Characterization of Drinking Water Treatment Residuals for Use as a Soil Substitute

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ABSTRACT: The beneficial use of drinking water treatment residuals (WTRs) as a potential source of topsoil for land reclamation was evaluated. Seventeen WTRs were characterized for use as soil substitutes by comparing chemical and physical properties and plant nutrients of the WTRs with soil. A tomato (Lycopersicon esculentum) bioassay was performed to determine the ability of soil chemical tests to measure WTR phosphorus (P) adequacy. The WTR chemical and physical properties were typically adequate for crop growth. None of the WTRs were considered unsuitable as soil substitutes based on plant nutrients, with the exception of P. Tomato vegetative yield and tissue P were poor either because of phytotoxic nitrate-nitrogen (NO$_3$-N) (>10 mg/kg) generated during the bioassay or because of WTR P deficiency. Limited data suggest that WTRs with NO$_3$-N less than 10 mg/kg and Olsen P greater than 50 mg/kg, water soluble P greater than 580 µg/L, or Mehlich III P greater than 54 mg/kg support growth but still produce inadequate tissue P in tomatoes. Water Environ. Res., 73, 52 (2001).

KEYWORDS: alum sludge, artificial soil, drinking water residuals, remediation, residuals, revegetation, sludge, soil quality, soil substitute, waste management.

Water treatment residuals (WTRs), byproducts of drinking water treatment, have soillike qualities and the potential to be used as soil substitutes. Alum (aluminum sulfate) or polymers (polyaluminum chloride) are coagulants used, in conjunction with lime, to form an amorphous aluminum hydroxide (Al(OH)$_3$) gel during drinking water treatment. Coagulation is used to remove turbidity, color, taste, and odor from raw water and speed sedimentation. Water treatment residuals contain sediments from the raw water and the reaction products of coagulation, amorphous aluminum oxides, which account for 50 to 150 g/kg of the total residuals (ASCE and AWWA, 1996).

Elliott and Dempsey (1991) reviewed the chemical and physical properties of WTRs and reported that WTRs have a low calcium carbonate equivalence (CCE) value ranging from 100 to 200 g/kg and have little value as a liming material. Nutrient content of WTRs tends to be low unless the raw water source is contaminated with nutrients. Elliott and Dempsey (1991) reported that total nitrogen (N) ranged from 4.4 to 10 g/kg and that the phosphorus (P) content of WTRs is typically low. Furthermore, they warn that the P-fixing capability of WTRs can make soil P unavailable to plants. Total organic carbon (C) is typically approximately 30 g/kg, which contributes to good aggregation and water holding capacity in soils amended with WTRs. Aluminum (Al) and iron (Fe) oxides in WTRs also have a cementing effect, which contributes to soil aggregation.

The WTRs are currently disposed of in landfills (at great expense to municipalities), stored on onsite lagoons, or discharged to sanitary sewer systems. Because WTRs predominantly contain sediment and humic substances from the raw water, they are similar to fine-textured soils and may be suitable for use as soil substitutes (Elliott et al., 1988 and 1990). The use of WTRs as soil substitutes could be of economic benefit to municipalities and provide economic and environmental benefits for the reclamation of disturbed sites.

Several studies have shown that, although they improve soil properties such as water retention or pH, WTRs caused P deficiency and decreased yields as WTR application rates increased. Bugbee and Frink (1985) used WTRs as an amendment to a potting media at rates of 0 to 670 g/kg and found that addition of WTRs resulted in reduced P availability and reduced lettuce (Lactuca sativa) yields but increased water-holding capacity in the growing media. Heil and Barbarick (1989) applied WTRs at rates of 0 to 25 g/kg, grew sorghum-sudangrass (Sorghum bicolor sudanense), and found decreased yields with WTR additions greater than 15 g/kg as a result of P fixation by WTRs. Skene et al. (1995) experienced decreased growth of broad beans (Vicia faba) when WTRs were spread in an even layer on the surface of sand at rates of 20, 40, and 100 g/kg, with and without fertilizer addition. In a similar study, WTRs were added at rates of 0.1 to 10 g/kg to a growing media. Soil properties improved and yields of corn (Zea mays) increased in fertilized and unfertilized pots amended with WTRs (Rengasamy et al., 1980). At the high rate of application (10 g/kg), however, P uptake was reduced.

