the entire pH range studied and on the soil below pH 7. The soil sorption plateau above pH 7 virtually disappeared after removal of calcite, indicating that calcite plays an important role in selenite sorption of calcareous soils.

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AS AN ESSENTIAL ELEMENT in animal nutrition, Se is required in small amounts, has a narrow concentration range of sufficiency, and produces toxicity symptoms at higher concentrations. Seleniferous soils yield enough Se to grow vegetation toxic to grazing animals. These types of soils occur in the semiarid states in the Western USA, especially in South Dakota (Lakin, 1961). Elevated concentrations of Se in agricultural drainage waters supplying Kesterson Reservoir Wildlife Refuge in the San Joaquin Valley of California have been implicated in deaths and deformities of migratory water fowl (Ohlendorf et al., 1986).

Selenium sorption in soils has been investigated previously by various researchers (Jones and Belling 1967; John et al., 1976; Elsokkary, 1980; Singh et al., 1981; Ylaranta, 1983; Neal et al., 1987). The sorption behavior of Se is dependent on its oxidation state. Selenate did not sorb onto clay, fine sand, or peat (Ylaranta, 1983). Less than 1% of selenate added to a lignite overburden material was sorbed (Ahlrichs and Hossner, 1987). In contrast, Singh et al. (1981) found that selenate sorption on five different soils was consistently higher than selenite sorption. Selenite was sorbed strongly by a clay soil (Ylaranta, 1983). Selen-

ite sorption on lignite overburden material was greatest at pH 3 and decreased slightly with increasing pH value (Ahlrichs and Hossner, 1987). Selenite sorption on five alluvial soils from California exhibited a peak near pH 4, a rapid decline in the pH range 4 to 6, and a gradual decline in the pH range 6 to 9 (Neal et al., 1987). Amount of sorbed selenite was significantly correlated with extractable aluminum, iron, and silica for 66 New Zealand surface soils (John et al., 1976). Soil Se content was significantly correlated with total carbonate, free Fe oxide, clay (Elsokkary, 1980), and extractable Al and Fe (Levesque, 1974).

The Se sorbing mineral constituents of soils are Al and Fe oxides, clay minerals, and calcite. Sorption of selenite on goethite was greatest near pH 3, decreased gradually to pH 8, and decreased rapidly above pH 8 (Hingston et al., 1968). Sorption of selenate on amorphous Fe oxyhydroxide was also maximal at pH 3, but decreased very rapidly to pH 7 (Davis and Leckie, 1980; Benjamin and Bloom, 1981). Selenite sorption on hydrous ferric oxide gel at pH 6.5 (Ryden et al., 1987) and on goethite at pH 4.5 to 9.5 (Balistrieri and Chao, 1987) was much greater than selenate sorption. Removal of selenate from solution by coprecipitation into Fe oxyhydroxide was slight, while that of selenite was substantial (Merrill et al., 1986). Siu and Berman (1984) suggested that selenite and selenate can be separated quantitatively because of the extremely limited coprecipitation of selenate by hydrous Fe oxide.

Selenite sorption on hydrous alumina occurred via a ligand exchange mechanism with reactive surface OH ions (Rajan, 1979); ligand exchange is a mechanism for specific adsorption of anions. A shift in the zero point of charge of hydrous alumina (Rajan, 1979) and goethite (Hingston et al., 1968; Hansmann and Anderson, 1985) was observed following selenite sorption providing evidence for specific adsorption. Direct spectroscopic evidence for specific adsorption of selenite on goethite was obtained using extended x-ray absorption fine structure (EXAFS) measurements (Hayes et al., 1987).

Selenite sorption on clay minerals occurs via the same mechanism as on oxides, ligand exchange with reactive surface hydroxyl groups at clay edges (Neal et al., 1987). Selenite sorption on the clay minerals kaolinite and montmorillonite was highest at pH 3, decreased at pH 6, and was low at pH 9 (Hamdy and Gissel-Nielsen, 1977; Frost and Griffin, 1977). These researchers found that the decrease in sorption with increasing pH was much more gradual for kaolinite than for montmorillonite clay. Bar-Yosef and Meek (1987) found similar magnitudes for selenite sorption on kaolinite and montmorillonite. They also found selenite sorption to be greater than selenate sorption on kaolinite.

