Treatment of simulated oil and gas production wastewater using *Typha latifolia* in a pilot-scale constructed wetland

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ABSTRACT

During hydraulic fracturing (‘fracking’), large volumes of high-pressure, chemically-treated water are pumped into subsurface strata to free trapped petroleum and natural gas. Chemically-enriched water, along with brine and groundwater, collectively termed oil and gas production water (PW), are eventually recovered from the well. PW poses environmental and health risks; however, it can be reused if potentially hazardous constituents are removed. A two-stage pilot-scale constructed wetland containing cattail (*Typha latifolia*) was tested for treatment of synthetic PW. After 49 days, PW pH increased from 4.2 to 7.0, and electrical conductivity decreased from 22,100 to 3,300 µS·cm⁻¹. *Typha* shoots had bioconcentration factors for Pb ranging from 2.8 (Stage 1 of constructed wetland) to 8.0 (Stage 2). Transfer factors for Pb were 0.67 (Stage 1) and 1.37 (Stage 2). These results indicate that *Typha* may be effective for Pb removal from PWs. The present study may be of practical value to oil and gas production companies that plan to recycle or properly dispose of large quantities of oil and gas production wastewater.

INTRODUCTION

Wastewater produced by hydraulic fracturing

Hydraulic fracturing (‘fracking’) is defined as the process of creating and propagating a fracture within a rock layer by means of applying hydraulic pressure in order to release petroleum, natural gas, coal seam gas, or other potential fuels for extraction (Earthworks 2015). The injection of a highly pressurized fluid creates new channels in the strata which increase extraction rates and recovery of fossil fuels (Veil et al. 2004). The injected fluid is typically a slurry of water, proppants, and various chemical additives.

Following initial injection into the well, a portion of the injected water returns to the surface immediately and is termed ‘flowback.’ The remaining fluids either permeate the formation or return to the surface over the life of the active well and are termed ‘produced water’ (Earthworks 2015). Both types of wastewater may contain hydraulic fracturing fluids, naturally occurring brines, heavy metals, and other compounds from the formation (Fontenot et al. 2013; Kassotis et al. 2015; Warner et al. 2012). The United States generates an estimated 21 billion barrels of produced water annually (Aquatec 2015). In this report, oil and gas flowback water, produced water, and hydraulic fracturing fluids collectively will be termed oil and gas production wastewater (PW).

A number of papers have described the effects of hydraulic fracturing on surface water quality, groundwater quality, and soil properties (Fontenot et al. 2013; Pichtel 2016; Warner et al. 2012). PW may pose a threat to public
health and the environment as some constituents are known to be acutely toxic, some are carcinogenic, and others are believed to be endocrine disruptors (Bergman et al. 2013; Kassotis et al. 2015). The composition of PW varies markedly and its chemical and physical properties depend on the geographic location of the field, the geologic formations in contact with the water over time, and the treatment chemicals and extraction techniques that are used (Benko and Drewes 2008; Murray Gulde et al. 2003; Veil et al. 2004). However, three common fractions of PWs are salts, metals/metalloids and hydrocarbons (O’Rourke and Connolly 2003; Veil et al. 2004).

PW management falls under two broad categories: underground injection and surface management. Selection of a management option for PW is based on numerous considerations, including the composition and volume of the PW, the technical feasibility of a treatment option, and the options allowed by regulations (NPC 2011). Current PW management strategies include minimization of the volumes generated, utilization for enhanced recovery and pressure maintenance, disposal into underground formations, surface discharge, and beneficial use at the surface (Khatib and Verbeek 2003; Veil et al. 2004).

In the United States, more than 98% of produced water from onshore wells is injected annually; approximately 59% is injected into producing formations to maintain pressure, and 40% into non-producing formations for disposal. The remaining produced water is managed in evaporation ponds, shipped offsite for commercial disposal, or applied for beneficial purposes, for example, irrigation, land application, and livestock and wildlife watering (Clark and Veil 2009; Pichtel 2016).

The need for alternative sources of water resources and increasingly stringent water quality discharge standards have stimulated the drive for increased water reuse (USEPA 2004). Integrating water reuse into water management strategies can lessen the demand on existing water resources and partially alleviate the need for development of new water sources (Veil et al. 2004).

**Constructed wetlands for treatment of PWs**

A range of technologies, both chemical and physical, are available for treatment of contaminants from PW. These technologies vary in terms of complexity of operation, portability, and cost.

