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Hexavalent Chromium Reduction in Soils Contaminated with Chromated Copper Arsenate Preservative

JINKUN SONG,[a,*] TIMOTHY TOWNSEND,[a]
HELENA SOLO-GABRIELE,[b] AND YONG-CHUL JANG[a,**]

[a]Department of Environmental Engineering Sciences, University of Florida, Gainesville, Florida, USA
[b]Department of Civil, Architectural, and Environmental Engineering, University of Miami, Coral Gables, Florida, USA

The toxicity and mobility of chromium in the environment greatly depends upon its speciation. The reduction of hexavalent chromium to trivalent chromium in a soil environment was examined by spiking three soil types (sandy, clayey, and organic soils) with a common wood preservative solution known as chromated copper arsenate (CCA). Chromium in the CCA preservative solution exists in the hexavalent form. The total and hexavalent chromium concentrations (mg/kg) were measured over a period of 11 months. Leachable chromium concentrations (mg/L) were assessed using the synthetic precipitation leaching procedure (SPLP). The degree and rate of hexavalent chromium reduction were similar for the sand and clayey soil, but much greater for the organic soil. Most of the chromium reduction occurred within the first month of the experiment. At the end of the experiment, approximately 50% of the hexavalent chromium was converted to the trivalent form in the sand and clayey soils. Hexavalent chromium concentrations were below detection in the organic soil at the end of the experiment. Nearly all of the chromium observed in the SPLP leachates was in the form of hexavalent chromium. Chromium leaching was thus greatest in the sand and clay soils where the hexavalent chromium persisted. The results indicate that hexavalent chromium in soils can persist for considerable time periods, in particular in soils with low organic matter content.

Keywords Chromium, hexavalent chromium, chromated copper arsenate, CCA, soil contamination

Introduction

Chromium has been widely used in a variety of industrial applications, including metal finishing, wood preserving, petroleum refining, and chrome tanning (Barnhart, 1997). Its

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[*] Present affiliation for Jinkun Song: Graduate Research Assistant, Department of Civil Engineering, Texas A&M University, Texas 77843.

[**] Present affiliation for Yong-Chul Jang: Assistant Professor, Department of Environmental Engineering, Chungnam National University, Daejeon 305-764, South Korea.

Address correspondence to Timothy Townsend, Department of Environmental Engineering Sciences, University of Florida, Gainesville, FL 32611-6450, USA. E-mail: ttown@ufl.edu

chemical characteristics and degree of toxicity depend upon its oxidation state, which is dominated by the trivalent or hexavalent forms (Bartlett and Vesilind, 1998; Katz and Salem, 1993). Hexavalent chromium (Cr(VI)) is highly toxic, whereas trivalent chromium (Cr(III)) is significantly less toxic and much less soluble (James *et al.*, 1995; Wang and Vipulanandan, 2001). When a pollutant or waste containing Cr(VI) is introduced to the soil environment, Cr(VI) will tend to be reduced to Cr(III). The degree and rate of conversion depend on many factors, including pH, oxidation reduction potential (ORP), and the content of organic matter, iron, and sulfides (Saleh *et al.*, 1989; Rai *et al.*, 1989; Richard and Bourg, 1991; Weng *et al.*, 1994; Wittbrodt and Palmer, 1995. Wittbrodt and Palmer, 1996; Lee *et al.*, 1999; Wang and Vipulanandan, 2001; Schlautman and Han, 2001).

One common industrial use of Cr(VI) in recent decades has been the wood preservative chromated copper arsenate (CCA). CCA was the most common water-borne wood preservative used in the United States during the 1980s, 1990s, and early 2000s (AWPI, 1999). The wood preservation industry in the US stopped using CCA to treat most wood products intended for residential applications at the beginning of 2004 (US EPA, 2002). The use of CCA for preserving wood for industrial purposes (e.g., utility poles, marine pilings) continues. The arsenic in CCA acts as an insecticide, while copper functions as a fungicide. The hexavalent chromium in the preservative solution fixes or binds the arsenic and copper to the wood. The reduction of Cr(VI) to Cr(III) in the wood fiber matrix results in the formation of many sparingly soluble precipitates, including $CrAsO_4$, $Cu(OH)CrAsO_4$, $CuCrO_4$, and other wood-chromium complexes (Dahlgren and Hartford, 1972; Greaves, 1974; Bull *et al.*, 2000). The bulk of the fixation process occurs within a few days, with the rate of chromium fixation depending on environmental factors such as temperature (Cooper *et al.*, 1997).

