

Arsenic immobilization in soils amended with drinking-water treatment residuals

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Land-applied drinking-water treatment residuals immobilize arsenic in soils.

Abstract

Use of Fe/Al hydroxide-containing materials to remediate As-contaminated sites is based on the general notion that As adsorption in soils is primarily controlled by Fe/Al (hydr)oxides. A low-cost and potentially effective substitute for natural Fe/Al hydroxides could be the drinking-water treatment residuals (WTRs). Earlier work in our laboratory has shown that WTRs are effective sorbents for As in water. We hypothesized that land-applied WTRs would work equally well for As-contaminated soils. Results showed that WTRs significantly ($p < 0.001$) increased the soil As sorption capacity. All WTR loads (2.5, 5, and 10%) significantly ($p < 0.001$) increased the overall amount of As sorbed by both soils when compared with that of the unamended controls. The amount of As desorbed with phosphate (7500 mg kg⁻¹ load) was ~50%. The WTR effectiveness in increasing soil As sorption capacities was unaffected by differences in both soils' chemical properties.

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1. Introduction

Inorganic arsenicals are classified as the number one toxin in the USEPA list of prioritized pollutants. Arsenic can be found in surface, subsurface water bodies, and many foods, although the risk exposure is greater in drinking-water than food (Brown and Ross, 2002). Arsenic occurs naturally in the geosphere, but there are anthropogenic inputs of As, as well. Over the last 100 years, arsenical compounds have been used in several industrial applications, such as, electrophotography, catalysts, pyrotechnics, antifouling paints, pharmaceutical substances, dyes and soaps, ceramics, alloys (automotive solder and radiators), battery plates, optoelectronic devices, semiconductors, and light emitting diodes in digital watches (NRC, 1999). Sixty percent of the anthropogenic As inputs come from only two sources: Cu-smelting and coal

combustion (Matschullat, 2000). Other anthropogenic sources include herbicide, pesticide and rodenticide use, as well as, waste incineration, steel/glass production, and pressurized wood production (Matschullat, 2000).

The major sources of As in soils are pesticides, desiccants, and fertilizers (Smith et al., 1998). Sodium arsenate (SA) is a pesticide that has been extensively used in the past in agricultural land, elevating soil As concentrations beyond background levels. The risk of human contact with soil As has greatly increased in the last two decades as a result of expanding residential areas into former agricultural land. Elevated As concentrations were found in soils used for residential developments located on former apple orchards that had been amended with arsenical pesticides for years (Murphy and Aucott, 1998).

Conventional remediation techniques involve excavation and some form of ex situ treatment (soil washing, solidification, etc.), followed by disposal and long-term monitoring, which are often expensive and disruptive to the surrounding landscape (Seaman et al., 2003). Therefore, it is imperative

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to develop techniques that can treat and stabilize contaminants in situ in an efficient and cost-effective manner. Examples of in situ treatment techniques include soil flushing, electro kinetics, bioremediation, vacuum or air stripping, and immobilization. In situ immobilization is a cost-effective approach where land-applied amendments are used to remove contaminants via adsorption and or precipitation reactions that render the contaminant immobile (Adriano, 1987). Numerous inorganic amendments (clays, Al/Fe/Mn oxides and hydroxides) may be land-applied to As-contaminated soils as a means of reducing As mobility (Hartley et al., 2004).

Use of Fe/Al hydroxide-containing materials to remediate As-contaminated sites is based on the general notion that As adsorption in soils is primarily controlled by Fe/Al (hydr)oxides (Livesey and Huang, 1981; Goldberg, 1986, 2002). A low-cost and potentially effective substitute for natural Fe/Al hydroxides could be the drinking-water treatment residuals (WTRs). The WTRs are the waste material generated during the drinking-water treatment process, and are primarily composed of Fe/Al (hydr)oxides, but they may also contain some activated C, and high molecular weight, long-chain, water soluble organic compounds (Elliott and Dempsey, 1991).