Constructed wetlands (CWs) are engineered systems designed to improve the quality of point and nonpoint sources of water pollution including storm water runoff, domestic wastewater, agricultural wastewater, and coal mine drainage. Constructed wetlands are also used to treat petroleum refinery wastes, compost and landfill leachates, and pretreated industrial wastewaters, such as those from paper mills and textile mills (USDA-NRCS 2015). Constructed wetlands are classified into two categories according to mode of water flow and dominant aquatic plant species (Kadlec et al. 2010):

1. Free water surface CWs consist of basins with a layer of soil or sediment to support the growth of rooted macrophytes. The wastewater is directed horizontally through a dense mat of plants. Water flow is above the ground surface.
2. Subsurface flow CWs consist of basins filled with a granular medium such as gravel, sand, or soil. Water flow is below the ground surface. A drainage tube at the base of the outlet area allows for effluent removal.

**Typha latifolia**

Some aquatic plants have been documented as having the ability to transfer nutrients (Chung et al. 2008; Gottschall et al. 2007; Moshiri 1993; Mungur et al. 1997) and heavy metals (Deng et al. 2004; Fritioff and Greger 2006; Maine et al. 2006; Miretzky et al. 2004; Skinner et al. 2007) from aquatic environments. Researchers have addressed the phytoremediation of aquatic environments and contaminated sediment by aquatic macrophytes (Osmolovskaya and Kurilenko 2001; Panich-Pat et al. 2004). *Typha latifolia* L. (cattail) is a wetland plant that grows extensively in temperate regions (Ye et al. 1997). *T. latifolia* is widely used in CW systems due to its high yield, non-invasive characteristics and resilience (Brisson and Chazarenc 2009). *T. latifolia* additionally has a high capacity for tolerating and taking up heavy metals without serious physiological damage (McNaughton et al. 1974; Pip and Stepaniuk 1992).

The objective of the reported research was to determine the effectiveness of pilot-scale constructed wetlands (CWs) for treating PWs for possible reuse. The specific objectives were as follows: (1) to evaluate the ability of *T. latifolia* to ameliorate pH and electrical conductivity, and to sorb heavy metals from PW; and (2) to assess the ability of *T. latifolia* to take up and accumulate Na, Cu, and Pb for phytoextraction purposes.

**EXPERIMENTAL METHODS**

**Synthetic PW**

Synthetic PW was prepared using reagent grade chemicals (Sigma) mixed with deionized water. The chemical composition was based on data from Maguire-Boyle and Barron (2014), FracFocus (2015), and Marcellus (2010). Salts included AlCl₃, AlF₃, Al(NO₃)₃, CuSO₄, MgCO₃, Mg(NO₃)₂, K-acetate, KCl, Na-acetate, Na₂CO₃, and NaCl. Hydrocarbons included diesel fuel, ethanol, ethylene glycol, glycerol, hexane, 2-propanol, and toluene. For pH adjustment, NaOH, acetic acid, H₃BO₃, HCl, and H₃PO₄ were used.

The pH of the PW was determined with a glass electrode pH meter (Accumet® model AP115), and its electrical conductivity with an EC meter (Hanna Instruments model HI 993310). Total Ca, Mg, Cu, Na, and Pb concentrations were determined via flame atomic absorption spectrophotometry (FAAS) (Perkin Elmer AAnalyst 200), and total K via flame atomic emission spectrophotometry (APHA 2017).
**Constructed wetland study**

Bench-scale constructed wetlands (Free-Water-Surface mode) were established in a greenhouse. The system included 20-liter plastic trays measuring 90cm in length, 40cm in width and 15cm in height. Each tray was filled with 10kg of Glyndon soil (fine, illitic mesic Aquic Haplustalf). Soil was obtained from the surface 20cm of agricultural fields in central Indiana, air-dried, and sieved to pass a 2-mm mesh sieve.

There were three variants, the treatment and two controls, and three replicates of each variant. Each variant consisted of two trays (‘Stage 1’ and ‘Stage 2’) in series (Figure 1). In the treatment group and Control 1, the trays were planted with 15 rhizomes of cattail (*T. latifolia*) in each tray; no rhizomes were planted in Control 2.

