

**Retention of As, Cu, and Cr leached from CCA-treated
wood products in select Florida soils**

Tait Chirenje^{1*}, L.Q. Ma², M. Szulczewski², K. Hendry² and C. Clark³

¹B108 NAMS, Richard Stockton College of New Jersey, PO Box 195,
Pomona, NJ 08240; Phone: 609 652 4588, Fax: 609 748 5515
tait.chirenje@stockton.edu (corresponding author)

²Soil and Water Science Department, University of Florida
Gainesville, FL 32611-0290, Ph: 352 392 1951 ext 215, Fax 352 392 3902

³Civil and Coastal Engineering Department, Gainesville, FL 32611, Ph: 352 392 9537 x 1441

Abstract

Up until recently, the use of chromated copper arsenate (CCA)-treated wood had been growing steadily in the United States. Chromated copper arsenate treatment arrests microbial and fungal decay of wood products. Due to the scale of the wood preserving industry, CCA-treated timber may form a significant source of the trace elements: chromium (Cr), copper (Cu), and arsenic (As), to the environment. The aim of this study was to determine the retention of As, Cu and Cr in common soil types in Florida. Soil samples from six soil groups (Histosols, Entisols, Alfisols, Ultisols, Spodosols and Marls) were collected from across the state. Profile samples were collected, with emphasis on surface and diagnostic subsurface horizons, where applicable. Column sorption and desorption studies conducted to determine the retention of leachates from construction and demolition debris-packed columns showed that fine textured soils had the largest retention capacity for Cu, Cr and As. Horizons in which large quantities of soil organic matter (SOM), fine clay, iron (Fe) and aluminum (Al) compounds accumulate had higher retention capacity of Cu, Cr and As than horizons lacking in these components. There were very little differences between surface horizons of all soil types, with the exception of Histosols, which are organic in nature, and Marls. Although the concentrations of the three elements in CCA-treated wood were not very different from each other, As leached out of the wood the most, with leachate concentrations at least an order of magnitude higher than those of Cr and Cu. These results are helpful for CCA-treated wood product users as a reference to determine the leaching potential CCA components from the most common soil groups of Florida.

Abbreviations:CCA, chromated copper arsenate, OC, organic carbon, SCTL, soil clean-up target level; SOM, soil organic matter; MDL, method detection limit

Keywords: CCA-treated wood, natural, anthropogenic background

Introduction

Untreated wood products do not last long because they are prone to fungal, bacterial and termite attacks. Consequently, differently treated wood products have found their way into the lumber market. Pressure treated wood products have gained popularity because of their durability, especially in warm humid climates like Florida. The most commonly used water based pressure treatment is chromated copper arsenate (CCA), where the copper (Cu) acts as a fungicide and the arsenate, an insecticide (Dawson et al., 1991). Chromium (Cr) is used to fix the other two elements to the cellulose and other components of the wood. Arsenic is generally used in two anionic forms, arsenite [As(III)] and the more mobile arsenate [As(V)]. Copper exists mostly in the cationic form Cu^{2+} , and although Cr is a cation, it commonly exists in two anionic forms, the more mobile and toxic chromate ions from Cr(VI), and the less mobile and toxic Cr(III).

The use of CCA-treated wood products invariably leads to the release of the constituent elements, arsenic (As), Cr, and Cu and their compounds into neighboring soils (Carey et al., 1996; Cooper and Ung, 1997; Lebow, 1996; Stilwell and Gorny, 1997; Solo-Gabriele et al., 2000). The mobility and retention of these CCA constituents is governed by the form they are released in. A considerable number of studies have shown significantly elevated metal concentrations near CCA-treated decks compared to background concentrations (Stilwell and Gorny, 1997; Chirenje et al., 2003b). Arsenic, Cr and Cu concentrations as high as 550, 200, and 1,000 mg kg^{-1} , respectively have been reported in the vicinity of utility poles (Cooper and Ung, 1997). Other studies have also looked at the effects of CCA-treated wood in aquatic systems due to the proliferation of both residential and public decks in coastal waterways (Hingston et al., 2001).

