Influence of Soil Solution Salinity on Molybdenum Adsorption by Soils

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Abstract: Molybdenum (Mo) adsorption on five arid-zone soils from California was investigated as a function of equilibrium solution Mo concentration (0–30 mg L^{-1}), solution pH (4–8), and electrical conductivity (EC = 0.3 or 8 dS m^{-1}). Molybdenum adsorption decreased with increasing pH. An adsorption maximum was found near pH 4. Molybdenum adsorption as a function of solution pH was independent of solution salinity from pH 4 to 8. Molybdenum adsorption for five soils as a function of solution Mo concentration conformed to the Langmuir adsorption isotherm equation. The Mo adsorption maxima obtained with the Langmuir equation for both ECs were not statistically significantly different at the 95% level of confidence, with the exception of one soil. The constant capacitance model, a surface complexation model, was able to describe Mo adsorption as a function of solution Mo concentration and solution pH. Molybdenum adsorption was predicted using the soil chemical properties: cation exchange capacity, organic carbon content, inorganic carbon content, and iron oxide content. These results are advantageous, as they indicate that, under agricultural conditions (pH 4–8), Mo adsorption can usually be described without consideration of changes in soil solution salinity.

Key Words: Constant capacitance model, electrical conductivity, surface complexation model

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Molybdenum (Mo) is an important micronutrient element required by both plants and animals. Molybdenum deficiencies have been reported for many agronomic crops throughout the world, and fertilization is necessary (Murphy and Walsh, 1972). Molybdenum occurs in anionic form in most soil solutions, is readily taken up by plants, especially forage legumes, and can accumulate to levels detrimental to grazing ruminant animals (Reisenauer et al., 1973). Soils with high Mo content can occur on pastures growing on both alkaline soils such as those in the San Joaquin Valley of California (Barshad, 1948) and acid soils such as those in northwest Oregon (Kubota et al., 1967). Evaluation of the plant availability of Mo in soil systems requires an understanding of its adsorption chemistry. Detailed knowledge of the fate and transport of Mo in soils and groundwaters is necessary to understand and address a range of agricultural problems.

Adsorption sites on organic matter, oxides, and clay minerals act as sources and sinks for Mo in soils. Molybdenum adsorbed on these solids equilibrates with solution Mo concentrations (Goldberg et al., 1996). Plant availability of Mo is affected by a variety of factors including solution pH, soil texture,

soil temperature, clay mineralogy, oxide and organic matter content, and competing anion concentration (Reisenauer et al., 1973).

Molybdenum adsorption reactions by soil minerals and soils have generally been studied in single anion solutions or, at most, in the presence of one competing anion species. Some previous studies have evaluated the effect of competing sulfate, selenate, phosphate, arsenate, silicate, or tungstate concentrations on Mo adsorption by aluminum and iron oxides (Manning and Goldberg, 1996; Vissenberg et al., 2000; Wu et al., 2001; Gustafsson, 2003; Xu et al., 2006), clay minerals (Goldberg and Forster, 1998), and soils (Gorlach et al., 1969; Gonzalez et al., 1974; Roy et al., 1986; Smith et al., 1987; Xie and MacKenzie, 1991; Brinton and O'Connor, 2000). Phosphate, arsenate, selenate, or tungstate concentrations equimolar to Mo concentrations depressed Mo adsorption by Al and Fe oxides (Manning and Goldberg, 1996; Goldberg and Forster, 1998; Wu et al., 2001; Gustafsson, 2003; Xu et al., 2006), whereas silicate concentrations had little effect (Xu et al., 2006). The presence of sulfate depressed Mo adsorption on Al oxides (Vissenberg et al., 2000; Wu et al., 2001) but not on Fe oxide (Xu et al., 2006). Arsenate concentrations equimolar to Mo concentrations had little effect on Mo adsorption by clay minerals (Goldberg and Forster, 1998). Results for the competitive effect of sulfate on Mo adsorption by soils are contradictory. The presence of sulfate substantially reduced Mo adsorption by three soils from Texas (Smith et al., 1987) and two biosolids-amended soils from Florida (Brinton and O'Connor, 2000) but did not affect Mo adsorption by two soils from Poland (Gorlach et al., 1969) and 16 volcanic ash soils from Chile (Gonzalez et al., 1974). The presence of phosphate and arsenate decreased Mo adsorption by soils in all investigations (Gorlach et al., 1969; Gonzalez et al., 1974; Roy et al., 1986; Xie and MacKenzie, 1991; Brinton and O'Connor, 2000).