The rhizomes were collected from a natural wetland located in central Indiana. The plants were treated with tap water for 63 days (1000mL per week) and allowed to acclimate to greenhouse conditions. Then the treatment group and Control 2 received 1000mL of PW weekly for 49 days, while Control 1 received tap water only. The PW was added by gravity from carboys, and all trays were maintained in a saturated condition.

**Characterization of soil properties**

Soil pH was determined using a glass electrode pH meter (Accumet® AP115) with a 1:10 mixture of soil:deionized H₂O. Electrical conductivity (EC) was measured using an EC meter (Hanna Instruments HI 993310). Total organic carbon was determined via the Walkley-Black method (Walkley and Black 1934), and total N by the Kjeldahl method (Black 1965). Soil P was measured by the Bray-II method (Bray and Kurtz 1945), and extractable K via flame atomic emission spectrophotometry (Perkin Elmer AANalyst 200) after NH₄OAC extraction (Helmke and Sparks 1996). Extractable metal (Na, Cu, Pb) concentrations were measured after DTPA extraction; samples were extracted with 0.05M diethylene triamine pentaacetic acid (DTPA) for 2h on an oscillating shaker (120osc·min⁻¹) (Bureau 1982). The mixtures were filtered through Whatman no. 2 filter paper and analyzed using FAAS. Soil particle size distribution was determined by the hydrometer method (Allen et al. 1974).

**Characterization of constructed wetland (CW) effluent and Typha latifolia**

After 49 days of treatment, liquids exiting Stage 2 of each variant were collected for analysis. Effluents were filtered using Whatman no. 42 filter paper to remove sediment before determination of pH, EC, and concentrations of Ca, Mg, Cu, Na, and Pb, as described for the raw PW.

Entire plants were removed from the trays and separated into shoots and roots using an Exacto® knife. Plant tissue was oven-dried at 80°C for 4 days and its dry weight was recorded. Dried plant tissue was cut into small pieces with stainless steel scissors. One-half gram (d.w.) of plant tissue was transferred to an acid digestion vessel. Concentrated (70%) HNO₃ was added and the mixture digested using a MARS microwave digestion apparatus (CEMCorporation). Total metal (Na, Cu, Pb) concentrations of the digests were determined using FAAS.

**Metal uptake by Typha**

Several factors were calculated to determine metal uptake and translocation by *Typha*. The bioconcentration factor (BCF) is defined as the ratio of the metal concentration in a plant part to the extractable metal concentration in the rhizosphere soil (Rezvani and Zaefarian 2011):

\[
\text{BCF}_{\text{shoot}} = \frac{C_{\text{shoot}}}{C_{\text{soil}}} \\
\text{BCF}_{\text{root}} = \frac{C_{\text{root}}}{C_{\text{soil}}}
\]
where \( C_{\text{shoot}} \) and \( C_{\text{root}} \) are the metal concentrations in the shoot and root, respectively, and \( C_{\text{soil}} \) is the metal concentration in the soil.

The translocation factor (TF) was calculated by dividing the metal concentration in the shoot by the metal concentration in the root:

\[
TF_{\text{shoot}} = \frac{C_{\text{shoot}}}{C_{\text{root}}}
\]

where \( C_{\text{shoot}} \) and \( C_{\text{root}} \) are the metal concentrations in shoot and root, respectively.

RESULTS AND DISCUSSION

Properties of PW

The synthetic PW had a pH of 4.2 and an EC of 22,100 µS·cm\(^{-1}\) (Table 1). Concentrations of Na, Cu and Pb were 4.7, 4.4, and 4.1 mg·L\(^{-1}\), respectively. Iggunu and Chen (2014) measured a pH of 4.3 in oil field produced water. In contrast, ALL (2009) noted a pH of 8.1. Iggunu and Chen (2014) measured an EC of 4,200 µS·cm\(^{-1}\) in produced water and Na concentrations ranging from 132 to 97,000 mg·L\(^{-1}\). The same authors found Cu concentrations ranging from <0.02 to 1.5 mg·L\(^{-1}\), and Pb concentrations from 0.002 to 8.8 mg·L\(^{-1}\). ALL (2009) measured Na levels of 486 mg·L\(^{-1}\). Such variations in pH and in other analyte concentrations in PWs are common, due to the wide range of formulations for preparing hydraulic fracturing fluids as well as the varying geochemistry of subsurface water.

Soil properties

The Glynwood soil had a pH of 6.7 (Table 2). Total C and N contents were 3.9 and 0.36%, respectively. Levels of extractable Na, Cu and Pb were consistent with those of non-contaminated soil. The soil texture was silt loam.