While the extent of release of As, Cu and Cr from in-service CCA-treated wood products and its contribution to environmental quality degradation is still a matter of great debate, it is generally agreed that the release of CCA constituents is governed by many factors. These include (i) the nature and surface area of the wood (Solo-Gabriele et al., 2000), (ii) the type (A, B, or C) and retention factor (0.25 to 2.5 lb ft^{-3} , depending on whether it is aboveground, marine or belowground; Hingstrom et al., 2001) of the CCA, (iii) climatic conditions (temperature, humidity and rainfall; Chirenje et al., 2003b), and (iv) soil factors (texture, pH, organic matter content, cation exchange capacity [CEC], ammonium oxalate extractable iron [Fe] and aluminum [Al]; Cooper and Ung, 1997; Kaminski and Landsberger, 2000). However, relatively little is known about the behavior of leached components in different soils and their long term impact on soil quality and environmental health.

Soil As concentrations range between 0.1 and 40 mg kg^{-1} worldwide, with an arithmetic mean (AM) concentration of $5\text{-}6 \text{ mg kg}^{-1}$ (Kabata-Pendias and Pendias, 1992). Baseline concentrations of As in relatively unimpacted Florida soils vary from 0.01 to 61.1 mg kg^{-1} , with a geometric mean (GM) of 0.27 mg/kg (Chen et al., 1999; Ma et al., 1997). Recent studies on anthropogenic baseline concentrations of As in urban areas, where there is likely to be greater anthropogenic impact as well as increased use of CCA-treated wood, revealed considerably higher As concentrations of about 0.40 mg/kg (GM; Chirenje et al., 2001; 2003a). The other two CCA constituents have not received the same attention as As, and data on their distribution is not as easily accessible.

The specific objective of this study was to determine the retention of As, Cu and Cr in six common soil types (Histosols, Entisols, Alfisols, Ultisols, Spodosols and Marls)

in Florida. Spodosols, Entisols, and Ultisols are the most prevalent soil orders in Florida, covering about 73 % of the state (Fig. 1). Although Marls do not constitute a soil order (they are mostly within the order Entisols), they have unique characteristics that warrant their treatment as a separate group. Attention was paid to both surface soils, which constitute the greatest route of exposure to human beings and other animals, and subsurface horizons, which present the route of exposure of groundwater pollution by these three elements. Results obtained from this study will improve our understanding of the retention of As, Cu and Cr in different types of soils and facilitate the differentiation of the effects of CCA-treated wood from those of naturally occurring baseline concentrations of As, Cu and Cr.

Methods

The primary objective of this study was to classify the retention of the three elements that can potentially leach out from CCA-treated wood (Cu, Cr, and As) in different soils of Florida. This was accomplished in three stages:

- i. sample collection from around Florida
- ii. sample analysis (adsorption, desorption, physical & chemical characteristics)
- iii. classification of sorption and desorption capacities of the different soil types

Soil sample collection

Soil samples from six soil orders were collected from various places in Florida (Fig 2):

1. A Histosol sample from Belle Glade, South Central Florida
2. A Marl sample from Homestead, South Florida

3. Seven samples from Austin Cary Memorial Forest near Gainesville, North Central Florida
 - a. One Spodosol
 - b. Three Entisols
 - c. One UltisolTwo were not classified (only A horizons were sampled)
4. Two samples from Lakeland and surrounding areas, Central Florida
 - a. One Entisol
 - b. One Spodosol
5. Six samples from Panama City, Tallahassee and Marietta, Northwest Florida.
 - a. Five Entisols
 - b. One Alfisol

Soil sample analyses

Soil concentrations of As, Cu and Cr were determined by digestion using an EMS hot block digester, modified from the United States Environmental Protection Agency (USEPA) Method 3050 (USEPA, 1995; 1996). Half a gram of soil was weighed into a 50-mL Teflon tube and digested in 9 mL concentrated HNO₃. The resulting solution was diluted to 50 mL and filtered through a 0.45 micron membrane filter. Arsenic, Cu and Cr concentrations in the digestates were determined with a SIMAA 6000 graphite furnace atomic absorption spectrophotometer (GFAAS, Perkin Elmer, Norwalk, CT), using USEPA method 7060A (USEPA, 1995). Arsenic, Cu and Cr concentrations in column leachate samples were determined the same way after filtration through a 0.45 micron

membrane filter. Standard reference materials (SRM 2709, Montana soil from NIST) were used to check the extraction efficiency of the digestion method used for all soil samples. Approximately 20 % of the samples analyzed were spikes, duplicates and reagent blanks, which were used for quality assurance/quality control. Digestion sets showing a relative standard difference of more than 20 % from the known values (for standards and spikes) were repeated.