The effect of solution ionic strength on Mo adsorption was investigated for Al oxide, Fe oxide, clay minerals, and two soils (Zhang and Sparks, 1989; Goldberg et al., 1998) and was found to be slight. This indicates a strong specific adsorption mechanism for Mo on these solids. In natural systems, adsorption takes place from solutions of mixed electrolyte composition. The effect of increasing salinity from mixed electrolyte solutions on Mo adsorption by soils has not yet been evaluated.

Chemical models of Mo adsorption have more general predictive capability than empirical adsorption isotherm equations. This has been demonstrated in prior studies of Mo adsorption using the constant capacitance model (Goldberg et al., 2002, 2008). Prediction equations were developed that relate the Mo surface complexation constant to the easily measured soil chemical parameters: cation exchange capacity (CEC), organic carbon content (OC), inorganic carbon content (IOC), and free iron oxide content (Fe). The equation reliably predicted Mo adsorption as a function of solution pH with reasonable accuracy on 36 soils primarily from California (Goldberg et al., 2002). These applications provided an independent evaluation of the predictive capability of the model to describe Mo adsorption by soils.

The purpose of this study was to evaluate the effect of mixed electrolyte solutions on Mo adsorption by diverse soil samples from California. The results will be described using the

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TABLE 1. Classifications and Chemical and Physical Characteristics of Soils

Soil Series	Depth	Clay	CEC	SA	IOC	OC	Fe	Al
	cm	%	mmol$_c$ kg^{-1}	m^2 g^{-1}	---------g kg^{-1}----------			
Arlington (coarse-loamy, mixed thermic Haplic Durixeralf)	0–25	17.5	107	61.1	0.30	4.7	8.2	0.48
	25–51	14.0	190	103	0.16	2.8	10.1	0.60
Pachappa (coarse-loamy, mixed, thermic Mollic Haploxeralf)	0–25	13.0	39	15.1	0.026	3.8	7.6	0.67
	25–51	7.0	52	41	0.014	1.1	7.2	0.35
Ramona (fine-loamy, mixed, thermic Mollic Haploxeralf)	0–25	12.0	66	27.9	0.02	4.4	4.5	0.42
Lillis (very fine, smectitic, chromic Halic Haploxerert)								
BWTM 5/2002	0–45	48.9	27.4	168	2.45	7.6	11.3	0.84
BWTM 8/2002	0–45	35.4	27.2	173	2.45	7.2	11.6	0.88

constant capacitance model. The ability of the model to predict Mo adsorption by these soil samples using the prediction equation of Goldberg et al. (2002) will be evaluated.

MATERIALS AND METHODS

Molybdenum adsorption was investigated using five surface and two subsurface soil samples from California. Soil classifications and physical and chemical characteristics are listed in Table 1. The two soils classified as Lillis soil differed in their clay content. Surface areas were measured using ethylene glycol monoethyl ether adsorption as described by Cihacek and Bremner (1979). Free Fe and Al oxides were determined using the extraction method of Coffin (1963). Extractable Fe and Al concentrations in the solutions were analyzed using inductively coupled plasma optical emission spectrometry (ICP-OES). Organic carbon and inorganic carbon contents were measured with a carbon coulometer. Total carbon was analyzed by combustion at 950 °C; IOC was quantified using an acidification module and heating, and OC was determined by their difference. Particle size distributions for the soils were obtained using the basic hydrometer method (Gee and Bauder, 1986) to determine the clay content.