Earlier work by the authors has demonstrated the efficacy of WTRs in removing As from water (Makris et al., 2006). However, there is no published work on As sorption by WTR-amended soils. Arsenic-contaminated soils pose serious risk to human health and such soils that are characterized by low As sorption capacities due to low Fe/Al hydroxide content may be most vulnerable to As mobility and transport. We hypothesized that land-applied WTRs would significantly increase the As retention capacity of such soils, decreasing As mobility and transport risk. Another batch of the same Al-WTR used in the study was proven to effectively immobilize soluble phosphorus in a manure-impacted Spodosol (Silveira et al., 2006). This incubation study aims at addressing the effectiveness of two WTRs in reducing soluble As concentrations in two FL soils incubated with sodium arsenate pesticide. In effect, the main objectives of this study were: (i) to evaluate the effects of WTR type (Fe- or Al-based) and application rate on As immobilization in two soils with contrasting physicochemical properties, and (ii) to determine the As desorption potential in the presence of WTRs from the As-loaded soils using high rates of common fertilizer P.

2. Materials and methods

2.1. Soil and WTR sampling and characterization

Two surface (0–12 cm depth) FL soils were used in this study: soil samples from the Immokalee series were collected from the Southwest Florida Research and Education Center, Immokalee, Florida, and Millhopper soil samples were collected from the University of Florida campus at Gainesville, Florida. The soils were selected based on their presumed As retention capacities; Immokalee soil is a typical FL sand (93%) with minimum As retention capacity, Millhopper soil is a sandy loam with relatively higher concentration of Fe/Al hydroxides than that for Immokalee soil, thus, it is expected to have higher As sorption capacity than Immokalee. Prior to their use, soil samples were air-dried and passed through a 2-mm sieve. The WTR samples were also collected from two FL-based drinking-water treatment facilities:

the Al-WTR was obtained from the Manatee County water treatment plant in Bradenton, FL and the Fe-WTR was obtained from the Hillsboro river water treatment plant in Tampa, FL. The WTRs were air-dried, ground and passed through a 500- μ m sieve before use.

Soil and WTR samples were analyzed for pH, electrical conductivity, and water content using standard protocols. Organic matter content was determined using the loss-on-ignition method (Klute, 1996). Total C and N were determined by combustion at 1010 °C using a Carlo Erba NA-1500 CNS analyzer. Oxalate-extractable Fe and Al concentrations of the soils and WTRs were determined using Tamm's reagent (Klute, 1996). Total-recoverable Ca, Mg, Fe, Al, P, and As concentrations were measured in acid digests according to USEPA method 3050B (USEPA, 2000). Toxicity Characteristic Leaching Procedure (TCLP) was used for the WTRs to evaluate their potential for waste leaching in a landfill environment (EPA method 131). The concentrations of several metals and metalloids in the extract of the TCLP method were then evaluated against regulatory levels to determine whether WTRs exhibits the Toxicity Characteristic (TC).

Phosphorus was colorimetrically measured with a UV/vis spectrophotometer, using the molybdate–ascorbic acid method (Watanabe and Olsen, 1965). Soluble Fe, Al, Ca, and Mg were analyzed using flame atomic absorption spectrometry (FAAS). Total soluble As was analyzed using graphite furnace atomic absorption spectrometry (GFAAS).

2.2. Soil spiking

Incubation studies were conducted to evaluate the ability of WTR to bind As spiked to Immokalee and Millhopper soils. Stock As solutions were prepared in 0.01 M KCl using sodium hydrogen arsenate ($\text{NaH}_2\text{AsO}_4 \cdot 7\text{H}_2\text{O}$) as the As source. Potassium phosphate monobasic (KH_2PO_4) was used as the P source and stock P solutions were also prepared in 0.01 M KCl. The water holding capacity of the soils was calculated; WTRs were thoroughly mixed with the soils at 2.5, 5, and 10% rate by weight, and equilibrated for 7 d. Four solid:solution ratios, 1:5, 1:10, 1:25 and 1:50 (g solid:mL of 0.01 M KCl) were selected to determine the optimum solid:solution ratio for the As sorption/desorption studies of the WTR-amended soils. WTR samples were reacted with As solutions to attain initial As loads of 225, 2250 and 7500 mg kg^{-1} . No pH control was imposed. However, pH of all the solutions was measured before and after arsenic adsorption experiment. Suspensions were shaken on a reciprocating shaker at 180 rpm for 2 d. After equilibration, samples were centrifuged at 4000 rpm for 20 min, filtered, and analyzed for As with GFAAS.