Determination of the relative Cu, Cr, and As sorption capacity of soils

Determination of adsorption isotherms

The soil samples were exposed to a minimum of five predetermined Cu, Cr, and As concentrations and sorption was determined at each of those concentrations. This was achieved by adding 10 mL of solution of desired Cu, Cr, or As concentration to 10 g of soil in a scintillation vial, mixing and centrifuging and then measuring the Cu, Cr, and As concentration in the soil.

Determination of Cu, Cr and As desorption capacity

The soil samples used in adsorption were leached with 5mL portions of a weak salt, 50 mM KCl over a period of 6 weeks. The concentrations of Cu, Cr and As in each aliquot were measured. Other soil chemical properties of relevance e.g. ammonium oxalate extractable iron (Fe) and aluminum (Al), soil organic matter (SOM), pH were determined as needed.

Determination of retention indices

Regression analyses was used to establish an empirical relationship between soil retention capacity for Cu, Cr, and As and soil properties including pH, SOM, cation exchange capacity (CEC), ammonium oxalate extractable Fe and Al, and texture. In the end, soils were grouped into the following classes for each element:

Class 1: Soils with the greatest retention and minimal likelihood for leaching,

Class 2: Soils with moderate retention with moderate potential for leaching, and

Class 3: Soils with the least retention, greatest risk for leaching

Results and discussion

Variation in soil properties

Soil properties varied greatly between orders, with surface soil pHs ranging from 4.4 in Ultisols to 7.9 in Marls (Fig. 3). The low pHs in Ultisols and Spodosols were a result of the vegetation in the areas where these samples were collected. Most Spodosols in Florida are associated with pine stands whose needles acidify surface soils when they decompose on the ground. On the other extreme end, the high pH in Marls was a result of the mineral components of the soils, which are mostly composed of calcite. Subsurface pHs did not vary to the same extent as surface pHs. Soil pH is critical because the speciation and subsequent leachability of trace elements is considerably affected by pH. Most elements with high association with OC, e.g. Cu, are leached out more at higher pH due to the dissolution and subsequent mobilization of SOM. Those elements that form strong association with CaCO_3 , e.g. As, have higher retention at higher pH, but they are more mobile at lower pHs as the calcite is dissolved by acid. However, in the presence of

high concentrations of ammonium acetate extractable Fe and Al, As is not very mobile at low pH due to its precipitation with both ammonium acetate extractable Fe and Al.

Surface soil textures also varied considerably, with Marls having the highest clay + silt content (> 90 %) and Spodosols having the least (< 10 %; Table 1). Table 1 shows the variation of soil texture with horizon across soil orders. There was very little variation observed between A and E horizons although B horizons in both Ultisols and Spodosols showed lower clay+silt content than expected. Soil texture affects soil surface area, with finer textured soils having more surface area and being more reactive, and therefore becoming more likely to retain higher amounts of trace elements than coarse textured soils (Chen et al., 1999; Berti and Jacobs, 1996). Figure 3 demonstrates the increased As retention in the finer size fraction of the different soil types in the study. The same trend was observed for Cu and Cr (data not shown). The silt+clay fractions varied between soil types and therefore any inferences from comparisons between soils should take this into consideration. Arsenic concentrations were highest in the silt+clay fraction in the Marl, which translated to this soil having the highest concentration of As in all the soils studied. No fractionation was done for the Histosol because it is an organic soil.

Apart from increased surface area for reaction, fine textured soils also have higher CEC, which leads to higher retention for cationic species like Cu (Chen et al., 1999). One is also more likely to find higher concentrations of OM in finer textured soils with high CEC than in sandy soils with low CEC. Oftentimes, high OM leads to high CEC, mostly from pH dependent charge. Conditions in fine textured soils are also more conducive to OM accumulation and retention. Organic matter increases retention of both cationic and anionic species. This is achieved through cationic bridging by Al and Fe, leading to anion

retention, and the dissociation of edges of organic complexes in response to changes in pH (leading to retention of both cations and anions, depending on pH [pH dependent charge]). pH dependent charge is the predominant charge in Histosols (USDA, 1996).