Molybdenum adsorption experiments were carried out in batch systems to determine adsorption envelopes (amount of Mo adsorbed as a function of solution pH per fixed total Mo concentration) and adsorption isotherms (amount of Mo adsorbed as a function of equilibrium solution Mo concentration). For the Mo adsorption envelopes, five grams of air-dried soil was added to 50-mL polypropylene centrifuge tubes and equilibrated with 25 mL of a mixed electrolyte solution on a reciprocating shaker for 20 h. The mixed electrolyte solutions had been prepared for a study of boron movement in large soil column lysimeters (Shouse et al., personal communication), varied in electrical conductivity (EC), and contained bromide as a conservative tracer. The chemical compositions of these solutions are presented in Table 2. The solutions had been adjusted to the desired pH range using 0.5 mL of a solution containing varying proportions of 1 M HCl, 1 M NaCl, and 1 M NaOH. This adjustment increased the salinity of the low-EC solution to 0.52 dS m^{-1} and the salinity of
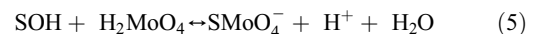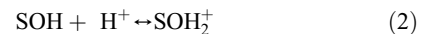
the high-EC solution to 8.1 dS m^{-1}. The reaction temperature was 23.4 ± 0.1 °C. After reaction, the samples were centrifuged, and the decantates were analyzed for pH, filtered, and analyzed for Mo concentrations using ICP-OES.

For the Mo adsorption isotherms, 200 g of soil were equilibrated with 100 mL of mixed electrolyte solution in 420-mL plastic containers. The containers were covered with snap-tight lids and equilibrated at 23.5 ± 0.2 °C for 18 h. The mixed electrolyte solutions consisted of 0, 1, 2, 3, 5, 10, 15, 20, 25, or 30 mg Mo L^{-1} and were prepared using low EC = 0.32 dS m^{-1} or high EC = 7.9 dS m^{-1} water. After equilibration, the soil pastes were transferred to funnels fitted with no. 50 Whatman filter paper. The filtrate solutions were collected under vacuum and analyzed for Mo using ICP-OES. Amount of Mo adsorption was determined as the difference between the initial electrolyte solution Mo concentration and the equilibrium Mo concentration. The Langmuir adsorption isotherm equation:

$$\text{Mo}_{ads} = \frac{K\text{Mo}_{eq}M}{1 + K\text{Mo}_{eq}} \tag{1}$$

where Mo$_{ads}$ is the Mo adsorption (mmol kg^{-1}), Mo$_{eq}$ is the equilibrium Mo concentration (mmol L^{-1}), M is the maximum Mo adsorption (mmol kg^{-1}), and K is a parameter (L mmol^{-1}) fit to the Mo adsorption data.

A detailed explanation of the theory and assumptions of the constant capacitance surface complexation model of adsorption was provided by Goldberg (1992). In the present model application to Mo adsorption, the following surface complexation reactions were considered:

$$\text{SOH} + \text{H}^+ \leftrightarrow \text{SOH}_2^+ \tag{2}$$

$$\text{SOH} \leftrightarrow \text{SO}^- + \text{H}^+ \tag{3}$$

$$\text{SOH} + \text{H}_2\text{MoO}_4 \leftrightarrow \text{SHMoO}_4 + \text{H}_2\text{O} \tag{4}$$

$$\text{SOH} + \text{H}_2\text{MoO}_4 \leftrightarrow \text{SMoO}_4^- + \text{H}^+ + \text{H}_2\text{O} \tag{5}$$

where SOH, the surface functional group, represents both reactive surface hydroxyl groups on oxides and aluminol groups