Application of WTRs at rates of 20 and 100 g/kg to a silt loam enhanced tomato (Lycopersicon esculentum) growth (Elliott and Singer, 1988). The authors attributed the increased growth to reduced Al and manganese (Mn) toxicity in the soil caused by an increase in pH from 5.3 to 8.0 that resulted from a liming effect of the WTRs. Additionally, heavy metal uptake in the plant shoots was reduced as a result of soil fixation at the higher pH. The WTRs have also been used to amend soil on field crops. Water treatment residual application rates greater than 4.5 g/kg decreased yields of wheat, even with P fertilizer additions (Cox et al., 1997). Alum- and polymer-WTRs applied to forests at rates ranging from 0.8 to 2.5 g/kg had no effect on growth or nutrient content after at least 1 year (Bugbee and Frink, 1985, and Novak et al., 1995).

Materials rich in amorphous Al oxides, such as WTRs, have the potential to adsorb labile P, making it unavailable to plants. Results from P fractionation experiments showed that the addition of WTRs to soil resulted in a decreased labile P fraction and an increased less-soluble chemisorbed Al- and Fe-bound P fraction (Cox et al., 1997, and Jonasson, 1996). Typically, high application rates of WTRs (>10%) have caused P deficiency in crops. Little
information is available about the use of 100% WTRs as soil substitutes. Topsoil is needed for the reclamation of disturbed sites such as abandoned strip mines, road construction sites, and landfill cover. Mining native topsoil for these purposes is environmentally unsound because it creates more disturbed sites. The beneficial use of municipal or industrial residuals as soil substitutes is a potential source of topsoil and may provide an economical disposal option for residuals.

For a residual material to be considered as a soil substitute, it must function like a soil. Soil quality has been defined as "the capacity of a soil to function, within ecosystem and land-use boundaries, to sustain biological productivity, maintain environmental quality and promote plant and animal health" (Doran and Parkin, 1996). A soil substitute suitable for plant growth should have desirable chemical properties (e.g., pH 5 to 8 and electrical conductivity <4 dS/m) and desirable physical properties (e.g., aeration, drainage, texture, and structure). To be beneficial, a soil substitute should not have toxicity problems (e.g., excessive heavy metals).

Lindsay and Logan (1998) suggest developing soil substitutes by blending residual materials (e.g., alum WTRs and alkaline-stabilized biosolids) according to each residual's chemical and physical properties. However, little information is available about the chemical and physical properties of many types of WTRs to determine their suitability to serve as a soil substitute. The primary objective of this work was to determine the suitability of WTRs as soil substitutes by measuring the physical and chemical characteristics of a variety of WTRs. A secondary objective was to evaluate the ability of soil chemical tests to measure WTR P availability by using a tomato bioassay.

Materials and Methods

Seventeen WTRs, collected from municipalities across Oklahoma, were used in this study. Fourteen were alum based, and three were polymer (polyaluminum chloride) based. All WTRs were air dried and crushed to less than 2.0 mm.

Water Treatment Residuals Chemical and Physical Properties. The WTR pH was determined in a 1:2 WTR:0.01 M calcium chloride (CaCl₂) solution using a glass electrode (McLean, 1982). Cation-exchange capacity (CEC) was determined by sodium (Na) displacement (Rhoades, 1982). Electrical conductivity (EC) was measured in a 1:2 WTR:deionized water solution (Rhoades, 1996). Gravimetric water content was measured at 0.033 and 1.5 MPa in a pressure plate apparatus (Klute, 1986). Plant-available water (PAW) was considered the water content between 0.033 and 1.5 MPa. Bulk density was determined by measuring the dry weight of the WTRs in the tomato pots, followed by estimating volume by replacement of WTR with water (Blake and Hartge, 1986). Organic C was determined by dichromate oxidation, followed by colorimetric analysis (Nelson and Sommers, 1982).