The concentrations of ammonium oxalate extractable Fe and Al also varied widely among the soils, with Histosols, Spodosol (A) and Entisol (A and E) having high Fe concentrations (Fig 5). Ammonium oxalate extractable AL was higher in the Bh and Bt horizons, as well as in Entisols (A and E; Fig. 5). Determining total Fe and Al in soil does not give an accurate reflection of their reactivity in the soil because the most reactive parts of Fe and Al are the ammonium acetate extractable fraction (Schwertmann and Taylor, 1989). The high concentrations of reactive Fe and Al in subsurface diagnostic horizons has important implications for both cation and anion retention as shown later.

Retention of As, Cu and Cr

The initial concentrations of As, Cu and Cr are shown in Table 2. These soils generally had very low concentrations of Cu, with the exception of Histosols and Marls. It did not seem like any of the soils were impacted by As, Cr and Cu, hence all of them were used in the retention studies. Sorption and desorption experiments showed wide ranging distribution coefficients (K_d values). The K_d is the ratio of the amount of metal sorbed onto particles to that of the amount still in the solution around the same particles (Anderson and Christensen, 1988). The K_d value is important because, apart from giving an estimate of the partition of an element between the solid and liquid phases, it can be used to calculate the retardation factor using the relationship (the higher the K_d , the higher the retardation factor):

$$R = 1 + K_d * \rho_b / \phi$$

where: R = retardation

K_d = distribution coefficient

ρ_b = soil bulk density

ϕ = soil porosity

The K_d values calculated for a leaching solutions with compositions up to 48 mg/L As and less than 5 mg/L Cu and Cr respectively, ranged from 0.6 – 85, 0.4 – 64, and 0.7 – 111 in columns for As, Cu and Cr, respectively (data not shown). The retention capacity for each element depended on the initial concentrations of the solution that was eluted through the columns, the number of leaching events and the soil type. The K_d values for Cu and Cr were possibly exaggerated by the low concentrations used. Increasing the concentrations of Cu and Cr over a range yielded different K_d values, which decreased with increasing concentration. Different sorption maxima were also reached for different initial solution concentrations.

Although the concentrations of the three elements in CCA-treated wood are generally not very different from each other, As leaches out the most from the wood products, with leachate concentrations at least an order of magnitude higher than those of Cr and Cu. Therefore, this discussion places more emphasis on the behavior and retention of As than Cu and Cr. Because distribution coefficients varied with the initial concentrations of As, Cu and Cr that were added, it was not possible to calculate a unique K_d value that would represent each soil order over a wide range of pH and soil solution As, Cu and Cr concentrations. Batch sorption and desorption studies corroborated these observations, with batch studies consistently yielding lower K_d values than column

studies. This was possibly due to the lower solution to solid ratio (greater partitioning) and the higher solution-solid contact time in column studies (more likely to reach equilibrium at higher sorption rates).

Figure 6 shows the partitioning of As, Cu and Cr between the solid and liquid phase. Although none of the soils tested reached their sorption maxima at the concentration used, the gradients of the curves showed that the Histosols and Marls had the highest sorption potential. The A horizons of Ultisols, Alfisols and Spodosols showed the least potential. Studies are being done with the same soils with solutions with considerably higher concentrations of As, Cu and Cr to determine sorption curves up to their sorption maxima. Figure 7 showed how desorption curves can be used to determine the mechanisms of adsorption and the retention capacity of soils for different metals. The ease with which desorption of an element is achieved speaks to the type of sorption mechanism involved, non-specific (exchangeable) versus specific (non-exchangeable) sorption (Gomes et al., 1997). This is critical because, although As generally leaches out more easily from CCA-treated wood than Cu and Cr, it is not retained by soils more than Cu and Cr (Chirenje et al., 2003; Chen et al., 2002). Copper, which is retained by soils through both specific and non-specific sorption showed very high retention, while Cr, which is retained through mostly specific sorption, demonstrated even higher retention capacity (Fig. 7; Gomes et al., 2001). Berti and Jacobs (1996) showed that elements with high retention are more likely to displace elements (they have higher selectivity) with low retention capacity in soils.