While no Se sorption data is available for calcite, Jones and Belling (1967) found that a soil consisting of 74% CaCO₃ retained almost twice as much selenite against leaching as did an acidic laterite soil. Singh et al. (1981) also found a calcareous soil to sorb more selenite and selenate than a normal, saline, or alkaline soil. Neal et al. (1987) indicated that, in the range of 0 to 3%, CaCO₃ content was not a major factor in determining selenite sorption for five soils. This result

was likely due to the small amounts of calcite present in their soils which may have been partially dissolved in the sodium saturation pretreatment procedure.

Arsenic is a toxic element that has become elevated in some soils through the use of arsenical pesticides. Arsenate is a specifically adsorbing anion that adsorbs by the same ligand exchange mechanism with reactive surface hydroxyl groups as selenite (Goldberg and Glaubig, 1988). Arsenate and selenite are highly competitive with phosphate for sorption sites on goethite (Hingston et al., 1971) and hydrous ferric oxide gel (Ryden et al., 1987). Thus the competitive sorption behavior of these two anions is of interest.

The constant capacitance model is a surface complexation model appropriate for specific adsorption. This model successfully described selenite sorption on goethite (Goldberg, 1985) and gibbsite, as well as phosphate and boron sorption on soils (Goldberg, 1986).

In the present study, the sorption behavior of selenite and selenate on a calcareous, montmorillonitic soil from the Imperial Valley of California was investigated. We also studied the sorption behavior of selenite onto reference minerals representative of the dominant mineral constituents in the above soil: montmorillonite, kaolinite, and calcite. Subsequently, the ability of the constant capacitance model to describe these sorption reactions was investigated.

MATERIALS AND METHODS

Selenite and selenate sorption was studied using a surface (0–7.6 cm) sample of the Imperial soil series (fine, montmorillonitic [calcareous], hyperthermic Vertic Torrifluent). The <2-mm fraction was used. It contained 44% clay as determined using the hydrometer method described by Day (1965). Specific surface area was found to be 36.1 m² g⁻¹ using a single point BET N₂ adsorption isotherm obtained with a Quantachrome Quantasorb Jr. surface area analyzer (Quantachrome Corp., Syosset, NY). Calcium carbonate equivalent and organic C content, determined by wet combustion (Nelson and Sommers, 1982), were 14.7 and 0.83%, respectively. Montmorillonite and kaolinite constituted about two thirds of the <2- μ m fraction as determined by conversion of x-ray diffraction peak areas directly to clay mineral contents using the method of Klages and Hopper (1982). Free Fe and Al were determined by the method of Coffin (1963) to be 0.67 and 0.043%, respectively.

Selenite sorption was studied on SAz-1 Ca-montmorillonite "Cheto" and KGa-2 poorly crystallized kaolinite obtained from the Clay Mineral Society's Source Clay Repository, and on Purecal U CaCO₃ from the Wyandotte Chemical Co. (Wyandotte, MI). The CaCO₃ was identified as calcite using x-ray diffraction analysis (J.J. Jurinak, 1987, personal communication). These materials were used without any pretreatment. Specific surface areas of the clay minerals, determined from a multi-point BET N₂ adsorption isotherm obtained using a Cahn electrobalance model RG, were 18.6 m² g⁻¹ for montmorillonite and 20.5 m² g⁻¹ for kaolinite. The montmorillonite contained 27% clay, while the kaolinite contained 60% clay. Specific surface area of the calcite was 12.1 m² g⁻¹ using a single-point BET N₂ adsorption isotherm obtained with a Quantachrome Quantasorb Jr. surface area analyzer. This specific surface area is in excellent agreement with that of 13.5 m² g⁻¹ obtained by Hasset and Jurinak (1971) for this calcite using stearic acid adsorption.