Table 1. Chemical characteristics of synthetic oil and gas production water (PW).

| Characteristic | Value |
|---------------|-------|
| pH            | 4.2   |
| EC (µS·cm\(^{-1}\)) | 22,100 |
| Metals (mg·L\(^{-1}\)) |       |
| Mg            | 4.4   |
| Ca            | 7.1   |
| K             | 4.8   |
| Na            | 4.7   |
| Cu            | 4.4   |
| Pb            | 4.1   |

Table 2. Chemical and physical characteristics of soil used in the constructed wetland.

| Characteristic | Value |
|---------------|-------|
| pH            | 6.7   |
| EC (µS·cm\(^{-1}\)) | 1,100 |
| TOC* %        | 3.9   |
| Total N %     | 0.36  |
| Bray-II P (mg·kg\(^{-1}\)) | 13.0 |
| K (mg·kg\(^{-1}\)) | 86.9 |
| Extractable metals (mg·kg\(^{-1}\)) |       |
| Na            | 0.31  |
| Cu            | 0.22  |
| Pb            | 0.50  |
| Texture (%)   |       |
| Sand          | 28.0  |
| Silt          | 51.0  |
| Clay          | 21.0  |

*TOC, total organic carbon
Properties of PW after CW treatment

After CW treatment, the effluent pH was 7.0 (Table 3); the effluent pH of Control 2 (no *Typha*) was 6.7. Glynwood soil is formed upon dolomitic limestone deposits (USDA-NRCS 2017) and imparts substantial acid buffering capacity. Salinity decreased markedly after CW treatment (Table 3). The values of EC were 2,900µS·cm⁻¹ after Stage 1 and 3,300µS·cm⁻¹ after Stage 2, constituting an 87 and 85% decrease, respectively, from that of the raw PW (Table 1). The EC of effluent from Control 2 was 18,600µS·cm⁻¹, which demonstrates that *Typha* was primarily responsible for reducing salinity. Cattail has been found capable of tolerating and treating saline wastewater (Coon et al. 2000). In a study of CW treatment of high-salinity wastewater, Jesus et al. (2014) measured salinity reductions of 52%.

Sodium concentrations after all CW treatments ranged between 2.7-4.3mg·L⁻¹, which is only a modest decline from that of the fresh PW (4.7mg·L⁻¹, Table 1). Copper concentrations in the effluent from Stages 1 and 2 ranged from 0.03-0.04mg·L⁻¹ (Table 3), which was a 99% reduction from the Cu concentration in raw PW (4.4mg·L⁻¹, Table 1). Similarly, Lesage (2006) and Gersberg et al. (1984) reported >90% copper removal in PW CWs. Using Table 1). Similarly, Lesage (2006) and Gersberg et al. (1984) reported similar results.

Total Pb concentrations after treatment was 0.69 (Stage 1) and 0.76mg·L⁻¹ (Stage 2). These values constitute an 83 and 82% decrease from the Pb concentration in the raw PW (4.1mg·L⁻¹). The observed Pb concentrations were slightly lower than those in Control 2 (0.88mg·L⁻¹). Khan et al. (2009) noted a 50% Pb removal rate with *Typha* in a CW. Arroyo et al. (2010) found a 48% reduction in Pb concentration. Lesage (2006) used a CW to treat domestic wastewater and reported 48.3% and 81% removal of Cu and Pb, respectively. Walker and Hurl (2002) monitored wetlands treating stormwater for metals over a four-month period. They found Cu and Pb levels were reduced by 48 and 71%, respectively.

Soil properties after CW treatment

Soil pH was 6.97 and 7.06 in Stages 1 and 2, respectively (Table 4). This compares with a pH of 4.18 in Control 2 (no *Typha*). CW Stages 1 and 2 had markedly lower EC values (3,000 and 3,400µS·cm⁻¹, respectively) than Control 2 (22,100µS·cm⁻¹) (Table 4). These results demonstrate the effectiveness of *Typha* in removing salts from the soil solution. Pantip and Suwanchai (2004) found that *Typha* had the ability to grow well in saline wastewater.