In terms of surface soil behavior, the Marls and Histosols retained As, Cu and Cr the most, followed by Entisols, Alfisols, and Ultisols, and finally by Spodosols, which

retained the least of all three elements. Marls and Histosols have been shown to have higher natural trace element concentrations than the other soil types in Florida (Chen et al., 1999; 2002). There are a multitude of factors that may have led to this distribution pattern, apart from the soil characteristics discussed earlier. For example, previous research on phosphorus (P) has shown that coated sand grains have a higher tendency to retain elements than bare quartz grains (Harris et al. 1987a, b) because the common coating components (metal oxides, aluminosilicates, etc) have high affinity for trace elements, including As, Cu and Cr. Some of the great groups (e.g. albic horizons of alaquods [Spodosols]) investigated here been exposed to extreme weathering, which strips the coatings from the sand grains (Harris et al. 1987a, b). Rhue et al. (1994) showed that some horizons within the same great groups retain the coatings and exhibit high retention while those that did not retain coatings exhibited low retention.

The concentrations of As, Cu and Cr in the subsurface horizons of soils that were not impacted by CCA-treated wood were not significantly greater than those in surface horizons (Table 1). Subsurface horizons tend to serve as a sink of trace elements only if there is a source. Therefore, if the surface horizons had not been exposed to considerably high concentrations of trace elements that would have leached to the subsurface, there is no reason to expect to find high concentrations in the subsurface. Using high concentrations of As, Cu and Cr, subsurface diagnostic horizons showed their potential to retain more metals than surface horizons (Fig. 7). The slow release of trace metals on desorption with KCl showed that these horizons have high As, Cr and Cu retention potential.

Correlation analyses using sorption and desorption curves for Cu and Cr showed that CEC, texture, and OC, played a significant role in sorption and retention (data not shown). The effects of texture, ammonium oxalate extractable Fe and Al, and organic carbon on As retention have been well documented (Chen et al., 1999; Jacobs et al., 1970). However, CEC also displayed a major role in As retention. This may not have been a direct effect. Rather, the increase in CEC may have been due to an increase in clay content and OC both of which lead to increased As retention (Chen et al., 2002).

Since none of the parameters responsible for As, Cu and Cr retention had domineering effects in all soils, our knowledge of specific behaviors of certain soils helps us distinguish the mechanisms involved for each soil type. For example, the Marl used in this study had very low ammonium oxalate extractable Fe but it was high in clay content, calcite and OC which also play a large role in retention of trace elements. The high ammonium oxalate extractable Fe in the Entisol led to increased retention in a soils that would otherwise have low retention. A lot of the E horizons used were obtained from Entisols, which are mostly young soils with little profile development. As discussed earlier, CEC correlated well with the clay fraction in the Marl and with OC in the Histosol. This suggests that the charge associated with the Marl is mostly permanent while that in the Histosol was variable. This mostly impacts elements that are specifically adsorbed, e.g. Cu, which is also highly correlated to the high OC in Histosols. Therefore environmental changes in Histosols are more likely to lead to significant changes in trace element retention than in Marls.

Soil classification

In terms of As retention, the soils were classified into three groups:

- i. Class 1: Soils with the greatest retention and minimal likelihood for leaching,
- ii. Class 2: Soils with moderate retention with potential for leaching, and
- iii. Class 3: Soils with the least retention, greatest risk for leaching

Using surface soil characteristics, two of the soils (Marls and Histosols) met the requirements for Class 1. The Entisol, Alfisol and Ultisol met the requirements for Class 2, and the Spodosol met the requirements for Class 3. These results are in line with our expectations. Spodosols, such as the ones that were sampled for this study, tended to be acidic and highly leached due to their location in areas where pines are prevalent. In some areas, the soils appeared white from a distance, hence the name ‘sugar sands’.