Plant-Available Nutrients. Total N was determined by the Dumas method using a Carlo Erba (Milan, Italy) 1500 series dry combustion analyzer (Bremner, 1996). Using automated colorimetric analysis, ammonium-nitrogen (NH₄-N) was determined by the indophenol blue method and nitrate-nitrogen (NO₃-N) by the Griess–Ilosvay method (Mulvaney, 1996). Available P in WTR was measured using Mehlich III (M III) extraction (Mehlich, 1985) and by the Olsen method (Kuo, 1996), followed by inductively coupled plasma (ICP) atomic emission spectroscopy analysis. Water-soluble (WS) P was determined by shaking 5 g WTRs in 25 mL deionized water for 1 hour and subsequent ICP analysis. Potassium (K), calcium (Ca), and magnesium (Mg) were determined by M III extraction (Mehlich, 1985), followed by ICP analysis. Sulfate (SO₄) was determined by calcium phosphate (Ca(H₂PO₄)₂) extraction, followed by ICP analysis (Tabatabai, 1982). Plant-available Fe (Olson and Ellis, 1982) and Zn (Baker and Amacher, 1982) were determined by diethyleneetriaminepentaacetic acid (DTPA) extraction, followed by ICP analysis.

Tomato Bioassay. Fourteen WTRs ranging in type (11 alum based and 3 polymer based) and M III extractable P (1.6 to 54.3 mg/kg) were selected for the tomato bioassay. Five tomato seeds were planted in 1 kg WTRs and grown in a controlled-environment growth chamber with daytime temperatures of 25 °C and nighttime temperatures of 23 °C. Three replicates of each WTR were potted and placed in a completely randomized design. To ensure that the bioassay reflected only P availability, excess plant available macronutrients (N and K) were maintained. Excess N and K were added as KNO₃ so that each pot had greater than 75 mg/kg N and greater than 125 mg/kg K. Plants were harvested after 8 weeks; foliage was washed with deionized water and dried for 48 hours in a forced-air dryer at 75 °C. The dried material was ground to less than 0.15 mm (100 mesh) and weighed to determine yield. Foliage was wet-digested in hot nitric acid (HNO₃) as described by Zarzinas et al. (1987), followed by analysis of P by ICP.

Potential Effects. Heavy metals were extracted from WTRs according to the U.S. Environmental Protection Agency toxicity characteristic leaching procedure (TCLP; U.S. EPA, 1988). Soluble Al and NO₃-N were measured to determine potential phytotoxicity by shaking 5 g WTRs in 25 mL deionized water for 1 hour. Extracted Al was determined by ICP analysis, and NO₃-N was determined by ion chromatography. Nitrite-nitrogen measurements were confirmed colorimetrically by the Griess–Ilosvay method (Mulvaney, 1996).

Results and Discussion

To determine the similarity of WTRs to soils, typical soil chemical and physical properties (soil quality parameters) and nutrient levels, adequate for most crop growth, were compared to WTR levels.

Chemical and Physical Properties. Typical soil levels of selected soil quality parameters adequate for plant growth (Brady and Weil, 1996) were compared to WTR levels (Table 1). The pH of WTRs ranged from 5.3 to 7.8, with a median of 7.1, within the typical range of 5.0 to 8.0 adequate for plant growth (Bohn et al., 1985). The EC of the WTRs ranged from 0.22 to 1.1 dS/m (Table 1), well below the 4 dS/m associated with reduced plant growth caused by soil salinity.

The CEC of the WTRs ranged from 13.6 to 56.5 cmol/kg, with a median of 30 cmol/kg, greater than the typical soil range of 3.5 to 35.6 cmol/kg. The high CEC associated with some WTRs indicates that these materials have the ability to supply cationic nutrients for plant growth. Bulk density of the WTRs ranged from 0.56 to 1.3 g/cm³, with a median of 0.9 g/cm³, lower than the typical range for soil of 1.0 to 1.55 g/cm³. Bulk density values less than 0.75 g/cm³ may indicate that the WTR is too porous to be suitable as a soil substitute because of excessive drainage and low water-holding capacity. The gravimetric water-holding capacity of the WTR, measured at 0.033 MPa, ranged from 187 to 710 g/kg, with a median of 400 g/kg.