Selenium sorption experiments were carried out in batch

systems at $23 \pm 1^\circ\text{C}$. Samples of solid (1.2 g for soil and clays and 0.3 g for calcite) were added to 50-mL polypropylene centrifuge tubes and equilibrated with 30.0 mL of a 0.1 M NaCl solution by shaking for 2 h on a reciprocating shaker. A 2-h-reaction time had been found to be sufficient to reach maximum sorption of selenite on five California soils (Neal et al., 1987). The equilibrating solution contained 1.9 or 19 mmol Se m^{-3} and had been adjusted to the desired pH values using 4 M HCl or 4 M NaOH. The samples were centrifuged at 7800 RCF (10 000 rpm) for 20 min in a Servall superspeed centrifuge. The decantates were analyzed for pH, filtered through a no. 42 Whatman filter, acidified with six drops of concentrated HCl, and analyzed for Se concentration. Selenium (IV plus VI) and selenium(IV) were determined using a Perkin-Elmer model 3030B atomic absorption spectrophotometer (Perkin-Elmer Corp., Norwalk, CT) with an electrodeless discharge lamp, a Varian VGA-76 hydride generator (Varian Assoc., Inc., Palo Alto, CA), and the method described by Voth-Beach and Shrader (1985).

Release of native Se from the soil into 0.1 M NaCl was studied. Conditions for this experiment were identical to the Se sorption studies except that no Se was added to the equilibrating solution. Concentrations of native Se released were low, ≤ 0.08 mmol m^{-3} for selenite and ≤ 0.8 mmol m^{-3} for selenate. Oxidation of selenite or reduction of selenate were not observed under our experimental conditions.

To investigate the competitive effect of the arsenate anion, selenite sorption on the soil was studied in the presence of 20 mmol m^{-3} (approximately equimolar) arsenate as described above. To investigate its importance in selenite sorption, calcite was removed from the soil using a modification of the procedure described by Kunze and Dixon (1986). The soil was washed three times with a 0.5 M sodium acetate solution which had been adjusted to pH 5 by addition of glacial acetic acid, washed twice with distilled water, air-

dried, and passed through a 2-mm sieve. Selenite sorption on this treated soil was determined under identical conditions as described above.

The constant capacitance model (Stumm et al., 1980) was used to describe Se sorption. The computer program FI-TEQL (Westall, 1982) was used to fit intrinsic conditional equilibrium constants to the experimental data. In the model specific surface area is a necessary parameter for relating surface charge to surface potential. A detailed description of the model assumptions, surface reactions, intrinsic conditional equilibrium constant expressions, mass balances, charge balance, capacitance density and modeling procedure is provided in Goldberg (1986). The intrinsic protonation-dissociation constants were initially fixed at $\log K_+(\text{int}) = 7.35$, $\log K_-(\text{int}) = -8.95$ for the soil and $\log K_+(\text{int}) = 7.38$, $\log K_-(\text{int}) = -9.09$ for the clays, but were optimized simultaneously with the Se surface complexation constants, if necessary.

RESULTS AND DISCUSSION

Selenite sorption as a function of pH is shown in Fig. 1a,b for two initial selenite concentrations. Both sorption curves show maxima near pH 3, followed by a sharp decline to pH 6, a slight rise, and a sorption plateau at high pH. The shape of the selenite sorption curves is very similar to that found by Neal et al. (1987) for five alluvial soils from California; the magnitude of the sorption plateau, however, is greater for our data. Using the computer program GEOCHEM (Sposito and Mattigod, 1979), we calculated that our systems were several orders of magnitude undersaturated with respect to CaSeO_3 and $\text{Fe}_2(\text{SeO}_3)_3$. No constants were available for the formation of a selenite precipitate with Al or a selenite coprecipitate with Fe or Al. Thus these sorption mechanisms cannot be ruled out.

