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Application of Phytoscreening to Three Hazardous Waste Sites in Arizona

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Abstract

The great majority of prior phytoscreening applications have been conducted in humid and temperate environments wherein groundwater is relatively shallow (~1-6 m deep). The objective of this research is to evaluate its use in semi-arid environments for sites with deeper groundwater (>10 m). To that end, phytoscreening is applied to three chlorinated-solvent hazardous-waste sites in Arizona. Contaminant concentrations were quantifiable in tree-tissue samples collected from two of the sites (Nogales, Park-Euclid). Contaminant concentrations were detectable, but not quantifiable, for the third site. Tree-tissue concentrations of tetrachloroethene (PCE) ranged from approximately 400 – 5000 ug/kg wet weight for cottonwood, palo verde, and velvet mesquite at the Nogales site. In addition to standard trunk-core samples, leaf samples were collected to test the effectiveness of a less invasive sampling method. Leaf-sample concentrations were quantifiable, but several times lower than the corresponding core-sample concentrations. Comparison of results obtained for the test sites to those reported in the literature suggest that tree species is a major factor mediating observed results. One constraint faced for the Arizona sites was the relative scarcity of mature trees available for sampling, particularly in areas adjacent to industrial zones. The results of this study illustrate that phytoscreening can be used effectively to characterize the presence of groundwater contamination for semi-arid sites with deeper groundwater.

Graphical abstract

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Keywords

phytoscreening; tree core samples; VOCs; site characterization

Introduction

Phytoscreening is a rapid and relatively low-cost characterization technology that can be used to screen for the presence of contamination in subsurface environments.

Phytoscreening is based on the sampling and analysis of plant tissues (i.e., tree core, branch, leaf) as a surrogate for sampling of soil, soil gas, and groundwater. Several field studies have demonstrated its usefulness as a characterization method, particularly for volatile organic constituents (VOCs) (e.g., Vroblesky et al., 1999, 2004; Struckhoff et al., 2005; Gopalakrishnan et al., 2007, 2011; Larsen et al., 2008; Sorek et al., 2008; Burken et al., 2011; Limmer et al., 2011, 2013, 2014; Wahyudi et al., 2012; Wittlingerova et al., 2013; Algreen et al., 2015; Rein et al., 2015; Yung et al., 2017).

The majority of prior phytoscreening applications have been conducted in humid and temperate climatic environments. Groundwater depths at the application sites were generally less than 3 meters, with a few ranging up to 6-7 m. Conversely, very few studies have been conducted for sites in semi-arid and arid climates (Cox, 2002; Vroblesky et al., 2004; Sorek et al., 2008), where depths to groundwater are often much greater. The results of these few studies are summarized in Table 1. It is observed that concentrations of VOCs in tree-tissue samples were measurable for some sites and not for others. These sites all have greater groundwater depths (8-20 m) and similar ranges of tetrachloroethene (PCE) and/or trichloroethene (TCE) concentrations in groundwater (~100 to a few 1,000s µg/L). The factor or factors causing the disparate results are not readily apparent.

Additional studies are needed to evaluate phytoscreening effectiveness for arid/semi-arid sites with deeper groundwater. The objective of this research is to evaluate the effectiveness of phytoscreening as applied to three hazardous-waste sites in Arizona. The primary contaminants of concern are chlorinated aliphatics at all three sites. Depth to groundwater ranges from 10 to 27 m for the sites. The results obtained from this work will be compared

to the results from the prior studies to evaluate the effectiveness of phytoscreening for sites with deeper groundwater, and to determine the factors influencing said effectiveness.

Materials and Methods

Field Sites

Nogales Grand Avenue Site—The Nogales Grand Avenue study site is located in the U.S.-Mexico border city of Nogales, Arizona. The site is located within the city limits along the Nogales Wash and surrounding Grand Avenue. The shallow subsurface consists primarily of coarser alluvial sediments, with the unit of interest named the Valle Verde. Groundwater depth is approximately 9-10 m below ground surface, with the gradient to the north-northwest. The primary contaminant of concern is PCE, with a contaminant plume spanning 0.5 km². The source of contamination for the site has not been fully delineated, but is suspected to be related to discharge of contaminated wastewater. Hence there is no defined source zone containing trapped immiscible organic liquids. A total of 35 monitoring wells are being used to monitor the concentrations of PCE at the site. As of October 2013, all groundwater concentrations were below the Arizona Aquifer Water Quality Standards detection limit of 5 ug/L for PCE. Annual precipitation is 42 cm for this site.