2.3. Arsenic sorption kinetics with WTRs

Selected time intervals ranging from 0.17 to 48 h were used to determine the effect of contact time on As adsorption by WTRs. Three As loads (225, 2250 and 7500 mg kg^{-1}) were used in this experiment at the optimum solid:solution ratio (1:5 g:mL). Samples were shaken at 180 rpm on a reciprocating shaker, and samples were withdrawn at the predefined time intervals. Samples were centrifuged at 4000 rpm for 25 min; pH of the samples was measured and filtered through Whatman filter papers. Filtered solutions were analyzed for total soluble As with GFAAS.

2.4. Arsenic sorption/desorption experiments with soils

After initial equilibration of the soil–WTR mixture, representative soil samples amended with and without WTRs were reacted with inorganic As(V) in 1:5 (g soil:5 mL of 0.01 M KCl) solid:solution ratio at As loads of 125–8000 mg kg^{-1} for 2 d to determine As sorption capacities at 23 ± 2 °C. The pH of the suspensions was measured before, during and after 2 d and it was maintained at a pH of 6 by adding minor quantities of 1 M NaOH or HCl. The selection of the above range of As loads was based on preliminary As sorption experiments with WTRs in the absence of soils (Makris et al., 2006). Initial As loads exceeded those typically found in As-enriched soils, but were selected to account for cases of highly-contaminated As sites. After completion of the reaction period, samples were centrifuged; pH recorded, and analyzed for As.

After adsorption, As desorption experiments were initiated to study the desorption potential of As from WTR-amended soils using phosphate. Desorption of As, accounting for entrained As during adsorption was initiated by adding a high P load of 7500 mg kg⁻¹ and reacted for 2 d. The high P load used in the As desorption step simulated long-term (10–20 years) cumulative P loads added as P-fertilizers in agricultural fields. Suspensions were shaken at 180 rpm on a reciprocating shaker for 2 d, but the pH was not controlled. Samples were withdrawn and centrifuged at 4000 rpm for 20 min. Samples were filtered, and total soluble As was measured by GFAAS.

2.5. Statistical analyses

Data were analyzed for the main and interaction effects of the WTR rates and As load treatments in a completely randomized design using JMP software. Potential outliers were identified and were eliminated from statistical analyses. Pearson correlation coefficients were computed and data are reported as the mean of triplicate measurements \pm one standard deviation.

3. Results and discussion

3.1. Soil properties

The two soils and both WTRs used in this study were acidic (Table 1). Organic matter (OM) and nitrogen content of the WTRs was greater than that of the soils as one would expect with WTRs (Table 1). Drinking-WTRs are primarily physical mixtures of either Fe, or Al hydr(oxides) or CaCO₃, but they also contain appreciable amount of organic carbon, reaching levels on the order of 200 g kg⁻¹ regardless of the WTR class (Makris et al., 2005). The inherent amount of organic C in WTRs presents them with physicochemical properties that differ from those of typical amorphous Fe/Al hydroxides (Makris et al., 2005). Total Al concentrations were higher in the Al-based WTR (Al_T = 113 g kg⁻¹) than the Fe-WTR or the soils, but total Fe concentrations were higher in the Fe-WTR than the Al-WTR or the soils (Table 1). The high total Fe and Al content of the WTRs is due to alum or ferric chloride/ferric sulfate use as coagulants during drinking-water treatment. Oxalate-extractable Fe + Al concentrations have been used as an index of the material's reactivity in soils, since oxalate selectively extracts the amorphous Fe/Al hydroxides (McKeague et al., 1971). Jacobs et al. (1970) observed an increase in As retention in a soil with increasing concentration of amorphous Al/Fe hydroxides. The native Immokalee sand was extremely low in oxalate-extractable Fe and Al (0.08 g kg⁻¹) whereas the native Millhopper contained higher