While CCA-treated products may contaminate soil from preservative leaching during in–service use (Stilwell and Gorny, 1997; Stilwell and Graetz, 2001; Townsend *et al.*, 2003), chromium in treated wood leachates exists primarily as the less-toxic Cr(III) species (unless exposed to strongly oxidizing conditions such as some deck cleaners). The chromium encountered in CCA preservative solution exists as Cr(VI), however, and at locations where the preservative has entered the environment (e.g., a spill at a wood preserving facility), Cr(VI) concentrations in the soil and groundwater may exist at levels of possible concern to human health. Contamination at current and former wood treatment plants has been the subject of investigation by a number of authors (Anderson *et al.*, 1996; Bhattacharya *et al.*, 2002; Jang *et al.*, 2002). Most of these studies report only total chromium concentrations, and make assessment of the probable form of chromium based on measurements such as pH and oxidation reduction potential. Balasoiu *et al.* (2001) spiked a number of soils with CCA solution and reported that immediately after spiking, over 50% of the chromium remained in the hexavalent form; Cr(VI) presence was not measured directly, but was inferred from sequential extraction results.

This paper reports and discusses results of an experiment conducted to examine the speciation of chromium in three different soils contaminated with CCA preservative. The changes in chromium speciation in the sand, clayey, and organic soils were assessed over time by spiking the soils with CCA solution. The total and hexavalent chromium concentrations were then measured over an 11-month period. In addition, the leachability of trivalent and hexavalent chromium from the same soils was examined using the synthetic precipitation leaching procedure (SPLP), a standard test for evaluating the leaching potential of pollutants from contaminated soils. The speciation of chromium measured in the soil matrix was then compared with the amount of chromium extracted using the SPLP test.

Table 1
Physical and chemical properties of soils

		Sandy soil	Clay soil	Organic soil
pH		6.23	4.96	6.53
Eh (mV)		328	435	302
Moisture Content (%)		1.54	8.55	6.54
Bulk Density (g/cm^3)		1.5	1.4	1.2
% Organic Matter		1.33	1.16	5.57
Soil Texture	% Sandy	93.0	53.8	87.2
	% Silt	4.9	11.2	9.6
	% Clay	2.1	35.0	3.2
Classification		Sand soil	Sand clay loam soil	Loamy sand

Materials and Methods

Sample Collection and Characterization

Three different soil types were collected from two sampling locations in Alachua County, Florida, based upon guidance from local soil scientists: a sandy soil, a sandy clayey loam, and a sandy loam. One site was a source of the sandy soil, while the other site was a source of the loamy sand. Each site contained distinct clayey soil horizons, and the clayey soils from the two sites were combined to form one sample. The soils were collected from depths below 30 cm using a hand auger and a small shovel and were stored in 15-liter polyethylene containers. Each soil was characterized for pH (Method 9045C, US EPA, 1996a), oxidation-reduction potential (ORP) (ASTM D 1498-76), moisture content (ASTM D 4959-89), bulk density (of the disturbed soil), and organic matter (Walkley-Black Wet Combustion Method). The fraction of sand, silt and clay were estimated using the results of a grain-size distribution analysis. The results of these analyses are summarized in Table 1. The sandy loam was found to have the greatest organic content of the three soils and is thus referred to in this paper as an “organic” soil.

Preparation of Soil Samples

Each soil type was sieved using a ASTM#10 sieve (2-mm), air dried for 48 hours, and placed in a 15-liter polyethylene container after drying. Because of the presence of clods in the clayey soil, this soil type was first ground using a hand–powered grinding mill. Prior to spiking with CCA, each soil was first mixed thoroughly with a stainless steel scoop for 5 minutes. Two sets of 50-g sub-samples of each soil type were collected from the container and placed into 250-ml polyethylene bottles. The first set contained thirty-two 50-g sub-samples and was prepared for the analysis of total and hexavalent chromium concentrations in the soil matrix. A second set of twenty 50-g sub-samples was prepared for evaluating total and hexavalent chromium concentrations in the soil SPLP leachates. The soil in each 50-g sub-sample was spiked with 10 ml of CCA stock solution, which was obtained from a wood-preservative manufacturer (Chemical Specialties Incorporated, Charlotte, NC). The solution contained 2,700 mg/l hexavalent chromium and consisted of 45.5% CrO_3, 18.2% CuO, and 36.3% As_2O_5 (AWPA, 1998).