The site was sampled in October 2013. Mature vegetation was scarce for most parts of the site. A total of 13 trees were sampled in the northwest region of the plume. Sampled trees were cottonwood, palo verde, velvet mesquite, and burrobrush. Two types of tissue were collected for this site. For standard trunk cores, the cottonwood and palo verde trees were sampled at 1.5 m above ground surface, while the velvet mesquite and burrobrush were sampled at ½ the total height of the plant above ground surface. In addition, leaf samples were collected for the cottonwood, velvet mesquite, and palo verde to test a less invasive tissue sampling method. The leaf samples were collected towards the bottom of the canopy, at 1.5-2 m above ground surface.

The sampling locations were selected to be as close as possible to groundwater monitoring wells, which were located approximately 20-50 m from the sampling sites. The mean groundwater concentration of PCE over the three years prior to this study was 2 ug/L. Minimal variation in groundwater concentrations has been observed over the past several years. In addition, concentration gradients are low in this region of the plume. As a result, the reported groundwater concentration should provide a reasonable representation of concentrations influencing the area in which the sampled trees reside.

Park-Euclid Water Quality Assurance Revolving Fund (WQARF) Site—The Park-Euclid site is located in Tucson, Arizona between the streets of 8th, Mountain Avenue, 14th and Park Avenue on the north, east, south, and west respectively. The shallow subsurface consists mainly of fine to coarse-grained alluvial sands and silts with some clay and gravel. A clay aquitard exists approximately 27-30 m below ground surface, and supports a perched or surficial aquifer. In some areas north and northwest of the facility fine-grained sediments, including silts, sandy silts, and clayey sands, exist near the top of the surficial aquifer. The contaminant plume in the surficial aquifer spans 0.15 km². The site was placed on the WQARF registry in 1999, with TCE and PCE being the primary contaminants of concern.

As of 2012, a multi-phase extraction (MPE) system has treated 32,400 liters of water and removed 246 kg of VOCs from the site. Annual precipitation is 32 cm.

The site was sampled in June 2014. Mature vegetation at this site was scarce, and only a single tree in the vicinity of the facility was available for sampling. The sampling location was located approximately 100 m to the northwest of the source area. The sampled tree is a species of Eucalyptus. Cores were collected at 1.5 m above ground surface. Groundwater in the surficial aquifer at the site flows generally to the north, and is at a depth of approximately 26 m below ground surface. Groundwater concentrations are 10 and 70 µg/L for PCE and TCE, respectively, based on data from the nearest groundwater monitoring well (~40 m).

Motorola 52nd Street Superfund Site—The Motorola 52nd Street Superfund site is located in Phoenix, Arizona between the streets of 52nd, Palm Lane, 7th Avenue and Buckeye Roads to the east, north, west and south respectively. It has been on the National Priorities List since 1989, with TCE as the primary contaminant of concern. The regional aquifer resides in a fractured bedrock system overlain by alluvial fan deposits. The alluvium consists of interbedded coarse and fine-grained deposits comprising sands and gravels and silts and clays. Groundwater at the site ranges between 12-30 m below ground surface (bgs), with the gradient towards the west-southwest. The contaminant plume spans an area of 2 km² in OU1. An SVE system was in operation from 1991 to 1993. In 1993 a groundwater treatment system was placed in operation to address the groundwater contaminant plume. Annual precipitation is 27 cm.

The site was sampled in July 2013. The sampling locations for the study were all located within OU1, with a minimum distance of approximately 200 m to the source zone. In total, 121 samples were collected from 29 trees spanning an area of less than 1 km². All sampled trees were cottonwoods, and were sampled at 1.5 m above ground surface. The depth to groundwater is approximately 27 m bgs at the primary sampling location located several hundred meters from the facility. TCE concentrations in groundwater were ~170 µg/L at this latter location, determined from data reported for the nearest monitoring wells, located approximately 150 m away.