Table 1
General chemical properties of the WTRs and soils used in this study

	Al-WTR	Fe-WTR	Immokalee	Millhopper
pH	5.1	5.4	6.1	5.8
EC ($\mu\text{S cm}^{-1}$)	363	164	59	145
OM (g kg ⁻¹)	330	400	8.4	43.8
C (g kg ⁻¹)	150	211	12.7	7.0
N (g kg ⁻¹)	6.0	10.0	1.06	0.66
Fe _T (g kg ⁻¹)	12.3	268	0.07	0.85
Al _T (g kg ⁻¹)	113.0	1.34	0.11	1.90
Fe _{ox} (g kg ⁻¹)	5.70	78.7	0.04	0.33
Al _{ox} (g kg ⁻¹)	82.0	0.36	0.04	0.51

oxalate-extractable Fe and Al concentrations (0.84 g kg⁻¹) (Table 1). The WTRs, as expected, have much higher Al_{ox} + Fe_{ox} concentrations than the soils. Oxalate extraction showed that 73 and 30% of the total metal was extracted with oxalate from the Al- and the Fe-WTRs, respectively (Table 1). This difference may suggest a lower reactivity of the Fe-WTR than the Al-WTR with respect to soluble As.

Toxicity characteristic values for As and other metalloids and metals for the WTRs according to the TCLP test were lower than those to classify them as hazardous wastes, permitting their land-application as a beneficial reuse of these waste materials (Table 2).

3.2. Effect of solid:solution ratio

This set of experiments was designed to optimize the effect of solid:solution ratio on the overall amount of sorbed As by both WTRs. Results from this experiment showed a significant interaction ($p < 0.001$) between the initial As load and the solid:solution ratios on the overall amount of sorbed As by both WTRs (Fig. 1). In effect, at low solid:solution ratios (1:5 and 1:10), there was no significant ($p < 0.05$) difference between different initial As loads on the amount of As sorbed by both WTRs. At the highest solid:solution ratios used (1:25, 1:50), there was a decrease in the amount of As sorbed when the initial As load was increased (Fig. 1). From this set of experiments, we chose the 1:5 solid:solution ratio as the optimum ratio to be used for the sorption As isotherms of the WTR-amended soils.

3.3. Effect of reaction time

This set of experiments was designed to optimize the effect of reaction time on the overall amount of sorbed As by both WTRs. Results from these experiments showed that As sorption by the Al-WTR was nearly complete (98%) almost instantaneously, regardless of the initial As load that ranged from 225 to 7500 mg As kg⁻¹ (Fig. 2). The high affinity of the Al-WTR for As prevented us from observing a kinetic effect, since the initial As loads used were inadequate to saturate the Al-WTR's As sorption capacity. The Fe-WTR showed a significant ($p < 0.001$) interaction between As load and reaction time. At small reaction times (up to 2 h), As sorption was linear for all As loads but proceeded slower thereafter, reaching finally 100% by the end of the contact time (48 h) (Fig. 2). The kinetic effect was observed clearly on the Fe-WTR but not on the Al-WTR, illustrating the greater amount of sorption sites of the Al-WTR (Makris et al., 2005). Arsenic sorption reached only an apparent equilibrium at 48 h due to the huge As sorption capacity of the WTRs that prevented us from observing a true equilibrium of the sorption process.

3.4. Arsenic sorption by Immokalee soil

Immokalee soil is a typical FL sand with minimum P sorption capacity (O'Connor et al., 2001), and thus, it is expected that As sorption capacity of Immokalee will be accordingly

Table 2

Toxicity characteristic values for several metals and metalloids of the Fe- and Al-based WTRs according to the TCLP method