After spiking, the soil samples were mixed with a spatula. The targeted final concentration of chromium in the soil was 540 mg/kg (dry weight). This target concentration was selected so that the chromium concentrations in the soil would exceed the risk-based concentration thresholds used in Florida for assessing site cleanup requirements. The Florida soil cleanup target level (SCTL) for a residential exposure scenario is 210 mg/kg and the SCTL for an industrial exposure scenario is 420 mg/kg (FDEP, 1999). Both of these risk-based thresholds were developed utilizing toxicology properties of hexavalent chromium, and are relatively similar to risk thresholds in other US states. The sub-samples were stored under a fume hood in the laboratory. Selected interval times for analysis were very short during the beginning of the experiment (0, 0.5, 1, 2 days, etc.) due to the rapid rate of hexavalent chromium reduction in the soil samples. The time of "zero day" represents immediate analysis (approximately 15 minutes) after spiking the soil with the CCA solution. Time intervals were much longer toward the end of experimentation as a result of slower rates of reduction. Chromium reduction in the soils was measured over a period of 11 months.

Soil Extractions

Soil samples were subjected to three different extraction methods. The first two were digestion methods for extracting total chromium and hexavalent chromium from the soil matrices. Trivalent chromium was computed as the difference between total and hexavalent chromium. The third extraction method was for generating leachates under simulated rainfall conditions.

Total chromium was extracted from the soil matrix using the traditional acid digestion method, Method 3050B (US EPA, 1996a). The procedure involved refluxing 1 to 2 g of soil in sequential additions of concentrated nitric acid, hydrogen peroxide, and hydrochloric acid. Hexavalent chromium was extracted from the soil matrix using an alkaline digestion procedure, Method 3060A (US EPA, 1996a). This procedure began by mixing 2 g of soil with 50 ml of alkaline digestion solution and 0.5 ml of 1-M phosphate buffer solution. The alkaline digestion solution ($pH > 11.5$) was prepared with 20 g of sodium hydroxide and 30 g of sodium bicarbonate in one liter of reagent water. The solution was heated at 90 to 95°C, cooled and filtered through a 0.45-μm membrane filter. The pH of the filtered digestate was adjusted to pH 9.0 ± 0.5 and then diluted to 100 ml with reagent water. The filtered extracts from each digestion method were analyzed for total chromium and hexavalent chromium, respectively.

Soil leachates were generated using the SPLP, Method 1312 (US EPA, 1996a), which is designed to simulate contaminant leaching from soils or wastes when exposed to acidic rainfall. The method was modified slightly in that 10 g of soil were tested instead of 100 g. The liquid-to-solid ratio (20:1) required by the method was maintained. Two hundred ml of SPLP extraction fluid ($pH = 4.2$ prepared by a 60/40 weight percent mixture of diluted sulfuric and nitric acids) and 10 g of soil were combined into a 250-ml polyethylene bottle and rotated end-over-end for 18 hours. The leachates were filtered using a 0.45-μm membrane filter and then analyzed for total and hexavalent chromium.

Analytical Procedures for Total and Hexavalent Chromium Determinations

Total chromium was analyzed in the acid digestates using an inductively coupled plasma atomic emission spectrometer (ICP-AES, Thermo Jarrell Ash Corp. Model 3600, Method

Table 2
Experimental conditions for Cr(VI) analysis using ion chromatography

Ion Chromatography	Experimental conditions
Eluent Pump	Perkin Elmer Series 40 liquid chromatograph pump
Postcolumn Reagent Pump	Gilson model 1302 pump
Integrator	Hewlett Packard HP 3396A integrator
Detector	Hitachi UV detector L-7400
Guard Column	IonPac NG1
Analytical Column	IonPac AS7
Eluent	250 mM Ammonium sulfate/100 mM Ammonium hydroxide
Eluent Flow Rate	1.5 ml/min
Postcolumn Reagent	2 mM diphenylcarbizide, 10% Methanol, 1 N Sulfuric acid
Postcolumn Reagent Flow Rate	0.6 ml/min
Detection Wavelength	520 nm
Sample Volume	250 μL

6010B, US EPA, 1996a). The detection limit for chromium on this instrument was 0.17 mg/L. Hexavalent chromium in the alkaline digestates was analyzed by using Ion Chromatography (Method 7199, US EPA, 1996a). The principle of analysis involved the separation of hexavalent chromium on an analytical column. The hexavalent chromium was then reacted with diphenylcarbazide and this complex was detected at a wavelength of 520 nm. Table 2 presents the experimental conditions for chromium speciation by the IC system. The detection limit of the IC method was 0.04 mg/L.