TABLE 2. Chemical Constituents of the Mixed Electrolyte Solutions

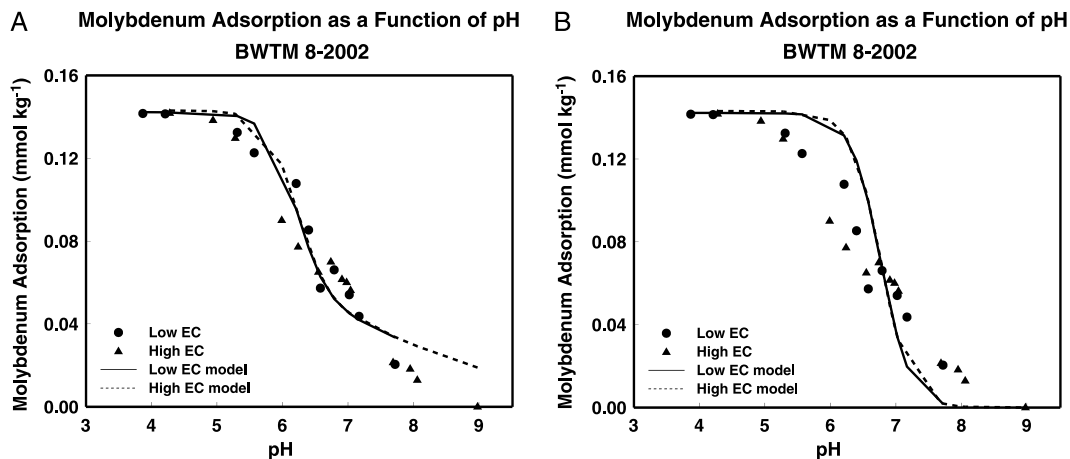|	pH	Cl$^-$	Na$^+$	Ca^{2+}	K$^+$	Mg^{2+}	SO$_4$$^{2-}$	HCO$_3$$^-$	B	Br$^-$	EC
		--------------------------------mEq L^{-1}------------------------------							---mg L^{-1}---		dS m^{-1}
Low EC	7.87	1.62	1.75	0.745	0.0415	0.729	0.078	1.41	0.309	17.9	0.32
High EC	8.35	30.4	72.3	2.51	0.983	11.8	49.3	6.40	0.277	20.9	7.9

FIG. 1. Molybdenum adsorption as a function of solution pH and EC for Lillis 8/2002 soil. A, Constant capacitance model fit optimizing $\log K^1_{Mo}(int)$ and $\log K^2_{Mo}(int)$ for both ECs simultaneously. B, Constant capacitance model prediction using $\log K^1_{Mo}(int) = 6.32$.

on clay mineral edges in soils. Equilibrium constant expressions for the surface complexation reactions are as follows:

$$K_+ \,(int) = \frac{\left[SOH_2^+\right]}{[SOH]\,[\,H^+]} \exp(F\psi/RT) \qquad (6)$$

$$K_- \,(int) = \frac{[SO^-]\,[\,H^+]}{[SOH]} \exp(-F\psi/RT) \qquad (7)$$

$$K^1_{Mo} \,(int) = \frac{[SHMoO_4]}{[SOH]\,[\,H_2MoO_4]} \qquad (8)$$

$$K^2_{Mo} \,(int) = \frac{\left[SMoO_4^-\right]}{[SOH]\,[\,H_2MoO_4]} \exp(-F\psi/RT) \qquad (9)$$

where square brackets indicate concentrations (mol L^{-1}), F is the Faraday constant (C mol_c^{-1}), ψ is the surface potential (V), R is the molar gas constant (J $mol^{-1}\,K^{-1}$), and T is the absolute temperature (K). The exponential terms represent solid-phase activity coefficients that correct for the charges on the surface complexes. These reactions and equilibrium constants were previously considered in modeling and prediction of Mo adsorption envelopes by diverse soils (Goldberg et al., 2002).

The computer code FITEQL 3.2 (Herbelin and Westall, 1996) uses a nonlinear least-squares optimization routine to fit equilibrium constants to experimental data and contains the constant capacitance surface complexation model of adsorption. As in prior studies (Goldberg et al., 2002, 2008), the FITEQL program was used to fit surface complexation constants to the experimental Mo adsorption data and to test the ability of the surface complexation constants calculated with the general regression model to predict Mo adsorption. The initial input parameter values were capacitance density: $C = 1.06$ F m^{-2} and surface site density: $N_s = 2.31$ sites nm^{-2}. The general regression model prediction equation for the Mo surface complexation constant is (Goldberg et al., 2002) as follows:

$$\log K^1_{Mo} \,(int) = 7.81 - 0.363\ln(CEC) + 0.219\ln(IOC/10)$$

$$+ 0.385\ln(OC/10) + 0.716\ln(Fe/10) \qquad (10)$$

where the units of CEC are in $mmol_c\,kg^{-1}$ and units of OC, IOC, and Fe are in g kg^{-1}. The surface complexation constant, $\log K^1_{Mo}$, was calculated from the chemical properties: CEC,