	As (mg kg ⁻¹)	Cd (mg kg ⁻¹)	Cr (mg kg ⁻¹)	Cu (mg kg ⁻¹)	Pb (mg kg ⁻¹)	Hg (mg kg ⁻¹)	Ni (mg kg ⁻¹)	Se (mg kg ⁻¹)	Zn (mg kg ⁻¹)
Al-WTR	<0.506	<0.166	<0.168	<0.936	<1.092	11	<1.62	15	<0.054
Fe-WTR	<0.506	<0.166	<0.168	<0.936	<1.092	10.8	<1.62	13.4	<0.054
EPA limit	75	89	3000	4300	840	57	420	100	7500

low. The effect of WTR application rates on soil As sorption capacities was determined in incubation studies at the optimized 1:5 solid:solution ratio (Fig. 3). All of the WTR loads used (2.5, 5, and 10%) resulted in a significant ($p < 0.001$) increase in the overall amount of As sorbed by the Immokalee soil when compared with that of the unamended control soil samples (Fig. 3). Specifically, increasing the WTR load from 2.5 to 5 and 10% by weight resulted in a significant ($p < 0.005$) increase in the Immokalee soil As sorption capacity, reaching almost 5000 and 3200 mg kg⁻¹ for the highest WTR rate (10%) of the Al- and Fe-WTRs, respectively (Fig. 3). Typical agricultural field WTR loads used to immobilize soluble P concentrations lie within the 2.5% by weight application rate (Elliott et al., 2002). The WTR loads used in this study (2.5–10%) suggest that the negligible As sorption capacity of the Immokalee soil can be substantially increased when amended with either Al- or Fe-WTR.

The Al-WTR was more effective than the Fe-WTR in increasing Immokalee sand's As sorption capacity, regardless of WTR application rates (Fig. 3). This trend was corroborated by similar As sorption experiments with WTRs in the absence of soils (Makris et al., 2006). Earlier work by the authors has shown that the Al-WTR has a greater As(V) sorption capacity (at least 15,000 mg kg⁻¹) than the Fe-WTR (at least

9000 mg kg⁻¹) on similar As sorption experiments in the absence of soil (Makris et al., 2006).

Arsenic sorption data for the WTR-amended Immokalee soil samples were fit equally well to both Langmuir and Freundlich empirical models, reaching r^2 values >94% (data not shown). Similar data were obtained in the absence of soils, where Langmuir-based As sorption maxima were not calculated for different batched of the same WTRs used here, because As(V) sorption followed a linear or Freundlich type of adsorption isotherm (23 °C) (Makris et al., 2006).

3.5. Arsenic sorption by Millhopper soil

Millhopper is another typical FL soil that has a greater amount of Fe/Al hydroxides and organic C content than the Immokalee soil (Table 1). It was shown that the Millhopper soil exhibited greater As sorption capacity than the Immokalee soil in the absence of WTR treatment possibly due to Millhopper's greater amorphous Fe/Al content (0.84 versus 0.08 g kg⁻¹) (Fig. 4). The WTR application rate treatment had a significant ($p < 0.001$) effect on the amount of As sorbed by Millhopper

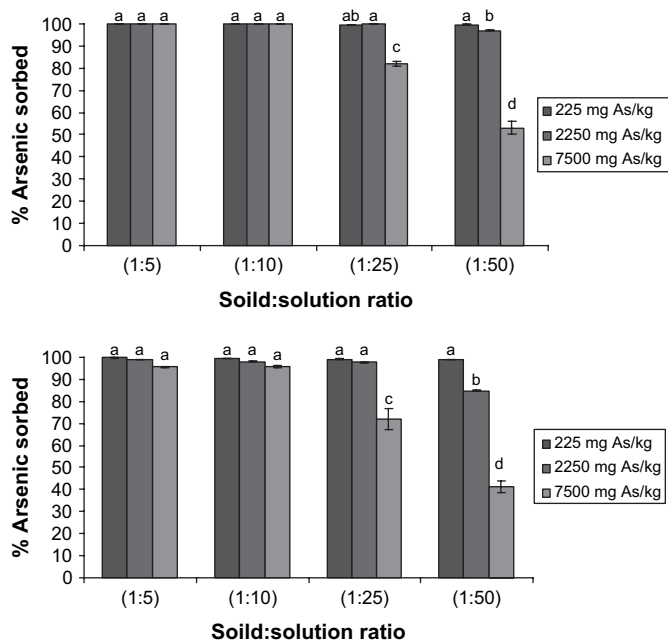


Fig. 1. Effects of solid:solution ratio and initial As load on the amount of As sorbed by the Al-WTR (top graph) and the Fe-WTR (bottom graph) after reaction for 2 d. Error bars represent ± standard error of the means. Means not connected by the same letter are significantly different from each other at the 95% confidence level.