The SPLP leachates were separated into two sub-samples: one analyzed for total chromium and the other analyzed for hexavalent chromium. The sub-sample used for total chromium analysis was digested through sequential additions of concentrated acids, Method 3010A (US EPA, 1996a) and then analyzed using ICP-AES as described above. Hexavalent chromium was analyzed using the 1,5-diphenylcarbazide colorimetric method (Method 7196; US EPA, 1996a). The colorimetric analysis was conducted by adding 1,5-diphenylcarbazide to a 25 ml sample of leachate. The intensity of the resultant purple color for samples containing hexavalent chromium was measured using a spectrophotometer (HACH Model DR/400U) at 540 nm. The detection limit was 0.003 mg/l. The colorimetric method was chosen for hexavalent chromium analysis of the SPLP leachates since minimal interferences were noted as opposed to the alkaline digestates, which resulted in considerable interference and thus required analysis by IC.

Results

Total and Hexavalent Chromium Concentration in Soil

The target concentration of total chromium in the soil samples was 540 mg/kg. Since the total chromium concentration was not expected to change through the course of the experiment, it was analyzed less frequently; the results were used to provide an estimate of the variability resulting from the spiking procedure. Six of the 18 sets of prepared soil

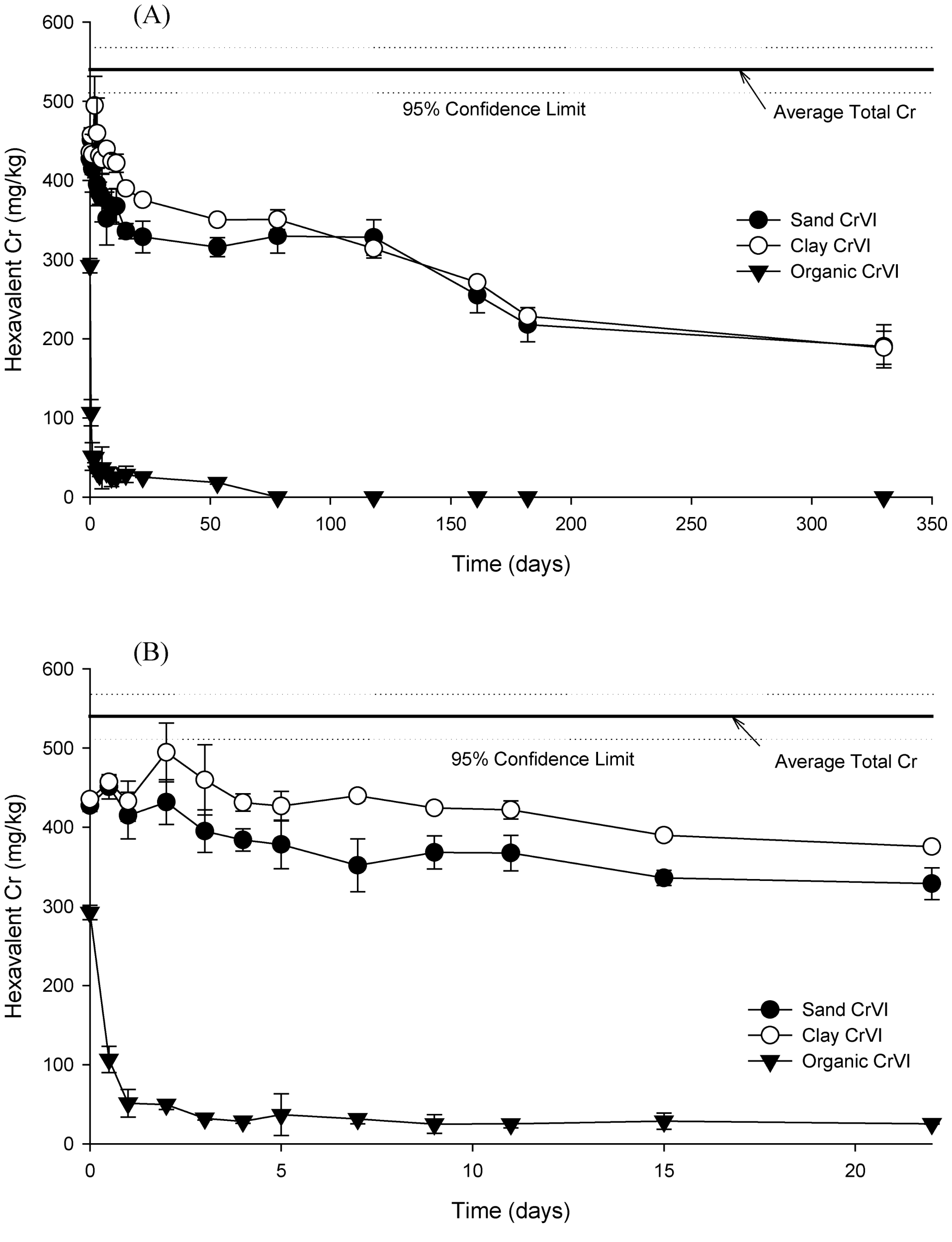

Figure 1. Hexavalent chromium reduction in soil matrices versus time (A) entire study, (B) 0–24 days.

samples were analyzed for total chromium (days 9, 22, 54, 79, 110, and 178). The average concentrations of total chromium for sandy, clayey, and organic soils were 516, 537, and 540 mg/kg, respectively. The overall average total chromium concentration for all 3 soils was 531 mg/kg. The standard deviation of this analysis was 21 mg/kg and the 95% confidence interval ranged from 509 and 554 mg/kg.

Figure 1 presents the concentration of hexavalent chromium measured in the soil samples over time. The error bars corresponding to the individual data points represent the standard deviation of triplicate measurements. For comparison purposes, the average measured total chromium concentration (531 mg/kg) is presented as a solid line and the 95% confidence interval is plotted as dashed lines. Since many more data points were collected during the early phases of the experiment, the data up through day 24 are illustrated separately. Several observations are readily notable (see Figure 1B). Reduction