Journal of Environmental Quality TECHNICAL REPORTS

WASTE MANAGEMENT

Nutrients and Nonessential Elements in Soil after 11 Years of Wastewater Irrigation

B. F. Faria Pereira, Zhenli He,* Peter J. Stoffella, Celia R. Montes, Adolpho J. Melfi, and Virupax C. Baligar

Irrigation of citrus (*Citrus aurantium* L. × *Citrus paradise* Macf.) with urban reclaimed wastewater (RWW) can be economical and conserve fresh water. However, concerns remain regarding its deleterious effects on soil quality. We investigated the ionic speciation (ISP) of RWW and potential impacts of 11 yr of irrigation with RWW on soil quality, compared with well-water (WW) irrigation. Most of nutrients (~53–99%) in RWW are free ionic species and readily available for plant uptake, such as: NH_4^+, NO_3^-, K^+, Ca^{2+}, Mg^{2+}, SO_4^{2-}, H_3BO_3, Cl^-, Fe^{2+}, Mn^{2+}, Zn^{2+}, Co^{2+}, and Ni^{2+}, whereas more than about 80% of Cu, Cr, Pb, and Al are complexed with CO_3^-, OH^-, and/or organic matter. The RWW irrigation increased the availability and total concentrations of nutrients and nonessential elements, and soil salinity and sodicity by two to three times compared with WW-irrigated soils. Although RWW irrigation changed many soil parameters, no difference in citrus yield was observed. The risk of negative impacts from RWW irrigation on soil quality appears to be minimal because of: (i) adequate quality of RWW, according to USEPA limits; (ii) low concentrations of metals in soil after 11 yr of irrigation with RWW; and (iii) rapid leaching of salts in RWW-irrigated soil during the rainy season.

World population is projected to grow from 6.8 billion today to 9.1 billion by 2050 (Nature, 2010), which will consequently increase competition for fresh water resources. Beneficial reuse of urban reclaimed wastewater (RWW) could be one solution to this challenge (O'Connor et al., 2008). Irrigation with urban RWW is becoming a common practice in suburban areas of major cities (Biggs and Jiang, 2009) due to low transport costs and benefits, including savings in water and fertilizer costs, and reduced pollutant discharges downstream (Anderson, 2003). In 1995, only 2% of RWW was reused in the United States, but it recently has increased to 7.4% (of the total 9.8×10^6 m^3 d^{-1}) at a rate of 15% rise in usage per year (Miller, 2006; O'Connor et al., 2008). In Florida, ~5300 ha of edible crop, such as citrus, tomatoes, strawberries, peas, beans, corn, and herbs, were irrigated with RWW in 2010 (FDEP, 2010).

Reclaimed wastewater irrigation is a source of macronutrients (N, P, K, S, Ca, Mg) (Barton et al., 2005; Jaiswal and Elliott, 2011; Sophocleous et al., 2009) and micronutrients (B, Cl, Cu, Co, Zn, Fe, Mn, Mo, Ni) (Pedrero and Alarcón, 2009; Xu et al., 2010), but they may not be in a balanced supply to meet plant nutritional needs. The RWW may also contain nonessential/toxic elements, such as Na, Al, Cd, Cr, and Pb. Therefore, one of the concerns regarding the use of RWW for irrigation in agriculture is its impact on soil quality, such as increases in heavy metals, salinity, and sodicity (Biggs and Jiang, 2009; Darvishi et al., 2010; Pedrero and Alarcón, 2009; Reboll et al., 2000; USEPA, 2004). Some micronutrients, especially B and Cl, and nonessential elements (Na, Al, Pb, Cd, Cr), were reported to accumulate in soil and plant after long-term irrigation, depending on the quality of RWW, irrigation rate, soil properties, and crop usage (van Oort et al., 2008; Pedrero et al., 2010; Rusan et al., 2007; Singh et al., 2009; Toze, 2006).

Many previous studies report total concentration of elements in RWW (Biggs and Jiang, 2009; Klay et al., 2010;

B.F.F. Pereira, Z.L. He, and P.J. Stoffella, Indian River Research and Education Center, Institute of Food and Agricultural Sciences, Univ. of Florida, 2199 S. Rock Rd., Fort Pierce, FL 34945. B.F.F. Pereira, Faculdade de Ciências Agrárias, Univ. Federal do Amazonas, 69077-000, Minicampus, Coroado, Setor Sul, Manaus, AM, Brazil. C.R. Montes, Center for Nuclear Energy in Agriculture, Univ. of Sao Paulo, Avenida Centenário, 303, 13416-903, Piracicaba, São Paulo, Brazil. A.J. Melfi, Univ. of Sao Paulo, P.O. Box 09, Piracicaba, São Paulo, Brazil, 13418-900. Virupax C. Baligar, USDA–ARS, Beltsville Agricultural Research Center, Beltsville, MD 20705-2350.
Assigned to Associate Editor Joselito M. Arocena.