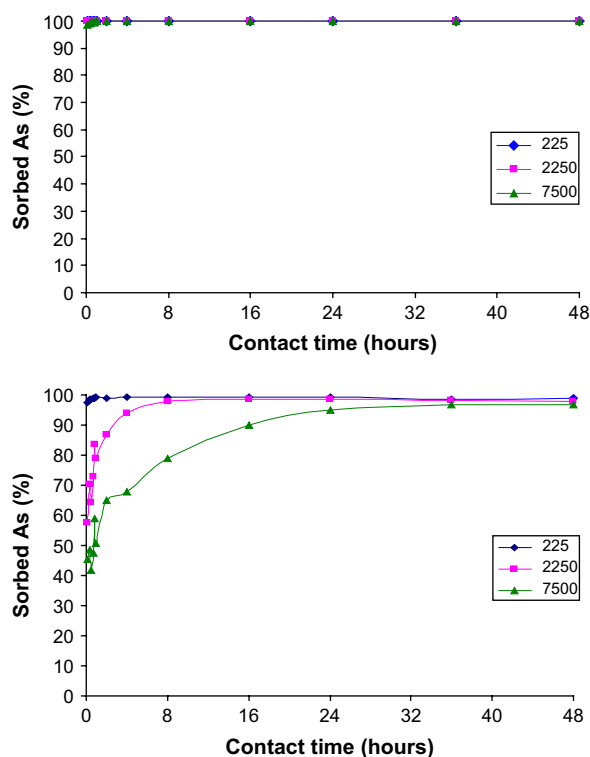


Fig. 2. Arsenic sorption kinetics for the Al-WTR (top graph) and Fe-WTR (bottom graph) at a 1:5 solid:solution ratio. Trendlines are not model fits and they are used only for visual purposes.

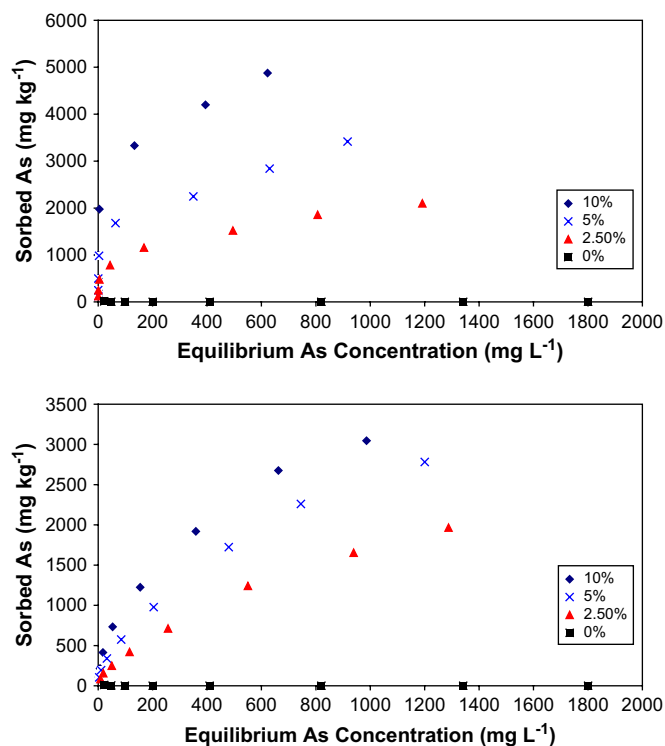


Fig. 3. Arsenic sorption isotherms (23 °C) for Immokalee soil amended with an Al-WTR (top graph) and Fe-WTR (bottom graph) at rates of 0, 2.5, 5, and 10% by weight. Solution pH was maintained at 6, and reaction time was 2 d. The solid:solution ratio was 1:5. Data are the mean of three replicates \pm one standard deviation.

soil, increasing the soil's As sorption capacity by at least 10-fold (Fig. 4). Similar to Immokalee soil, all of the WTR loads significantly ($p < 0.001$) increased the overall amount of As sorbed by Millhopper soil samples when compared with that of the unamended controls (Fig. 4). Interestingly, as you can see from both Figs. 3 and 4, WTR effectiveness in increasing soil As sorption capacities was unaffected by the soil's chemical properties despite the fact that Immokalee and Millhopper soils have distinctly different chemical properties (Table 1). Similar to Immokalee, both Langmuir and Freundlich models fit equally well the As sorption data for WTR-amended Millhopper samples, suggesting the huge affinity of both WTR-amended soils for As (data not shown).