of hexavalent chromium in all three soils is evident, but much more so for the organic soil. The Cr(VI) concentration in the organic soil decreased dramatically within 2 days, and then decreased slowly thereafter. The immediate reduction observed for the organic soil (within 15 minutes after spiking) was approximately 44%. No hexavalent chromium was detected after 78 days in any of the organic soil samples. Immediate Cr(VI) reduction on the order 15% was observed with the sand and clayey soils. A dramatic drop in Cr(VI) from day 0 to day 0.5 was not noted for these soils as was observed with the organic soil. The reduction rate in the first month of the experiment was greater than that observed in the remaining 10 months. Approximately 50% of the hexavalent chromium was reduced during the 11-month experiment.

Total and Hexavalent Chromium Concentration in SPLP Leachates

Figure 2 presents the total and hexavalent chromium concentrations in the SPLP leachates. As was the case with the total chromium measurements in the soil, the SPLP leachable total chromium measurements were measured less frequently than for Cr(VI). In Figure 2a, the theoretical maximum concentration of chromium represents the leachate concentration that would occur if 100% of the chromium spiked to the soil leached during the 18-hour SPLP. The total chromium SPLP concentrations decreased as the experiment progressed. The decrease was most rapid during the first 20 days and slower after that. The pattern of total chromium concentration decrease over time in the SPLP leachates was consistent with the decrease in Cr(VI) concentrations observed from the analysis of the soil. The organic soil was observed to have the smallest concentration of chromium leaching. The chromium concentrations in the sand and clayey soils were greater and of similar magnitude.

While Cr(VI) measurements in the SPLP leachates were performed in greater frequency relative to the total chromium concentrations in the SPLP leachates, the Cr(VI) leaching pattern was similar to that observed for total chromium. The comparison between the two is better illustrated in Figure 3 where the total and hexavalent chromium concentrations measured in the SPLP leachates are plotted together for each of the three soil types. In each case, the total and hexavalent chromium concentrations are very close, with the total concentrations measured only slightly higher than the Cr(VI) concentrations. The contribution of trivalent chromium to the SPLP leachates appears to be very minimal.