Selenate sorption was not observed for either initial selenate concentration (1.9 mmol m^{-3} and 19 mmol m^{-3}) at any of the pH values (2–12) investigated. Direct spectroscopic evidence for different adsorption mechanisms for the two Se oxidation states is available. Using EXAFS measurements, Hayes et al. (1987) found that selenate formed a weakly bonded, outer-sphere complex and that selenite formed a strongly bonded, inner-sphere complex when adsorbed on goethite.

Figure 2a-c shows selenite sorption as a function of pH for the reference minerals. The sorption curves for the clay minerals increase at low pH, exhibit peaks near pH 5, and decrease at higher pH. Bar-Yosef and Meek (1987) also found selenite sorption on these clays to be highest near pH 5. The magnitude of the selenite sorption maximum for the kaolinite is roughly twice that for the montmorillonite, despite very similar surface areas for these two materials. This is likely due to the greater proportion of edge surface area for kaolinite. Selenite sorption on the calcite increases from pH 6 to 8, peaks between pH 8 and 9, and decreases above pH 9. The magnitude of the sorption maximum for calcite is four times that for kaolinite and eight times that for montmorillonite. The high reactivity for this material is involved in selenite sorption rather than only the edge surface area as for the clay minerals.

Table 1 lists the Se surface complexation constants obtained from the application of the constant capac-

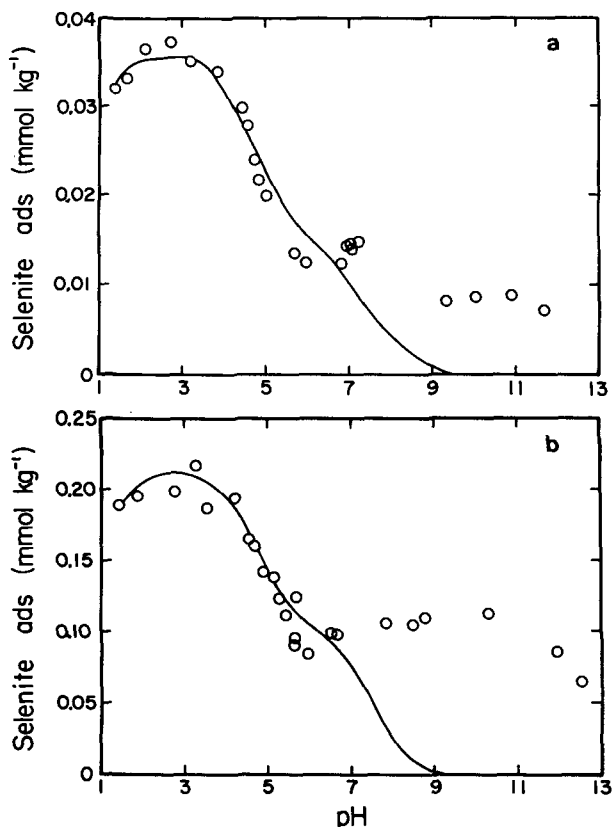


Fig. 1. Selenite sorption on Imperial soil: (a) $\text{Se}_T = 1.9$ mmol m^{-3} , (b) $\text{Se}_T = 19$ mmol m^{-3} . Circles represent experimental data points. Model results are represented by solid lines.

Table 1. Surface complexation constants obtained from FITEQL.†

Solid	Log $K_{+}(\text{int})$	Log $K_{-}(\text{int})$	Log $K_{\text{Se}}^{\text{e}}(\text{int})$	Log $K_{\text{Se}}^{\text{e}}(\text{int})$
Soil				
$\text{Se}_{\text{T}} = 1.9 \text{ mmol m}^{-3}$	2.67	‡	8.44	3.09
$\text{Se}_{\text{T}} = 19 \text{ mmol m}^{-3}$	2.99	‡	7.39	2.18
Montmorillonite	§	§	10.92	3.40
Kaolinite	§	§	11.07	3.34

† All surface complexation constants were obtained using concentrations in mol L^{-1} .