Abbreviations: APLR, annual pollutant load for each metal; ARI, annual rate of irrigation; EC, electrical conductivity; ISP, ionic speciation; M-3, Mehlich-3 solution; PP, plant population; RWW, reclaimed wastewater; SAR, sodium absorption ratio; TN, total nitrogen; WTP, wastewater treatment plant; WW, well water.

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J. Environ. Qual. 41:920–927 (2012)
doi:10.2134/jeq2011.0047
Received 14 Feb. 2011.
*Corresponding author (zhe@ufl.edu).
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5585 Guilford Rd., Madison, WI 53711 USA

Qian and Mecham, 2005) and often lack indications as to their bioavailability, mobility, and fate in soil (D'Amore et al., 2005). The ionic speciation (ISP) of elements can help overcome this deficiency of information (Sparks, 2003). Free ionic species in soil solution, such as Mg^{2+}, K^{+}, Ca^{2+}, Mn^{2+}, Co^{2+}, Cu^{2+}, Zn^{2+}, and Fe^{2+}, are readily available for plant uptake (Sposito, 2008). Higher plant response to nutrient additions is usually correlated with the activity of free ions in solution (M^{n+}) and largely indifferent to metal–ligand complexes (ML) (Parker and Pedler, 1997).

The interest in RWW irrigation for citrus in Brazil, the United States, and Spain has been recently increased as an alternative source of water and nutrients (Aucejo et al., 1997; Fonseca et al., 2007; Morgan et al., 2008; Pedrero and Alarcón, 2009; Pereira et al., 2011a; Reboll et al., 2000). For the safe use of RWW in agriculture, it is necessary to critically examine its effects on soil quality, particularly chemical properties using long-term field trials, compared with the common practice of well-water (WW) irrigation. In addition, there is limited information available on the ISP of RWW and the influence of long-term irrigation with RWW on soil quality in perennial citrus crop production systems. The objectives of this study were to: (i) determine plant availability of macronutrients and micronutrients, as well as nonessential elements in RWW using an ion-speciation model; and (ii) examine the potential impact of long-term irrigation with RWW on soil quality, including salinity, sodicity, nutrient concentration, and accumulation of nonessential elements.

Materials and Methods

Area Description and Irrigation System

This study was conducted in a commercial citrus grove in South Florida, USA. The soil is a loamy, siliceous, hyperthermic, Arenic Glossaqualfs (USDA, 1980). Physical and chemical characteristics of the soil were measured before field trials (Table 1). Two citrus blocks, each 8.1 ha, were selected based on similar soil properties, rate of irrigation, citrus cultivar, and tree density. Both blocks were planted with grapefruit (*Citrus paradise* Macf.) grafted on sour orange (*Citrus aurantium* L.) on an 8-m by 5-m spacing. One block was irrigated with urban RWW and other, the control, with WW (conventional source of irrigation water). Citrus trees were transplanted in 1982 for RWW and 1974 for the WW block. In the respective blocks, RWW and WW have been applied from 1997 through 2008 with micro-irrigation system, according to daily crop reference evapotranspiration estimated by Penman–Monteith equation (Allen et al., 1998). At the annual rate of 518 mm for RWW and 485 mm for WW, 360° microsprinklers (60.5 L h^{-1}) were used. Dry granular fertilizers were applied annually during the 11-yr period at 450 kg ha^{-1} (14–0–18 + 3 Mg) in spring, 450 kg ha^{-1} (12–0–16 + 3 Mg) in early summer, and 225 kg ha^{-1} (14–0–16 + 3 Mg) in autumn. In addition, 2.2 kg ha^{-1} of liquid fertilizers (0–29–26) were sprayed twice per year.