Plant-available water was considered the difference between the water content at 0.033 and 1.5 MPa and ranged from 26 to 416
Table 1—Comparison of soil chemical and physical properties of WTRs with typical soil levels adequate for crop growth.

<table>
<thead>
<tr>
<th>WTR type</th>
<th>pH</th>
<th>Electrical conductivity, dS/m</th>
<th>Cation-exchange capacity, cmol/kg</th>
<th>Total N, g/kg</th>
<th>Organic C, g/kg</th>
<th>Bulk density, g/cm³</th>
<th>Plant, available water, g/kg</th>
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<td>16.4</td>
<td>2.8</td>
<td>23</td>
<td>1.3</td>
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</table>

WTR range: 5.3–7.8, 0.22–1.1, 13.6–56.5, 1.3–18.4, 17–149, 0.56–1.3, 26–416

WTR median: 7.1, 0.5, 30.0, 7.0, 63, 0.9, 139

Soil typical: 5.0–8.0

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Organic C content of WTRs ranged from 17 to 149 g/kg, with a median of 63 g/kg, considerably greater than typical soil levels of less than 30 g/kg. Organic C measurements in WTRs, however, may partly be the result of the addition of activated charcoal or organic polymers during the water treatment process.

Nutrients. Nutrient levels in WTRs were compared to soil nutrient levels considered adequate for most crops (Table 2; Johnson et al., 1997). Available inorganic N (NH₄-N and NO₃-N) ranged from 28 to 263 mg/kg, with a median of 79 mg/kg, within the adequate soil available N range of most crops. Nitrate-nitrogen levels ranged from 3.5 to 123 mg/kg, with a median of 17 mg/kg. Nitrogen-nitrogen levels ranged from 2 to 44 mg/kg, with a median of 13.1 mg/kg.

Three soil extraction procedures (Olsen, M III, and water) were used to evaluate P nutrient adequacy in WTRs. Olsen-extractable WTR P levels ranged from 4 to 49 mg/kg, with a median of 13.1 mg/kg, slightly greater than the adequate soil level of 12 mg/kg (Tisdale et al., 1985) for most crops. The WTR M-III-extractable P level ranged from 1.6 to 54.4 mg/kg, with a median of 6.8 mg/kg, less than the 32.5 mg/kg soil level considered adequate for most crops. Water-soluble P levels ranged from 34 to 256 μg/L, with a median of 98 μg/L, within the adequate soil WS P range of 50 to 200 μg/L, with a mean of 125 μg/L (Fohse et al., 1988).

Mehlich III WTR K levels ranged from 19 to 278 mg/kg, with a median of 109 mg/kg, slightly less than the adequate soil K level of 125 mg/kg. Mehlich-III-extractable Ca levels ranged from 0.18 to 21 g/kg, with a median of 2.6 g/kg, considerably greater than the adequate soil Ca value of 0.38 g/kg. The high available Ca level is likely the result of pH adjustment with lime during water treatment.

The WTR SO₄ levels ranged from 12.5 to 453 mg/kg, with a median of 138 mg/kg, greater than the adequate soil SO₄ level of 14 mg/kg. Mehlich-III-extractable Mg levels ranged from 8.0 to 1231 mg/kg, with a median of 117 mg/kg, higher than the adequate soil Mg level of 50 mg/kg. The WTR DTPA-extractable Fe level ranged from 8 to 231 mg/kg, with a median of 60.4 mg/kg, considerably greater than the adequate soil Fe value of 4.5 mg/kg. The WTR DTPA-extractable Zn level ranged from 0.12 to 70 mg/kg, with a median of 3.0 mg/kg, greater than the adequate soil Zn level of 0.8 mg/kg.