‡ Indicates that this constant was insignificantly small (≤ -28).

§ Log $K_{\text{Se}}^{\text{e}}(\text{int})$ were optimized while holding log $K_{+}(\text{int})$ and log $K_{-}(\text{int})$ constant.

itance model to the experimental sorption data using the computer program FITEQL. For the soil, these values were obtained by optimizing all four surface complexation constants simultaneously. Optimization of solely the Se surface complexation constants produced extremely poor fits. This behavior had been observed previously in the modeling of B sorption on soil samples (Goldberg, 1986). For the clays, the intrinsic protonation-dissociation constants were fixed at $\log K_{+}(\text{int}) = 7.38$, $\log K_{-}(\text{int}) = -9.09$, as done previously for B sorption on kaolinite (Goldberg, 1986).

The constant capacitance model is able to describe selenite sorption on the soil up to pH 7, but is unable to fit the sorption plateau occurring in the pH range 8 to 11 (Fig. 1). The inability of the model to describe sorption over the entire pH range, suggests that selenite sorption is occurring on two types of competing sites. The optimized values for log $K_{-}(\text{int})$ are insignificantly small, implying that this constant is unnecessary and that the surface functional group, SOH, does not dissociate. This chemically unrealistic situation casts doubt on the chemical significance of at least one of the surface complexation constants obtained from the application of the model to selenite sorption on soil.

The constant capacitance model is well able to describe selenite sorption on the clay minerals, fitting a smooth curve through the experimental data (Fig. 2a,b). The model successfully describes sorption up to pH 9 using only two adjustable parameters, the Se surface complexation constants. It should be noted that the values of these sets of constants are almost identical, suggesting involvement of the same sorption mechanism for both clay minerals. The constant capacitance model assumes that sorption on clays occurs by ligand exchange with reactive surface aluminol groups, AlOH, located at the particle edges. The close fit of the model to the experimental data, suggests that this mechanism may be appropriate for selenite sorption on kaolinite and montmorillonite.

The constant capacitance model was not applied to selenite sorption on calcite because surface complexation constants for the potential determining carbonate and bicarbonate anions are not available. The high magnitude and the pH of the sorption maximum suggest that calcite may be responsible for the sorption plateau occurring on the soil above pH 7. Indeed, after removal of calcite, the sorption plateau virtually disappears (Fig. 3). These results indicate that calcite plays an important role in the selenite sorption of calcareous soils.