Sample Collection and Analysis

Generally, 2-3 core (or leaf) samples were collected from each tree. The core samples of several (5-10) cm in length were collected using a Haglof 3-thread increment borer (4" L, 0.169" (4.3mm) diameter) with a Haglof 12", 3-thread replacement bit. The samples were placed in 22-mL glass vials containing 20 mL of methanol (>99.9% pure) and capped with PTFE/Silicone septa, placed in a cooler, and transported to the laboratory. The methanol extraction method was modified from EPA method 8260B. The quality assurance and quality control measures include the collection of field blanks, trip blanks, and background samples.

Upon receipt in the laboratory, samples were placed on a shaker for 24 hours, after which they were centrifuged for 10 minutes. A 2-mL aliquot was removed from the vial with a 5-mL glass syringe and placed into a 2-mL no-headspace auto-sampler vial. Analysis of TCE

and/or PCE was by gas chromatograph/mass spectrometry (GC/MS/MS). Laboratory QA/QC included sample blanks and internal standards with acceptable recoveries.

A J&W DB-5MS (30 m, 0.25 mm, 0.25 μ m) column was used for separation with 99.999% helium carrier gas at 1.1 ml min⁻¹ constant flow rate. Samples were injected into the GC inlet using a split mode of 10:1 at 250°C. The transfer line temperature was 275°C. The GC temperature program held the oven at 30°C for 4 min and then increased the temperature at a rate of 30°C min⁻¹ up to 75°C followed directly by another temperature increase of 45°C min⁻¹ up to 275°C.

Limits of detection (LOD) and quantitation (LOQ) for the MS were determined by measuring a signal to noise ratio (by root mean squared method) of 3:1 and 10:1, respectively, for the selected ion chromatogram peak for TCE and PCE. Using serial dilution standard samples spanning 0.007 – 30 ppm, a calibration curve (r^2 value of 0.996) was produced by regression. All reported results fell within this range. The quantifiable method detection limit was determined to be ~65 μ g/kg for tree tissue.

Results and Discussion

Study Results

Four tree/brush species were sampled at the Nogales site (velvet mesquite, burrobrush, yellow palo verde, and cottonwood). The results are presented in Table 1. PCE was detected at different levels for the different tree species, with core-sample concentrations of 385, 588, 1195, and 5320 μ g/kg obtained for burrobrush, cottonwood, palo verde, and velvet mesquite, respectively. Recall that the long-term average groundwater concentration for the site is ~2 μ g/L. These results demonstrate that quantitation of PCE in tree-tissue samples is possible for a site with very low associated groundwater concentrations and relatively deep groundwater (~9-10 m).

The corresponding leaf-sample concentrations are 80, 185, and 246 μ g/kg for cottonwood, palo verde, and velvet mesquite, respectively (Table 2). The leaf concentrations are approximately 10, 6, and 20 times lower than the core concentrations for the three species, respectively (Table 1). These results demonstrate that leaf sampling can provide an alternate, less intrusive sampling method for phytoscreening applications. However, it appears to be less sensitive than standard trunk core sampling. Similar results were reported for branch and leaf sampling versus core sampling in two prior studies (Vroblesky et al., 2004; Gopalakrishnan et al., 2007).

The mean PCE and TCE tree-tissue concentrations at Park-Euclid were 2790 μ g/kg and 22760 μ g/kg, respectively. The tree-tissue concentration for TCE is ~8 times greater than the concentration for PCE. This is consistent with the ~7 times greater concentration of TCE in groundwater (70 vs 10 μ g/L).

TCE was detected in the cores sampled for the third site, Motorola 52nd St, but at levels below quantitation (< ~65 μ g/kg). Depth to groundwater at the Motorola 52nd St. site is 27 m bgs, similar to that for the Park-Euclid site. In addition, the groundwater TCE

concentrations for the Motorola 52nd St. and Park-Euclid sites are similar, 170 µg/L and 70 µg/L, respectively. Cottonwood trees were sampled at the Motorola 52nd St. site, whereas Eucalyptus trees were sampled at the Park-Euclid site. The average rooting depths are approximately 2-4 m for both cottonwoods and eucalyptus (Jackson et al., 1987; Laclau et al., 2001). The samples were collected at similar times of the year for both sites.