Soil Na concentrations were 2.9 and 43.0mg·kg⁻¹ (Stage 1 and Stage 2, respectively) (Table 4), and 4.3mg·kg⁻¹ in Control 2. These values for Na are considered relatively low and will not cause detrimental effects to *T. latifolia*. Coon et al. (2000) found that *T. latifolia* produced substantial biomass at Na concentrations of 12,000mg·kg⁻¹ in sediment.

Soil Cu concentrations measured 5.1 and 1.0mg·kg⁻¹ in Stages 1 and 2, respectively (Table 4), and 3.2mg·kg⁻¹ in Control 2. The Glynwood soil in this experiment contained 0.2mg·kg⁻¹ Cu (Table 2). This soil contained 3.9% TOC (Table 2), which gives it a moderate sorption capacity for metals. Copper forms strong bonds with organic matter (Zhou and Wong 2001); humic acids and other organic molecules interact readily with Cu (Klucakova 2012; Otero et al. 2015).

Soil Pb concentrations measured 8.8 and 2.9mg·kg⁻¹ (Stages 1 and 2, respectively), and 4.2mg·kg⁻¹ in Control 2 (no *Typha*). The non-contaminated Glynwood soil contained 0.5mg·kg⁻¹ Pb (Table 2). The results for Stages 1 and 2 are due in part to Pb uptake by *Typha*. It is also likely that Pb precipitated from solution and/or was sorbed in the *Typha* rhizosphere in Stages 1 and 2. Wetland sediments are generally considered a sink for metals (Arroyo et al. 2010; Burton and Scott 1992) and may contain high concentrations of metals in a reduced state (Weis and Weis 2004). The major processes responsible for metal removal in wetlands include adsorption to sediments and soils, precipitation as insoluble salts, and uptake by plants and microorganisms (Kadlec et al. 2010). Plant activities influence and modify the distribution of trace metals between the solid and aqueous phases (Kim et al. 2008).

| Table 3. Chemical characteristics of effluents from the constructed wetland after 49 days (n=3, mean±standard deviation). |
|---|---|---|---|---|---|---|---|
| Treatment | pH | EC (µS·cm⁻¹) | Mg (mg·L⁻¹) | K (mg·L⁻¹) | Na (mg·L⁻¹) | Cu (mg·L⁻¹) | Pb (mg·L⁻¹) |
| Stage 1 | 7.02±0.14 | 2,900±427 | 3.07±0.36 | 1.00±0.37 | 2.66±0.60 | 0.03±0.01 | 0.69±0.26 |
| Stage 2 | 7.05±0.19 | 3,300±279 | 2.95±0.14 | 1.31±1.01 | 3.10±1.10 | 0.04±0.02 | 0.76±0.23 |
| Control 1 | 8.20±0.06 | 840±123 | 2.28±0.15 | 4.87±0.01 | 4.27±0.02 | 0.20±0.21 | BDL* |
| Control 2 | 6.69±0.10 | 18,600±1,009 | 4.57±0.17 | 2.13±1.08 | 3.26±0.77 | 0.02±0.01 | 0.88±0.01 |

Control 1, treated with tap water, with cattails
Control 2, treated with PW, no cattails
BDL*, below detectable limits
Bioma ss production of Typha latifolia Shoot production by Typha was substantially greater in the CW stages treating PW than in Control 1 (Table 5). It is possible that, although toxic elements (such as Pb) and excess salinity were present in the PW, the high concentrations of Ca, Mg, and K not only compensated for the presence of these substances but even increased plant growth. Vegetative growth of Typha is increased by addition of nutrients, and Typha can quickly form monotypic stands in fertile wetland systems (Svengsouk and Mitsch 2000; Woo et al. 2002).

Root biomass production in Stage 1 of the CW (2.5g) was markedly lower than that in Stage 2 and Control 1 (7.2 and 6.6g, respectively) (Table 5). It is possible that Typha root was negatively affected by excess EC, and by high Na and Pb concentrations, as this was the first stage in which plants contacted the PW. Many plants will form Pb-phosphate and other Pb complexes in roots to prevent Pb uptake; however, root growth may be impaired (Blaylock and Huang 2000; Munzuroglu and Geckil 2002).

Metal uptake and accumulation by Typha Sodium content of Typha shoots was 15.2 and 94.3mg·kg⁻¹ in Stages 1 and 2 of CW treatment, respectively (Table 6). This compares with 35.8mg·kg⁻¹ in Control 1. Most freshwater wetland macrophytes have low Na requirements, with concentrations in above-ground biomass below 2,000mg·kg⁻¹ (Boyd 1978).