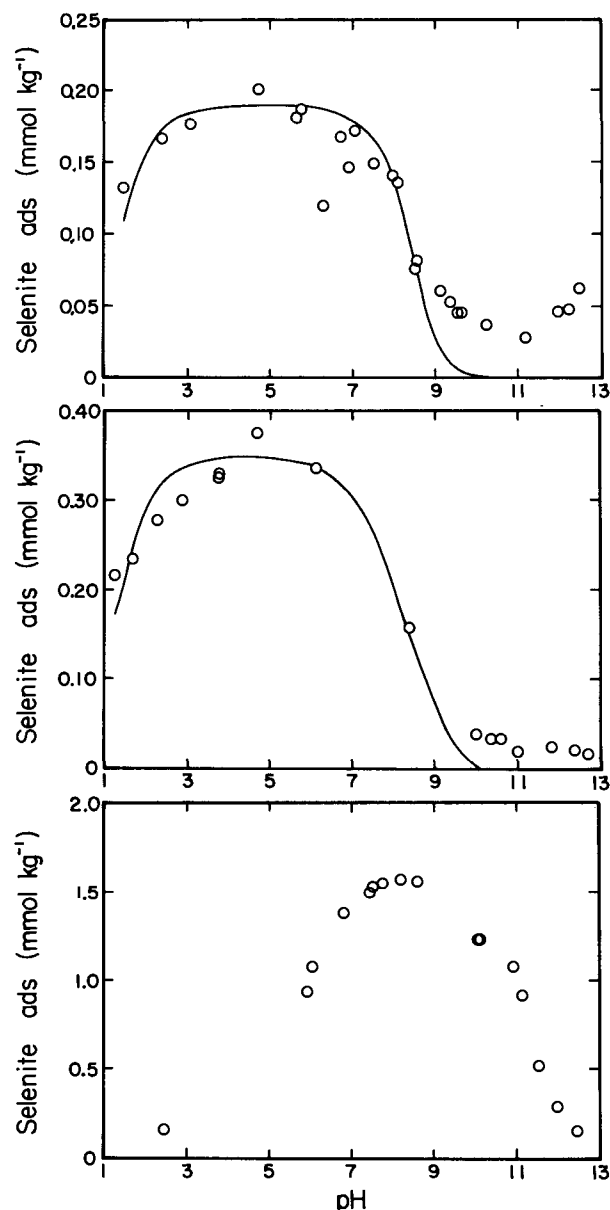


Fig. 2. Selenite sorption on reference minerals: (a) SAZ-1, Ca-montmorillonite, (b) KGa-2, poorly crystallized kaolinite, (c) Purecal U calcite. $\text{Se}_{\text{T}} = 19 \text{ mmol m}^{-3}$. Circles represent experimental data points. Model results are represented by solid lines.

The competitive effect of arsenate on selenite sorption was studied using equilibrating solutions containing both anions in approximately equimolar amounts. No reduction in selenite sorption is observed up to pH 9 (Fig. 3). Above pH 9, selenite sorption is much reduced by the presence of arsenate. These results are in agreement with those of Ryden et al. (1987) who found that selenite and arsenate were sorbed equally by hydrous ferric oxide gel at pH 6.5.

Since the Imperial soil series is in the montmorillonitic mineralogy class, montmorillonite constitutes the dominant mineral in the clay fraction (Soil Survey Staff, 1975). Comparison of the magnitude of the soil selenite sorption maximum to those for the two reference clay minerals, corroborates the predominance of montmorillonite. Since montmorillonite and kaolinite constitute about two-thirds of the clay fraction and the free oxide content is low, we conclude that

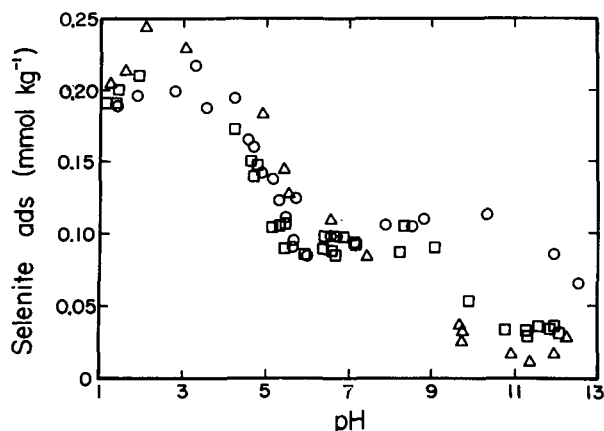


Fig. 3. Selenite sorption on untreated soil (circles), on soil after treatment to remove calcite (corrected for weight of calcite removed; triangles), and in the presence of arsenate (squares). $Se_T = 19 \text{ mmol m}^{-3}$. $As_T = 20 \text{ mmol m}^{-3}$.

these minerals are responsible for the soil sorption peak below pH 6. Our results indicate that above pH 7 selenite sorption occurs primarily on soil calcite. The magnitude of the sorption plateau indicates that soil calcite is very reactive toward selenite, sorbing about half as much selenite on a per gram basis as the highly reactive Purecal U calcite. The mean pH of a calcareous soil under a variety of moisture conditions in the field has been found to be 7.41 ± 0.13 (Suarez, 1986). Thus under field conditions selenite sorption on calcareous soils will occur predominantly on calcite. Since calcareous soils are common in arid and semiarid areas, including those exhibiting elevated Se concentrations, our findings are highly significant.

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