Discussion

Cr(VI) reduction was evident in all three soil types, with the reduction most pronounced in the loamy sand (the organic soil). The organic matter content in the loamy sand was 5.6%, compared with 1.3% in the sand and 1.2% in the clayey soil. Cr(VI) reduction was greater in the sandy soil relative to the clayey soil during the first few months of the experiment; although reduction was similar for both soils after 11 months. It is generally known that hexavalent chromium is reduced to trivalent chromium in the presence of organic matter in soil (Wittbrodt and Palmer, 1995; Deng and Stone, 1996). Kozuh *et al.* (2000), for example, spiked several soil types with aqueous solutions of $K_2Cr_2O_7$ and $CrCl_3$ and found greater Cr(VI) reduction in a high-organic peat relative to other soil types containing less

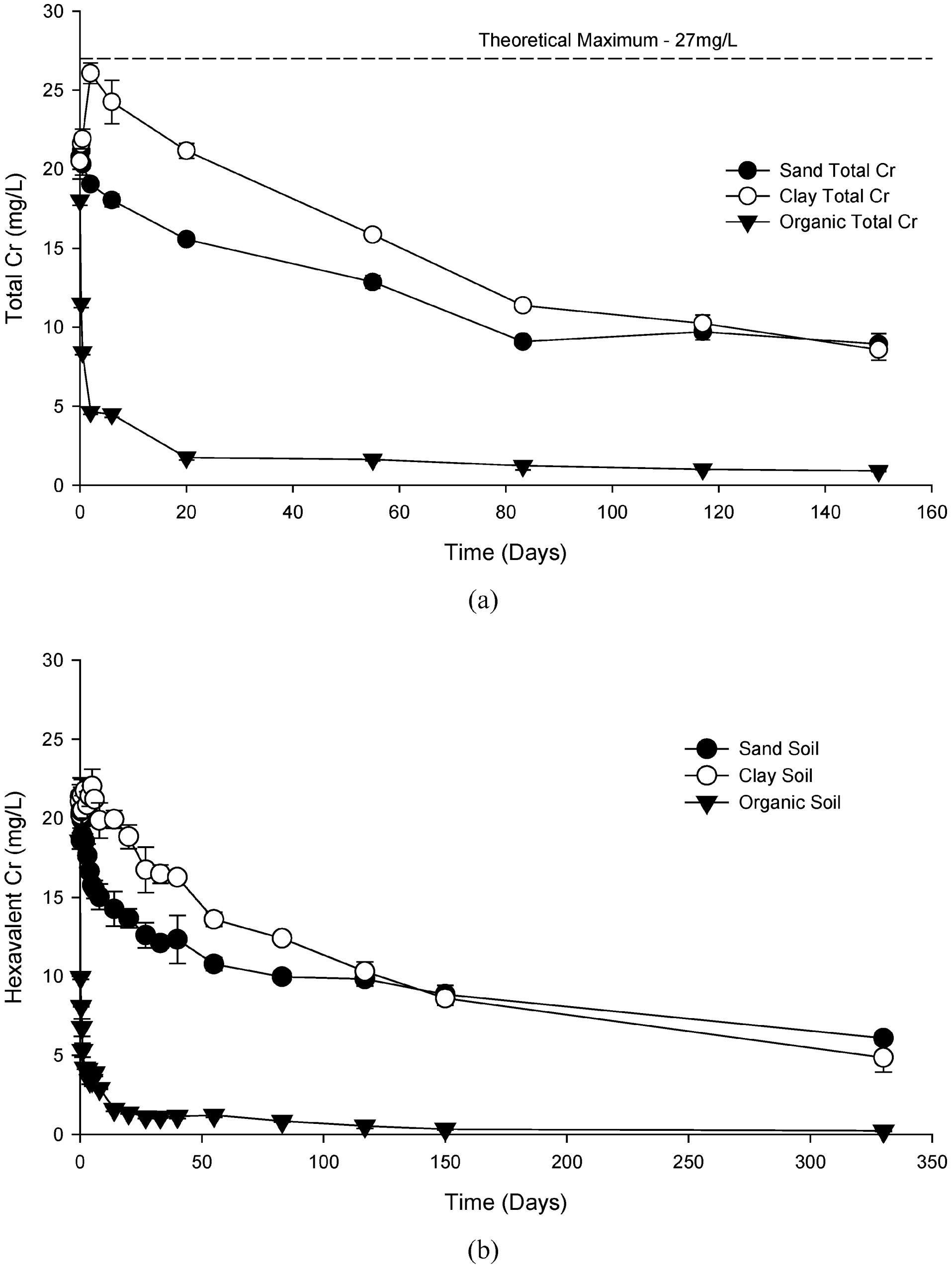

Figure 2. Total chromium(a) and hexavalent chromium(b) in SPLP extracts over time.

organic matter. Humic substances provide a reservoir of electron donors for reduction of hexavalent chromium to trivalent chromium. Possible Cr(VI) reduction by iron or sulfides was not investigated. The initial pH of the soils examined did not appear to dictate Cr(VI) reduction, as the soil with the lowest pH (the clayey soil) showed the smallest degree of reduction.