OC, IOC, and Fe and this equation. Using this predicted constant, Mo adsorption envelopes were predicted and compared with the experimentally determined Mo adsorption values.

RESULTS AND DISCUSSION

Molybdenum adsorption as a function of solution pH and salinity, EC, was determined for the two Lillis soil samples. Figure 1 presents the data for the 8/2002 soil. Molybdenum adsorption envelopes on both soil samples decreased with increasing solution pH. An adsorption maximum was found near pH 4. This type of adsorption envelope is characteristic of Mo adsorption behavior on soils (Goldberg et al., 1996, 1998, 2002, 2008). Molybdenum adsorption behavior by both soils was similar. The magnitude of Mo adsorption was independent of EC from pH 4 to 8. These results would suggest that, at agriculturally important soil solution pHs, changes in salinity do not affect the extent of Mo adsorption.

TABLE 3. Surface Complexation Constants for the Constant Capacitance Model

Soil	Optimized			From Prediction Equation
	$\log K^1_{Mo}$	$\log K^2_{Mo}$	SOS/DF[†]	$\log K^1_{Mo}$
Lillis				
5/2002	5.173 ± 0.044	0.255 ± 0.023	6.62	6.28
8/2002	5.400 ± 0.074	0.296 ± 0.029	4.06	6.32
Arlington				
Surface soil		-0.0628 ± 0.0084	14.4	4.84
Subsoil		-0.202 ± 0.0086	17.5	3.83
Pachappa				
Surface soil		0.0340 ± 0.0092	71.5	4.26
Subsoil		0.0437 ± 0.0085	43.7	3.52
Ramona				
Surface soil		-1.14 ± 0.012	49.6	3.67

[†]SOS/DF is the overall variance where SOS is the weighted sum of squares of the residuals, and DF is the degrees of freedom.

TABLE 4. Adsorption Isotherm Parameters

Soil	Low EC			High EC		
	K	*M*	*R²*	*K*	*M*	*R²*
Arlington						
Surface soil	0.82	0.46 ± 0.03	0.994[†]	0.36	0.76 ± 0.30	0.963[†]
Subsoil	1.4	0.25 ± 0.01	0.996[†]	0.70	0.60 ± 0.08	0.986[†]
Pachappa						
Surface soil	1.3	0.95 ± 0.08	0.991[†]	0.96	0.79 ± 0.06	0.994[†]
Subsoil	1.3	0.41 ± 0.02	0.994[†]	0.97	0.43 ± 0.04	0.988[†]
Ramona						
Surface soil	1.4	1.41 ± 0.13	0.993[†]	0.77	1.18 ± 0.11	0.995[†]

[†]Significance at the 95% level of confidence.

M = maximum adsorption (mmol kg^{-1}), K = parameter (L mmol^{-1}), R^2 = regression coefficient.

The constant capacitance model was fit to the Mo adsorption envelopes of both soil samples optimizing two Mo surface complexation constants: $\log K^1_{Mo}(int)$ and $\log K^2_{Mo}(int)$. These constants had been optimized previously to describe Mo adsorption envelopes by soils (Goldberg et al., 2002). The ability of the constant capacitance model to describe Mo adsorption on both soils was good and is shown in Fig. 1a for the Lillis 8/2002 soil. The optimized values of the Mo surface complexation constants are provided in Table 3. Both salinity treatments were optimized simultaneously for both soil samples. The values of the Mo surface complexation constant, $\log K^1_{Mo}(int)$, optimized in this study are not statistically significantly different at the 95% level of confidence from the average value of this constant determined for 36 soils by Goldberg et al. (2002): $\log K^1_{Mo}(int) = -4.84 ± 0.88$.