3.6. Arsenic desorption from WTR-amended soil samples

Following As sorption, As desorption was initiated with a KH_2PO_4 solution reaching P load equivalent to 7500 mg kg^{-1} soil. The amount of arsenic desorbed from Immokalee soil amended with 2.5, 5 and 10% Al- and Fe-WTRs was proportional to the amount of previously sorbed As concentrations (Fig. 5). Despite the fact that increases in WTR application rate resulted in greater As sorption by the Immokalee soil, there was no effect of WTR application rate on the amount of desorbed As with phosphate (Fig. 5). The maximum amount of As desorbed with the 7500 mg kg^{-1} P load was approximately 50% for all WTR rates, regardless of WTR type (Fe- or Al-WTR) (Fig. 5). Arsenate desorption,

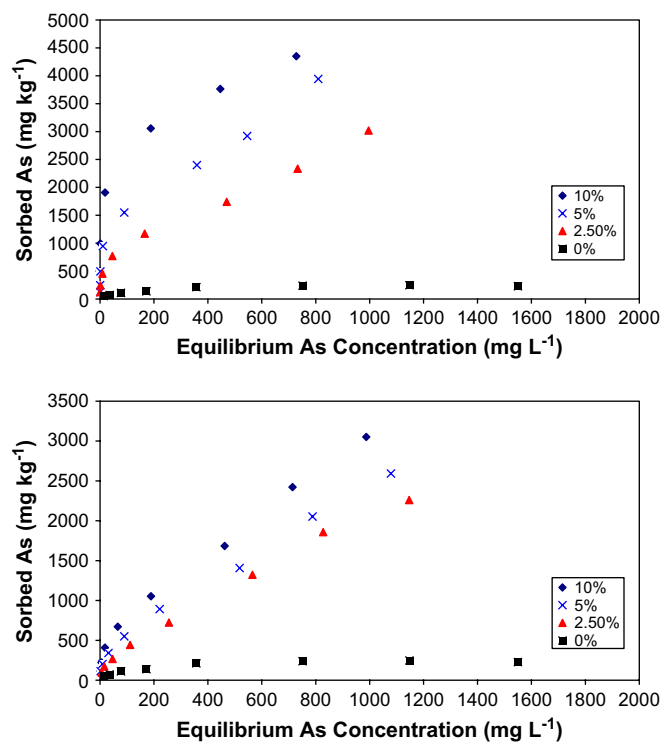


Fig. 4. Arsenic sorption isotherms (23 °C) for Millhopper soil amended with an Al-WTR (top graph) and Fe-WTR (bottom graph) at rates of 0, 2.5, 5, and 10% by weight. Solution pH was maintained at 6, and reaction time was 2 d. The solid:solution ratio was 1:5. Data are the mean of three replicates \pm one standard deviation.