Field Sampling and Laboratory Analysis

Soil Sampling and Analysis

In 2008, after 11 yr of irrigation with RWW or WW, soil samples were collected. Four grids (replications) were randomly distributed on each block. Each grid was 0.14 ha^2 and used to collect five soil cores to make one composite sample. The sampling points were previously located using Google Earth and a GPS instrument with accuracy of ±3 m. The soil samples were collected in April 2008, at the depth of 0 to 15, 15 to 30, 30 to 45, and 45 to 90 cm, using a bucket auger within the area wetted by the irrigation system (~1.0 m from the emitter and ~1.0 m from the trunk). A total of 80 soil samples were collected per block (20 points × 4 depths).

Air-dried soils were ground to <2 mm particle size before chemical analyses. Soil salinity was estimated by electrical conductivity (EC) at a soil:water ratio of 1:2 (Rhoades, 1996). The pH was determined in $CaCl_2$ (0.01 M) at 1:1 soil:water ratio (Thomas, 1996), using a pH/conductivity meter (Model 220, Denver Instrument Inc.). Total carbon and total nitrogen (TN) were determined using a CN analyzer (Vario Max CN, Elemental Analysensystem GmbH). A KCl (2 M) extraction procedure was used to estimate available N (NO_3^- and NH_4^+). Concentrations of NO_3^--N and NH_4^+-N in the extracts were determined using an auto analyzer (Easychem plus, Systea Scientific Inc). Extractable P, K, Ca, Mg, Mn, Mo, Cu, Fe, Al, Cd, Cr, Ni, Pb, and Zn in soil were estimated by the Mehlich-3 (M-3) method (Mehlich, 1984) and concentrations of the elements in extracts were determined using inductively coupled plasma–optical emission spectrometry (ICP–OES, Ultima, JY Horiba). Samples (0.2 g each) of surface soil (0–15 cm) were digested following USEPA Method 3050B to determine total recoverable metals (Cd, Co, Cu, Cr, Mo, Ni, Pb, and Zn) (USEPA, 1996). Available B in soil was extracted with hot water (Keren, 1996) and extracts were analyzed for B using ICP–OES. Saturated paste was prepared to obtain sufficient soil solution by centrifugation, which was then used for the determination of Ca, Mg, SO_4, Cl, and Na concentrations (Wolt, 1994). Sulfate

Table 1. Basic physical and chemical properties of the studied soil.†

Depth	Sand	Silt	Clay	Bulk density	Exchangeable cations				Exchangeable acidity	CEC‡	BS‡	OC‡	EC‡	pH_{CaCl2}
					Ca	Mg	Na	K						
cm	%			g cm^{-3}	$cmol_c$ kg^{-1}						%		µS cm^{-1}	
0–7	90.1	8.6	1.3	1.1	9.45	0.71	0.30	0.07	4.0	14.50	73	3.4	260	6.3
7–15	96.8	2.5	0.7	1.4	2.41	0.16	0.11	0.01	0.5	3.21	84	0.5	180	7.3
15–30	97.5	1.3	1.2	1.5	2.38	0.12	0.11	0.01	0.4	3.05	87	0.4	150	7.5
30–53	97.0	0.6	2.4	1.5	0.74	0.06	0.15	<DL§	0.2	1.12	86	0.1	250	7.4
53–86	97.2	1.0	1.8	1.6	0.54	0.04	0.11	<DL	0.3	0.95	76	0.1	380	6.1
86–96	96.7	1.2	2.1	1.6	1.24	0.12	0.29	<DL	0.4	2.08	79	0.1	550	6.6

† Adapted from Soil Survey (USDA, 1980).

‡ CEC = cation exchange capacity; BS = base saturation; OC = organic carbon; EC = electrical conductivity.

§ <DL, below detection limit (0.005 $cmol_c$ kg^{-1}).

and Cl^- were analyzed using an Ion Chromatograph (DX 500; Dionex Corporation). Sodium, Ca, and Mg were determined by ICP–OES and soil sodicity was estimated by the sodium adsorption ratio (SAR) equation (Richards, 1954).