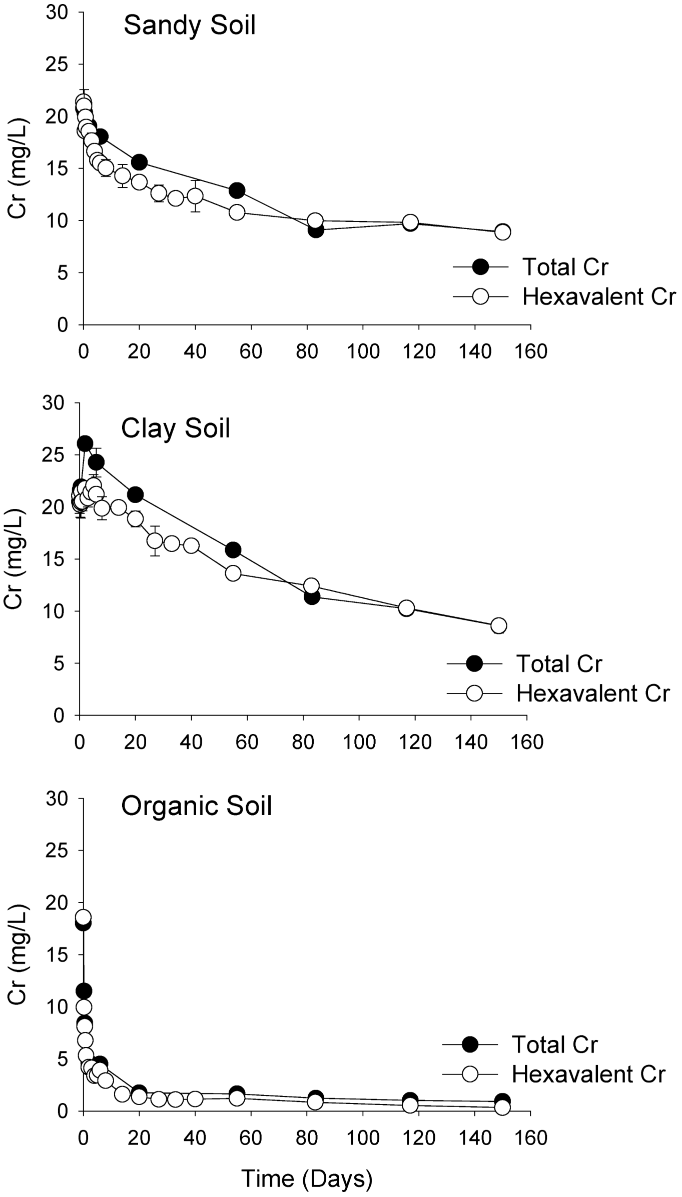

Figure 3. Total chromium and hexavalent chromium in SPLP extract from each soil.

The total and hexavalent chromium concentrations observed in the SPLP leachates decreased over time in a similar fashion as the reduction in Cr(VI) in the soil itself. This observation, coupled with the fact that nearly all of the leachable chromium occurred as Cr(VI), follows the classic description of Cr(VI) being much more mobile in soil in comparison to Cr(III). For example, when the US EPA developed soil screening levels to assess potential leachability of soils, the partition coefficient values they derived from chemical speciation modeling (at a pH of 6.0) were 200,000 L/kg for Cr(III) and 23 L/kg for Cr(VI) (US EPA, 1996b). The amount of Cr(VI) that leached from the soils was compared to the