Of all the nutrient levels tested, only some nutrient P levels seem grossly deficient, and this deficiency may be difficult to overcome because of the P-adsorption capacity of WTRs. Added P can become fixed to Al–OH groups and be unavailable to plants (Cox et al., 1997, and Jonasson, 1996). Individual deficiencies in other nutrients should be easily correctable with fertilizer.

Total N levels for WTRs ranged widely from 1.3 to 18.4 g/kg, with a median value of 7 g/kg (Table 1), greater than the typical...
soil test P (M III, Olsen, or WS) was not obtained. Growth, a reliable correlation between either yield or tissue P with
2000 mg/kg; 1000 mg/kg is considered deficient (Geraldson et al., early bloom stage is 2500 mg/kg, and at the intermediate stage is a median of 923 mg/kg. Sufficient tissue concentration of P at the median of 0.052 g. Tissue P ranged from 561 to 1840 mg/kg, with

Mean vegetative yield ranged from 0.017 to 12.1 g per pot, with a (34 to 576 [Lg/L], tomato vegetative yield and tissue P were low. M

mg/L) (Sparks, 1995). So Al toxicity problems are not expected. in soybeans 0.054 mg/L, less than the level found to cause toxicity symptoms

WTR heavy metal levels were significantly less than the regulatory levels for the TCLP and consistent with nonhazardous waste. The soluble Al level ranged from 0.02 to 0.92 mg./L, with a median of 0.054 mg/L, less than the level found to cause toxicity symptoms in soybeans (Glycine max [L.] Merr.) (1.8 mg/L) or corn (3.6 mg/L) (Sparks, 1995). So Al toxicity problems are not expected.

Potential Toxicity. The TCLP (U.S. EPA, 1988) is used to characterize municipal and industrial solid waste as hazardous or nonhazardous for the purpose of landfelling. All of the measured WTR heavy metal levels were significantly less than the regulatory levels for the TCLP and consistent with nonhazardous waste. The soluble Al level ranged from 0.02 to 0.92 mg/L, with a median of 0.054 mg/L, less than the level found to cause toxicity symptoms in soybeans (Glycine max [L.] Merr.) (1.8 mg/L) or corn (3.6 mg/L) (Sparks, 1995). So Al toxicity problems are not expected.

Table 2—Comparison of WTR nutrient levels* with soil nutrient levels adequate for growth of most crops.

<table>
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<tr>
<th>WTR</th>
<th>Soluble P, Olsen P*, M III P, NH₄-N, NO₃-N, K, Mg, Fe, Zn, Ca</th>
<th>µg/L</th>
<th>mg/kg</th>
<th>mg/kg</th>
<th>mg/kg</th>
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<td>17.7</td>
<td>23.3</td>
<td>63.1</td>
<td>16</td>
<td>102.4</td>
<td>89</td>
<td>22</td>
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<tr>
<td>17</td>
<td>AL</td>
<td>68.2</td>
<td>15</td>
<td>16.8</td>
<td>26.9</td>
<td>13.9</td>
<td>117</td>
<td>89.8</td>
<td>4.</td>
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</tbody>
</table>

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<th>WTR range</th>
<th>Soluble P, Olsen P*, M III P, NH₄-N, NO₃-N, K, Mg, Fe, Zn, Ca</th>
<th>µg/L</th>
<th>mg/kg</th>
<th>mg/kg</th>
<th>mg/kg</th>
<th>mg/kg</th>
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<td>34-576</td>
<td>4-49</td>
<td>1.6-54.4</td>
<td>22-140</td>
<td>3.5-123</td>
<td>19-278</td>
<td>8-1231</td>
<td>8-231</td>
<td>0.12-70</td>
<td>0.18-21</td>
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</tbody>
</table>

Adequate soil level: 50-200 µg/L

* Olsen P (Kuo, 1996); M III P, K, Mg, and Ca (Mehlich, 1955); NH₄-N and NO₃-N (Mulvaney, 1996); Fe (Olsen and Ellis, 1982); and Zn (Baker and Amacher, 1982).