Typha shoots contained 3.7 and 2.9mg·kg⁻¹ Cu in Stages 1 and 2, respectively (Table 6). This compares with 0.9mg·kg⁻¹ Cu in Control 1. Copper uptake by Typha roots was somewhat greater than that by shoots: the roots contained 5.8 and 3.6mg·kg⁻¹ in Stages 1 and 2, respectively. Typha roots in the Control contained 1.1mg·kg⁻¹.

Table 4. Chemical characteristics of soil in constructed wetlands after 49 days (n=3, mean±standard deviation).

| Treatment | pH       | EC (mg·L⁻¹) | K (mg·L⁻¹) | Na (mg·L⁻¹) | Cu (mg·L⁻¹) | Pb (mg·L⁻¹) |
|-----------|----------|-------------|------------|-------------|-------------|-------------|
| Stage 1   | 6.97±0.12| 2,998±475   | 0.64±0.01  | 2.91±1.19   | 5.12±1.25   | 8.83±0.90   |
| Stage 2   | 7.06±0.02| 3,481±728   | 0.38±0.05  | 43.0±4.5    | 1.03±0.28   | 2.88±0.94   |
| Control 2 | 4.18±0.10| 22,150±1,897| 4.87±0.01  | 4.27±0.02   | 3.20±0.21   | 4.24±0.26   |

Control 2, treated with PW, no cattails

Table 5. Total dry mass production (g) of Typha latifolia in the constructed wetlands (n=3, mean±standard deviation).

| Treatment | Shoots | Roots |
|-----------|--------|-------|
| Stage 1   | 18.30±0.79 | 2.50±0.48 |
| Stage 2   | 18.90±0.63 | 7.20±0.95 |
| Control 1 | 10.70±1.4  | 6.60±0.72 |

Control 1, tap water, with cattails

Table 6. Metal content (mg·kg⁻¹) in Typha latifolia shoots and roots in constructed wetlands after 49 days (n=3, mean±standard deviation).

|          | K         | Na         | Cu         | Pb         |
|----------|-----------|------------|------------|------------|
| Shoots   |           |            |            |            |
| Stage 1  | 25.64±0.76| 15.16±3.70 | 3.68±0.90  | 24.74±6.21 |
| Stage 2  | 142.36±35.80 | 94.32±35.30 | 2.92±0.40  | 22.92±7.07 |
| Control 1| 105.54±4.76| 35.78±1.40 | 0.90±0.20  | 1.45±0.20  |
| Roots    |           |            |            |            |
| Stage 1  | 48.24±0.04| 114.48±55.4| 5.84±1.10  | 36.82±2.27 |
| Stage 2  | 91.16±0.26| 51.62±16.80| 3.64±0.80  | 16.70±0.10 |
| Control 1| 40.86±0.26| 16.74±2.10 | 1.1±0.50   | 2.12±0.10  |

Control 1, tap water, with cattails
Table 7. Bioconcentration Factors (BCF) and Transfer Factors (TF) for Na, Cu and Pb in *Typha latifolia*.

|                | Na      | Cu      | Pb      |
|----------------|---------|---------|---------|
| **Bioconcentration Factors** |         |         |         |
| **Shoots**     |         |         |         |
| Stage 1        | 5.40    | 0.71    | 2.80    |
| Stage 2        | 2.20    | 2.80    | 8.00    |
| **Roots**      |         |         |         |
| Stage 1        | 40.90   | 1.10    | 4.19    |
| Stage 2        | 1.20    | 3.50    | 5.82    |
| **Transfer Factors** |         |         |         |
| Stage 1        | 0.13    | 0.63    | 0.67    |
| Stage 2        | 1.83    | 0.80    | 1.37    |
| Control 1      | 2.14    | 0.77    | 0.81    |

Control 1, tap water, with cattails

In Stage 1, shoots of *Typha* accumulated almost as much Pb as did roots (24.7 versus 36.8 mg·kg⁻¹, respectively) (Table 6). These concentrations of Pb were higher than concentrations considered phytotoxic (<5 mg·kg⁻¹) by Markert (1992). Kabata-Pendias (2010) reported that the Pb content of plants varies between 0.05 and 3.0 mg·kg⁻¹; however, the amount in roots varied widely (25-3628 mg·kg⁻¹). Carranza-Alvarez et al. (2008) reported Pb concentrations to range from 10 to 25 mg·kg⁻¹, with maximum Pb accumulation in roots. Dunabin and Bowmer (2002) reported that metal uptake by *Typha* was highest in roots, and that leaves had the lowest metal concentrations.