As noted above, the groundwater contaminant concentrations, depth to groundwater, average rooting depths, and sample collection periods are very similar for the Park-Euclid and Motorola 52nd St. sites. Hence, these factors can be eliminated as the source of the observed difference in tree-tissue concentrations (quantifiable for Park-Euclid and not quantifiable for Motorola 52nd St.). Two primary factors remain as possible discriminants. One critical factor may be the plant species and their associated uptake mechanisms, cottonwood versus eucalyptus. The potential significance of this factor will be further explored in the following section. Another set of factors relates to potential differences in subsurface properties and how they influence contaminant transport at the two sites.

It is important to note that for all three test sites, the sampling locations were located away from known surface or subsurface source zones. Thus, the source of contaminant for tree uptake is presumed to be the underlying groundwater. Both the Park-Euclid and Motorola 52nd St. sites have heterogeneous subsurface environments. In addition, it is unlikely that tree roots directly tap groundwater for these sites (or for the Nogales site) given their respective depths to groundwater. Horton et al. (2001) investigated the impact of groundwater depth on the physiological condition of two native Southwest US tree species (cottonwood and willow) and concluded that tree health rapidly declines after depth to groundwater exceeds a specified threshold in the range of 3-4 m. It is hypothesized that vapor-phase transport of the VOCs from groundwater, through the vadose zone, to the root zone provided the route of exposure for tree uptake. This transport route was also hypothesized for a prior study, for which the depth to groundwater was approximately 7 m (Struckhoff et al., 2005).

Comparison to Prior Studies

Sorek et al. (2008) presented the results of a phytoscreening study conducted for several sites in an arid environment (Tel Aviv, Israel). Eucalyptus trees were sampled for one site where depths to groundwater ranged from 18-20 m. They reported tree-tissue PCE concentrations ranging up to 10,000 µg/kg. Groundwater PCE concentrations of 117 µg/L were measured for samples collected from a well located 50 m upgradient. Eucalyptus trees were also sampled for a second site, with depth to groundwater of 12.5 m. Maximum tree-tissue PCE concentrations of 4000 µg/kg were observed, with reported groundwater concentration of 4700 µg/L (obtained from a well located ~50 m away). The range of tree-tissue concentrations are quite similar to those obtained for the Park-Euclid site, for which the sampled tree species (eucalyptus) is the same. Conversely, Sorek et al. reported orders-of-magnitude lower TCE concentrations for samples collected from rosewood trees located at a third site, where groundwater VOC concentrations and depths were similar to the prior two sites (Table 1).

Significantly lower tree-tissue TCE concentrations ($\sim 5 \mu\text{g/kg}$) were reported for a semi-arid site in Colorado (Air Force Plant PJKS) wherein depth to groundwater was 8 m (Vroblesky et al., 2004). This result represents samples collected from a single cottonwood tree located upgradient from the primary sampling location for which groundwater depths were much shallower. The TCE concentration in groundwater near the sampled tree was $200 \mu\text{g/L}$. This is very similar to the TCE groundwater concentration reported for the Motorola 52nd St site ($170 \mu\text{g/L}$). Recall that TCE was detected but not quantifiable for cottonwoods sampled for this site. The results for the PJKS and the Motorola 52nd St sites are consistent, with low tree-tissue concentrations reported for cottonwoods located above relatively deep groundwater.

The results reported for a phytoscreening project conducted at a military base in Washington, at which depth to groundwater is approximately 8-10 m (Cox, 2002), are also consistent with those of the Motorola 52nd St and PJKS sites. TCE was not detected in tree core samples collected from several trees overlying a groundwater contaminant plume with maximum concentrations of $\sim 90 \mu\text{g/L}$. The tree species sampled for this study were oak and fir.

Comparison of the results obtained from all of the studies cited above, as well as those from this study, are presented in Table 1 and Figure 1. For the Motorola 52nd St, PJKS, Tel Aviv (site 3), and Washington sites, very low to non-detectable tree-tissue concentrations were obtained for cottonwood, rosewood, oak, and fir. Conversely, much higher tree-tissue concentrations were obtained for eucalyptus trees at the Park-Euclid and Tel Aviv sites. The tree-tissue concentrations for eucalyptus are approximately two to three log units higher than those for the other tree species, for a given associated groundwater concentration. It is notable that significantly higher TCE concentrations were measured for cottonwood samples collected from trees located in areas of the PJKS site with much shallower groundwater ($< 1 \text{ m}$). Specifically, concentrations up to $> 100 \mu\text{g/kg}$ were obtained, compared to $\sim 5 \mu\text{g/kg}$ for the tree at the deeper-groundwater site. This indicates that cottonwoods are capable of greater VOC uptake at the site when roots are directly tapping groundwater. However, inspection of Figure 1 shows that these higher concentrations are still much lower than those obtained for the eucalyptus. These results indicate that tree species is a significant discriminant for the results obtained for these sites.