Although initially low, nitrite-nitrogen levels increased dramatically 3 and 2 months after the conclusion of the bioassay (week 16). WTRs before planting (week 0), during tomato growth (week 8), and 2 months after the conclusion of the bioassay (week 16). The activity of Nitrobacter, an NO₃ oxidizer, is inhibited by high pH and high NH₄ levels, more than that of NH₄ oxidizers, and, under these conditions, NO₃ can accumulate (Alexander, 1977, and Haynes and Sherlock, 1986). Alexander (1977) states that NH₄-N concentrations of 1.4 mg/kg can inhibit Nitrobacter while having no effect on ammonium oxidizers and that the NH₃, not the cationic NH₄, that forms under high pH conditions (pH 9.5) is toxic to Nitrobacter. However, none of the WTRs had a pH greater than 7.8. Purchase (1974) found a relationship between the growth of nitrite oxidizers and available P. He found that nitrite oxidizers grown in media were inhibited by low levels of available P. Addition of NH₄ to media with low P (less than 700 µg/mL) resulted in nitrite accumulation.

However, this study did not find a consistent trend between NO₃-N, soluble P, and NH₄ in the 14 WTRs studied. Figure 1 illustrates the change in NO₃-N concentrations in the five affected WTRs before planting (week 0), during tomato growth (week 8), and 2 months after the conclusion of the bioassay (week 16). Although initially low, nitrite-nitrogen levels increased dramatically.

soil total N content of 0.2 to 5.0 g/kg. High total N levels in WTR are likely caused by organic constituents (algae, detritus, etc.) removed from the raw water that is concentrated in the WTRs. Three of the WTRs have total N levels greater than 10 g/kg, which may present a NO₃ pollution hazard. Assuming a 1% mineralization rate, a 1% total N level will release 2000 kg/ha of N.
Figure 1—Water soluble NO$_2$–N levels of WTRs 1, 2, 3, 4, and 13 in tomato pots, from tomato bioassay at 0, 8, and 16 weeks.

Conclusions

For a WTR to be considered as a soil substitute, it must be able to function like a soil. For the purpose of this study, the function is to support crop growth while not harming the environment. Chemical and physical properties of most WTRs were adequate for plant growth. However, low PAW and bulk density associated with some WTRs may make them unsuitable as soil substitutes. With the exception of P levels, none of the WTRs were considered unsuitable as a soil substitute based on available nutrient levels. Aside from P, individual nutrient deficiencies should be correctable with moderate amounts of fertilizer.

The generation of phytotoxic levels of NO$_2$–N in some WTRs makes them unsuitable as soil substitutes. Of the 14 WTRs used in the tomato bioassay, 5 generated phytotoxic NO$_2$–N levels (greater than 10 mg/kg). Further study of NO$_2$ generation is necessary to determine if it is a temporal problem or if it can be mitigated by an adjustment of pH or nutrient status. There were no other toxicity problems evident in any of the WTRs. All of the TCLP contaminants measured were below the regulatory levels and water soluble Al (less than 1.0 mg/L).

In addition to chemical and physical characterization of WTRs, the ability to predict the potential for generation of phytotoxic NO$_2$–N is necessary to determine the suitability of WTRs as soil substitutes. Only one WTR had substantial growth (WTR 14), but tissue P concentration was still insufficient. Limited data from this study suggest that WTRs with NO$_2$–N levels less than 10 mg/kg and levels of Olsen P greater than 50 mg/kg, WS P greater than 580 µg/L, or M III P greater than 54 mg/kg support growth but still produce inadequate tissue P in tomatoes. Detailed studies are

Figure 2—Average tomato vegetative yield per pot (×10), Olsen-extractable P, water soluble P (divided by 10), Mehlich-III-extractable P, and tomato tissue P (divided by 100). ******* indicates soil adequate P level, and unfilled symbols denote WTRs with toxic levels of NO$_2$–N (>10 mg/kg).
needed to focus on the factors responsible for NO₂ generation and to evaluate the ability of soil P extraction methods to accurately measure the adequacy of WTR P levels for plant growth.

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