*Typha* tolerates increased levels of metals in tissue without serious physiological damage (Sasmaz et al. 2008). Many plants possess specific mechanisms to increase metal bioavailability and accumulate metals in roots (Romheld and Marschner 1986), for example, by blocking the binding of ions to ion carriers. This association often results in decreased plant growth (Pahlsson 1989); however, *T. latifolia* grew well and tolerated moderate Pb concentrations.

**Bioconcentration Factor (BCF)**

The bioconcentration factor reflects the progressive accumulation of metal from soil into a specific plant part (Branquinho et al. 2007). *Typha* shoots had BCF values for Cu ranging from 0.7 (Stage 1) to 2.8 (Stage 2), and for Pb ranging from 2.8 (Stage 1) to 8.0 (Stage 2) (Table 7). BCF values for shoots >1.0 indicates an ability of the plant to absorb and transport metals from sediment and subsequently store them in above-ground biomass (Baker et al. 1994; Brown et al. 1994; Wei et al. 2002). *Typha* roots had BCF >1 for Pb (4.19 in Stage 1 and 5.82 in Stage 2) (Table 7), indicating its ability to uptake Pb from sediments to roots. Kaewtubtim et al. (2016) found BCF values of vetiver (*Vetiveria zizanioides*) leaves to range from 0.4-9.0 for Cu and 0.2-22.7 for Pb.

**Transfer Factor (TF)**

The transfer factor (TF) is used to estimate a plant’s potential to transfer a metal between roots and aboveground parts (leaves and stems) for phytoremediation purposes. In the present study, *Typha* had a TF <1 for Pb in Stage 1 of the CW (Table 7). TF values <1 indicate low metal translocation to shoots. These results are consistent with those from terrestrial plants grown in other metal-contaminated soils (Meeinkuirt et al. 2012; Phaenark et al. 2009). In a study by Sasmaz et al. (2008), transfer factors for metals in *T. latifolia* ranged between 0.39 and 1.18. Kaewtubtim et al. (2016) found TFs in various species to range from 0.2 to 4.4 for Cu and 0.1 to 7.9 for Pb. Mojiri et al. (2013) found a TF of 1.0 using *Typha domingensis*, which was an effective accumulator plant for phytoremediation of several heavy metals.

The extent of metal accumulation depends upon both plant mechanisms and sediment chemistry (Chaudhuri et al. 2014). Aerial roots of many plant species diffuse oxygen into the substrate so that oxidation occurs in the rhizosphere, resulting in metal accumulation in fine roots (Chaudhuri et al. 2014; Machado et al. 2005; Marchand et al. 2011). The large surface area and high density of the *Typha* root system may encourage metal uptake, along with adsorption of metals (Lacerda et al. 1992, 1993; Marchand et al. 2011; Otero et al. 2006).

*Typha* had a TF of 1.4 for Pb in Stage 2 of the CW (Table 7). Plants with a TF >1 efficiently translocate metals from roots to above-ground parts (Murray et al. 2009). This effect is most likely due to efficient metal transporter systems (Zhao et al. 2003), and probably to sequestration of metals in leaf vacuoles and apoplasm (Lasat et al. 2000). Baker (1981) and Zu et al.
(2005) reported TFs that were greater than 1.0 in metal accumulator species, and TFs that were typically <1.0 in metal excluder species. In the current study, *Typha latifolia* may be categorized as a Pb accumulator as it had TF and BCF values >1 (except for Stage 1, where the TF was 0.67).

### SUMMARY

Saline and metal-enriched wastewater is a concern to oil and gas producing companies, many of whom rely on purchased water and recycled wastewater to support drilling operations. Recycling PW is a continuous, long-term process which requires a reliable and effective system. In a pilot-scale constructed wetland, the pH of PW increased markedly, from 4.2 to 7.0, and electrical conductivity decreased substantially. *Typha* plants grown in both Stages 1 and 2 produced substantial biomass, perhaps from receiving a large dose of Ca, Mg and K. Bioconcentration and transfer factors indicate that *Typha* is effective for metal removal from PWs. The present study may be of practical value to the oil and gas production industries, which generate large quantities of contaminated drilling wastewater.

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