The ability of the constant capacitance model to predict Mo adsorption as a function of solution pH is indicated in Fig. 1b. To obtain the model results, the Mo surface complexation constant was calculated after entering the soil chemical properties: CEC, IOC, OC, and Fe into the prediction Eq. (10). This surface complexation constant was subsequently used in the FITEQL 3.2 code to speciate the chemical system and to predict Mo adsorption. The lines represent the prediction of Mo adsorption behavior for the Lillis 8/2002 soil from its chemical properties (Goldberg et al., 2002). The model predictions are very close to the experimental adsorption data in the pH range 4 to 5.5 and 6.5 to 7.5. In the pH range 5.5 to 6.5, the model overpredicts the extent of Mo adsorption. Above pH 7.5, the model considerably underpredicts the amount of Mo adsorption. The surface complexation constants obtained using this prediction approach are listed in Table 3. Because the model results are predictions, no model parameters were adjusted or optimized. The prediction is less close to the experimental adsorption value than the optimization both because no parameters are adjusted and because only one Mo surface complexation constant was used in the prediction, whereas two Mo surface complexation constants were adjusted in the optimization. It should be pointed out that the Lillis soils contain considerably more clay than the soils in the database of Goldberg et al. (2002) used to develop the prediction equation.

Molybdenum adsorption isotherms were determined on five soils under conditions of low (EC = 0.32 dS m^{-1}) and high (EC = 7.9 dS m^{-1}) electrolyte concentration. The Langmuir isotherm equation was fit to the experimental adsorption data using nonlinear least-squares optimization (Kinniburgh, 1986). With the exception of the Arlington subsoil, the Mo adsorption

maxima obtained in this manner were not statistically significantly different at the 95% level of confidence for the two electrical conductivities (Table 4). This result indicates that in the range of EC = 0.32 to 7.9 dS m^{-1}, the magnitude of Mo adsorption is usually independent of soil solution salinity.

The constant capacitance model was fit to the Mo adsorption isotherms on all five soils optimizing both salinity treatments simultaneously. The model was unable to optimize a value for $\log K^1_{Mo}(int)$, indicating that the SHMoO$_4$ species is present in insignificant amounts. This is not surprising, given that the SMoO$_4^-$ species was found to dominate at pH values above 6.5 for the Lillis soils. Nevertheless, the ability of the constant capacitance model to describe the Mo adsorption isotherms on all five soils is good. A sample fit is shown in Fig. 2 for the Arlington surface soil. The optimized values of the Mo surface complexation constant, $\log K^2_{Mo}(int)$, are provided in Table 3.

This investigation has shown that Mo adsorption as a function of solution Mo concentration and solution pH is generally independent of salinity in the pH range 4 to 8 up to EC = 8.1 dS m^{-1}. For the purpose of describing Mo transport in soils under agricultural conditions, these results are advantageous as they indicate that Mo adsorption can be described and predicted without consideration of changes in solution electrolyte concentration.
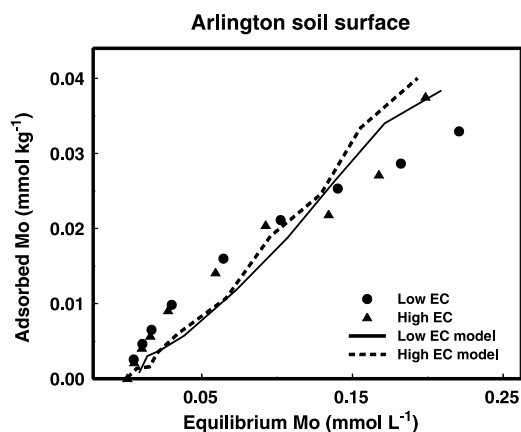

FIG. 2. Molybdenum adsorption as a function of solution Mo concentration and EC for Arlington surface soil. The constant capacitance model fit optimized $\log K^2_{Mo}(int)$ for both ECs simultaneously.

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