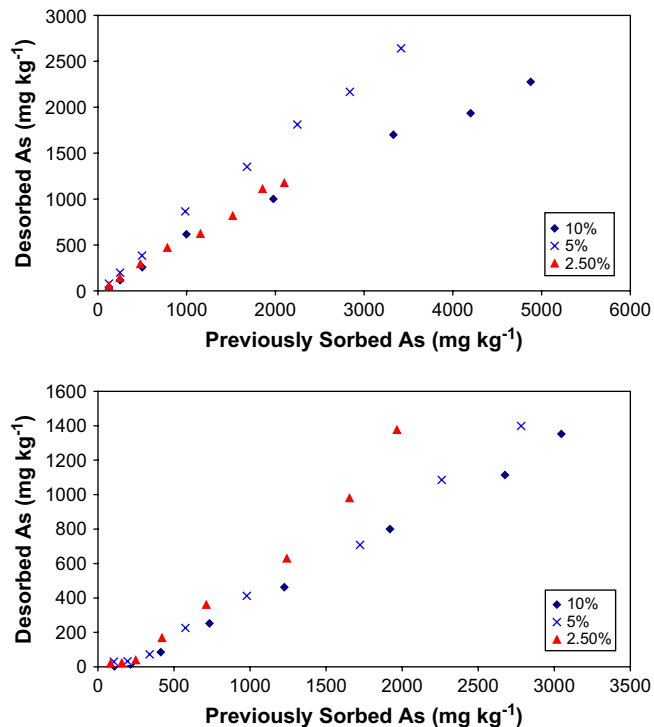


Fig. 5. Relationship between the amounts of previously sorbed As and the As desorbed with a $7500 \text{ mg P kg}^{-1}$ solution from the Al-WTR (top graph) and the Fe-WTR (bottom graph) amended Immokalee soil samples. The pH was not controlled and the reaction period was 2 d.

with almost half or less the P load used here, from amorphous Fe hydroxide at a pH of 7 was ~55% of previously sorbed As, whereas for crystalline goethite phosphate-induced As(V) desorption was only 25% (Jackson and Miller, 2000).

Similar results were obtained for the Millhopper soil (data not shown). In effect, As desorbed from the WTR-amended soil samples was not a function of the WTR application rates but a function of the amount of previously sorbed As. Similar to Immokalee, the amount of As desorbed with the 7500 mg P kg⁻¹ solution was approximately 50% for all WTR initial application rates (data not shown). The indeed high P load we added during the As desorption experiment was to simulate long-term (10–20 years) cumulative P added as fertilizers in agricultural fields. Even at such extremely high P loads, WTRs irrespective of type (Fe- or Al-based) or rate (2.5, 5, and 10% by weight) were able to retain at least 50% of previously sorbed As in two soils with varying physicochemical properties.

4. Conclusions

This incubation study demonstrated the effectiveness of WTRs in immobilizing As in As-contaminated soils. Two WTRs (one Al- and one Fe-based WTRs) known for their effectiveness in removing As(V) from contaminated water showed significant increase in a soil's As sorption capacity. Data from the solid:solution ratio experiment showed that the 1:5 solid:solution ratio was chosen as the optimum ratio to be used for the sorption As isotherms of the WTR-amended soils. Arsenic sorption by the Al-WTR was nearly complete (98%), almost instantaneously (within 0.5 h), regardless of the initial As load that ranged from 225 to 7500 mg As kg⁻¹. The high affinity of the Al-WTR for As prevented us from observing a kinetic effect, since the initial As loads used were inadequate to saturate the Al-WTR's As sorption capacity. The Fe-WTR showed a significant ($p < 0.001$) interaction between As load and reaction time. At small reaction times (up to 2 h), As sorption was linear for all As loads but proceeded slower thereafter, reaching finally 100% by the end of the contact time (48 h).

The WTRs were effective in immobilizing soluble As(V) in two FL soils that exhibited little As sorption capacities. The type of WTR used had a significant effect on the amount of As sorbed by the soils, since for the same WTR application rate, the Al-WTR was proven more effective in reducing soluble As concentrations in both soils. The WTR application rate had a pronounced effect on the overall amount of As sorbed by the soils, showing that the soil As sorption capacity was significantly increased in the order of 2.5 > 5 > 10% WTR rate. The amount of As desorbed from the WTR-amended soils was linearly related to the amount of previously sorbed As and was independent from the WTR application rate used. The maximum amount of As desorbed was approximately 50% due to the extremely high (7500 mg kg⁻¹) P load used to represent a worst-case scenario.

Data from this incubation study will be further tested in future field trials with As-contaminated soils. The effectiveness of WTRs in immobilizing As in As(III)-pesticide containing soils remains to be investigated. Data are encouraging but extra caution should be exercised before extrapolating this

set of data to other WTRs that differ in physicochemical properties from those used here.

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