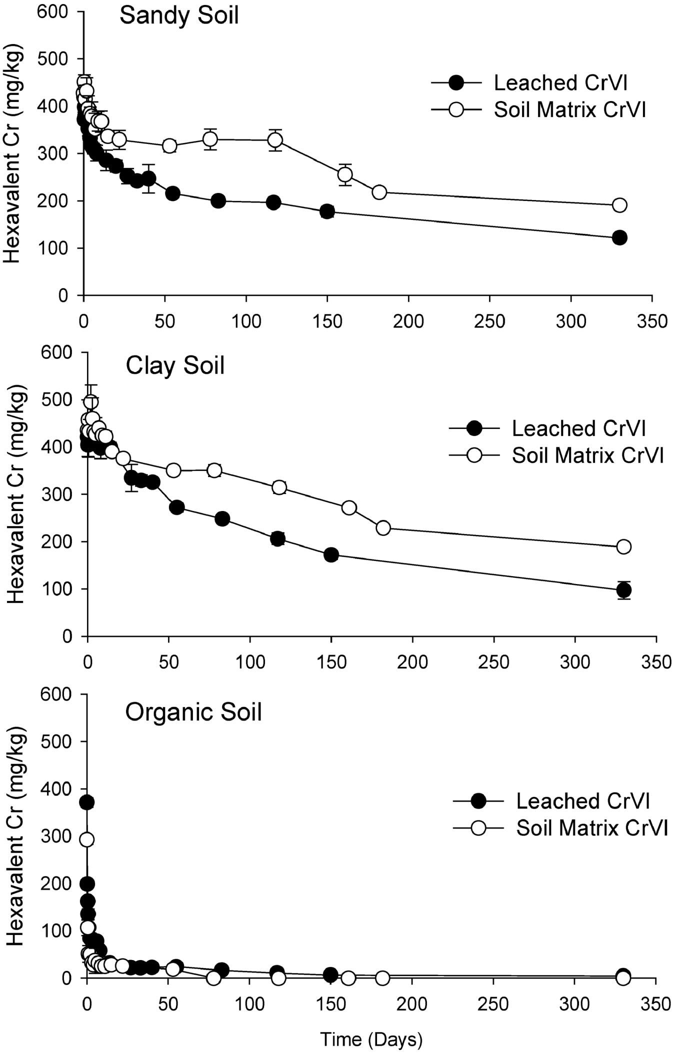

Figure 4. Comparison of Cr(VI) leached using SPLP and Cr(VI) in soil matrix.

Cr(VI) directly measured in the soils. This is illustrated in Figure 4 for each soil. The results indicate that for the sandy and clayey soils, while most of the Cr(VI) is leached off during the SPLP, some does remain bound to the soil at the end of the experiment (approximately 50% for the clayey soil and 35% for the sand). Cr(VI) concentrations were below detection at the end of the experiment for the organic soil.

The results do indicate that Cr(VI) can remain in soils where CCA preservative has been spilled for relatively long periods of time, and thus poses a possible risk to humans through direct exposure and to groundwater from leaching. The amount of CCA solution added to the soil samples was selected so that the initial Cr(VI) concentration would be greater than Florida's Cr(VI) risk-based generic cleanup guideline for direct exposure. The Cr(VI) concentrations in the sandy and clayey soils decreased to Florida's industrial exposure setting SCTL (420 mg/kg) after 3 days and 15 days, respectively, while more than six months elapsed before the Cr(VI) chromium concentrations declined to the residential exposure setting SCTL (210 mg/kg). In contrast, organic soil required only 12 hours to reach the residential SCTL, while it reached the non-residential SCTL immediately. After 11 months, hexavalent chromium concentrations were observed at 190 mg/kg for the sandy soil, 189 mg/kg for the clayey soil, and below the detection limit for organic soil. The results suggest that while reduction should occur naturally in the environment, Cr(VI) concentrations may pose health risks for substantial periods of time after being introduced into the environment. Certainly the organic content of soil will play a major factor in determining the rate and capacity of Cr(VI) reduction, as will other mineralogical properties of the soil.

A common practice for evaluating the groundwater contamination potential of an impacted soil is to compare SPLP concentrations to risk-based water quality thresholds. In many cases, states adopt drinking water standards for these thresholds. The groundwater cleanup target level (GWCTL) for chromium in Florida is 0.1 mg/L (based on the toxicity of Cr(VI)). All three soils exceeded this concentration at the end of the study. The ability to measure the Cr(VI) was more sensitive for the SPLP leachates relative to the soil extractions. From a risk assessment standpoint, risk to groundwater would appear to be more limited than direct exposure. Certainly other factors would dictate the true impact to groundwater, including depth of the vadose zone in relation to the amount spilled and the characteristics of the aquifer. An interesting observation is that the SPLP did provide a reasonable method for extracting and quantifying leachable Cr(VI). Since the chemical makeup of a leaching fluid may cause reduction of leached Cr(VI) to Cr(III), it is common for regulatory agencies to require leach testing using deionized water if Cr(VI) is to be measured in the leachate. For example, California requires that wastes leach-tested for Cr(VI) must use deionized water instead of the buffered citric acid solution normally utilized for leach testing of solid wastes. For the soils examined in this study, Cr(VI) concentrations were maintained at the end of the 18-hour SPLP.

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