Higher tree-tissue concentrations of PCE were observed for cottonwood trees, however, for the Nogales site. This is in contrast to the low concentrations obtained for cottonwoods for the PJKS and Motorola 52nd St sites. The subsurface at the Nogales site consists primarily of alluvium with minimal lower-permeability deposits within the profile, unlike the other two test sites. This illustrates that subsurface conditions also likely influence uptake for the compared sites, as would be expected.

Conclusion

The objective of this research was to evaluate the use of phytoscreening in semi-arid environments for sites with deeper groundwater. Contaminant concentrations were detected in tree-tissue samples collected for all three sites. This includes one site for which associated

groundwater concentrations are very low. In addition to standard trunk core samples, leaf samples were collected for one site to test the effectiveness of a less invasive sampling method. Concentrations of PCE in leaf samples were quantifiable, but were several times lower than the corresponding core-sample concentrations.

Comparison of the few phytoscreening studies reported in the literature for sites with deeper groundwater showed conflicting results, with some sites showing measureable tree-tissue concentrations and other sites not showing measurable concentrations for similar site conditions. Analysis of the integrated results of the present study and the literature data indicates that tree species is a major factor mediating the magnitude of tree-tissue concentrations observed. Specifically, tree-tissue concentrations for eucalyptus were approximately two to three log units higher than those for the other tree types, for a given associated groundwater concentration.

One constraint faced for the Arizona sites was the relative scarcity of mature trees available for sampling, particularly in areas adjacent to industrial zones. This issue may limit the ability to adequately sample large areas at high resolution for some sites. Overall, the results of this study illustrate that phytoscreening can be used effectively to characterize the presence of groundwater contamination for semi-arid sites with deeper groundwater.

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Highlights

Phytoscreening is tested for three arid sites with deep groundwater

VOCs were detected in tree tissue samples for all three sites

The levels of VOC in tree tissue corresponded to groundwater concentrations

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Figure 1.

Comparison of phytoscreening data for sites with deeper groundwater- impact of tree species. “Other” represents cottonwood, rosewood, oak, and fir. “Cottonwood-shallow” represents data collected from trees in areas with groundwater depth <1 m. Data sources reported in Table 1.

Table 1
Information for phytoscreening applications conducted at sites with deeper groundwater

Site	GW Depth (m)	Tree Species	Number of Trees Sampled	GW Conc (µg/L)	Tree Core Conc (µg/kg)
Tel Aviv site 1 ^a	18-20	Eucalyptus	~3	117 PCE	10,000 PCE
Tel Aviv site 2 ^a	12.5	Eucalyptus	~3	4,700 PCE	4,000 PCE
Tel Aviv site 3 ^a	19	Rosewood	~3	2,620 TCE	56-150 TCE
PIKS site 1 ^b	8	Cottonwood	1	200 TCE	5
PIKS site 2 ^b	<1	Cottonwood	4	39-190 TCE	13-107
WA site ^c	8-10	Oak, Fir	several	90 TCE	Non-detect
Nogales site ^d	9-10	Burrobrush	1	2 PCE	385
		Cottonwood	4		588
		Palo Verde	2		1,195
		Velvet Mesquite	2		5,320
Park-Euclid site ^d	26	Eucalyptus	1	70 TCE	22,760
				10 PCE	2,790
Motorola ^d	27	Cottonwood	29	170 TCE	<65

^aSorek et al., 2008
^bVroblecky et al., 2004
^cCox, 2002
^dThis study

Table 2
Comparison of PCE concentrations ($\mu\text{g/kg}$) as a function of sample type for the Nogales site (samples collected from the same tree)

Sample type	cottonwood	palo verde	velvet mesquite
Core	821	1195	5320
Leaf	80	185	246
Ratio	~10	~6	~20