However, when subsurface horizons were included, the Marls and Histosols still fit in Class 1 (there was no profile differentiation in these soils), while the remaining soils met the requirements for Class 2. These results were unexpected because soils with subsurface diagnostic horizons (Bt and Bh) generally show higher retention in these layers. The inclusion of the Entisol in this category was the most unexpected.

In fact, when the diagnostic horizons (Bt and Bh) were compared to the E horizons from the Entisol, the E horizon had significantly lower retention of As than the Bt and Bh. However, when the results were adjusted for the E overlying Bh and Bt horizons, there was no difference between Entisols, Alfisols, Spodosols and Ultisols.

Conclusions

Both sorption and desorption experiments showed that the soil order was not a good indicator of retention of As, Cu and Cr in soils. It was texture, the concentration of

ammonium oxalate extractable Fe and Al, and OC that were reliable indicators of soil As retention. Since most surface soils are similar, except in Histosols, Marls and some Spodosols (acidic), the retention of As, Cu and Cr in the surface layers of these soils is not affected significantly by the soil type. However, As, Cu and Cr retention in the subsurface was considerably affected by the soil type because the presence or absence of subsurface diagnostic horizons manifests itself in different amounts of ammonium oxalate extractable Fe and Al, OC and fine colloids.

Table 1. Percentages of silt + clay content in study soils (except Histosols[†])

	n	Mean	Range
A _{horizon}	18	14.3 ± 15.1	0.54 - 63.1
Marls	5	95.0 ± 2.28	91.5 - 97.8
Spodosols-A	2	13.7 ± 10.7	6.14 - 21.3
Ultisols-A	3	7.33 ± 0.97	6.55 - 8.41
Entisols-A	2	3.85 ± 4.69	0.54 - 7.16
Alfisol-A	1	8.8 ± 0.00	8.80 - 8.80
E _{horizon}	13	15.1 ± 16.8	2.77 - 48.9
Bt	2	7.97 ± 5.49	4.09 - 11.9
Bh	3	11.4 ± 7.76	5.98 - 20.3

[†]Textural analysis not done for histosols because they are organic soils

where:

A_{horizon} represents all A horizons collected for the study

Marls represent the surface layer of this soil type

E_{horizon} represents all E horizons collected for the study

Bh and Bt represent all the spodic and argillic horizons in the study

Table 2. Concentrations of As, Cu and Cr in study soils[†] (mg kg⁻¹)

	n	As	Cr	Cu
A _{horizon}	19	0.41 ± 0.37	4.87 ± 0.84	0.46 ± 0.39
Histosols	5	3.58 ± 0.19	12.0 ± 3.10	7.41 ± 1.79
Marls	5	19.4 ± 4.98	57.8 ± 7.49	6.46 ± 1.20
Spodosols-A	3	0.14 ± 0.00	3.30 ± 0.36	0.19 ± 0.00
Ultisol-A	3	0.14 ± 0.00	4.10 ± 0.23	0.19 ± 0.00
Entisol-A	2	0.49 ± 0.02	5.10 ± 0.44	0.49 ± 0.22
Alfisol-A	1	0.14 ± 0.00	3.73 ± 0.79	0.19 ± 0.00
E _{horizon}	14	0.63 ± 1.25	5.70 ± 1.02	0.19 ± 0.00
Bh	3	0.14 ± 0.00	3.46 ± 1.30	0.19 ± 0.00
Bt	3	1.46 ± 1.90	7.81 ± 2.43	0.19 ± 0.00

[†]These values do not necessarily represent background concentrations of As, Cu and Cr. They are just an indication of concentrations of As, Cr and Cu before the experiment

The method detection limits (MDLs) for As, Cu and Cr were 0.28, 0.38 and 0.52 mg kg⁻¹, therefore values less than these MDLs were replaced by 0.14, 0.19 and 0.26 mg kg⁻¹, for As, Cu and Cr, respectively.

Table 3. Classification of soil orders according to the classes we defined

Soil Class	Surface Soil Characteristics	Subsurface & Surface Soil Characteristics
Class 1	Marls, Histosols	Marls, Histosols
Class 2	Alfisol, Ultisol, Entisol	Entisol, Alfisol, Utisol, Spodosol
Class 3	Spodosol	<i>-None-</i>

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