Reclaimed Wastewater and Well-water Analysis

The urban RWW was provided by a municipal wastewater treatment plant (WTP) in Okeechobee, FL. The primary treatment of wastewater removed heavy solids and floatable materials, whereas the secondary treatment consisted of extended aeration and chlorine disinfection. Ninety RWW samples were collected and analyzed at the WTP from 2001 to 2008, whereas five WW samples were collected from the microsprinklers in the WW-irrigated block in 2008. The RWW and WW samples were analyzed according to the following methods: EC (American Public Health Association, 1992), pH, biochemical oxygen demand (5 d at 20°C), chemical oxygen demand, total Kjeldahl N, total P, total suspended solids, NO_3^--N, NO_2^--N, PO_4^{3-}–P, SO_4^{2-}–S, B, Ca, Cu, Fe, K, Mn, Na, Zn, Cd, Cr, Al, Mg, Ni, Pb, and Se (USEPA, 1983), and Li (USEPA, 1986). The ISP of relevant elements in RWW was conducted using a geochemical model where the measured concentrations of elements were used as inputs to the visual MINTEQ version 3.0, β (Gustafsson, 2010). The organic ligands were estimated according to the "Gaussian DOM" model (Grimm et al., 1991), oversaturated solids were allowed to precipitate, the ionic strength was calculated, and the pH was fixed as determined in RWW. A concentration of 5.9 mg dissolved organic carbon L^{-1} was used for RWW (Jarusutthirak et al., 2002). The concentration of silicon was not considered in this model on the belief that alumino-silicate minerals are minimal in the municipal wastewater

Statistical Analysis

Means and standard errors of analyzed parameters were used to compare statistical differences between RWW and WW (Table 2). The Shapiro–Wilk test ($P > 0.05$) was used to evaluate the normality of the data of yield and dependent variables analyzed in soil. Then, the t-test at $P < 0.05$ was used to compare the effect of RWW with that of WW. Data for total recoverable concentrations of Cd, Co, Cu, Cr, Mo, Ni, Pb, and Zn were analyzed for the 0- to15-cm layer (Table 3). For pH, EC, SAR, saturated past extracted elements, and M-3 solution extracted elements, mean values at all depths were compared between the two water sources (Fig. 1 and 2).

Results and Discussion

Characteristics of Reclaimed Wastewater, Well Water, and Ionic Speciation

The RWW contained greater concentrations of nutrients and nonessential elements, except for Al and Ca, than WW (Table 2). The charge balance of ISP in RWW was ~12%, revealing a proper ionic distribution. Thus, the data are adequate for ISP calculation. Charge imbalance >30% is considered improper for the purpose of ISP calculation (Allison et al., 1991). The ISP revealed that the majority of macronutrients (NO_3^-, NH_4^+, K^+, Mg^{2+}, SO_4^{2-}), micronutrients (H_3BO_3, Cl^-, Co^{2+}, Mn^{2+}, Ni^{2+}, Zn^{2+}), and some nonessential elements (Ba^{2+}, Cd^{2+}, and Li^{2+}) were present as free ionic species (Table 2). Free ionic species in soil solution are readily available for plants (Sposito, 2008). However, dissolved Cu, Cr, Pb, and Al were mostly bound with CO_3^-, OH^-, and dissolved organic matter at pH 8.1 (Table 2), thereby decreasing their ionic activity, bioavailability, and potential phytotoxicity (Wolt, 1994). The free metal ions (M^{n+}) are generally the most bioavailable and toxic form of metals (McLean and Bledsoe, 1992).

In general, RWW is suitable for irrigation in agriculture, according to the measured properties. Reclaimed wastewater pH, SAR, alkalinity, biological oxygen demand, total suspended solids (TSS), nutrients (N, P, K, Ca, Mg, S, B, Cl, Cu, Fe, Zn), nonessential elements (Cd, Co, Cr, Pb, Se, and Al), and counts of pathogens (*Escherichia coli* and fecal coliforms) (Table 2) did not exceed reference limits established by USEPA (2004) or suggested by previous studies (Fatta et al., 2004; FAO, 1992; Feigin et al., 1991; James et al., 1982; Reboll et al., 2000; USEPA, 2004).

Total elemental concentrations in RWW decreased in the order of Ca > SO_4^{-2} > K > Mg > NO_3^-- *N* > PO_4^{3-}–P > NH_4^+-N for macronutrients, Cl > B > Zn > Fe > Cu = Mn > Ni for micronutrients, and Na > Al > Pb > Ba = Cr = Co > Cd for nonessential elements (Table 2). The elemental abundance suggested an imbalanced supply of nutrients in RWW, compared with the standard Hoagland nutrition solution (Epstein and Bloom, 2005) used for growing many plant species, which are: *N* > K > Ca > P > S > Mg for macronutrients and Fe > Cl > B > Mn > Zn > Cu = Ni for micronutrients.

The quality of WW used in this study is also adequate for irrigation. According to Food and Agriculture Organization irrigation water quality guidelines (FAO, 1985), WW used for irrigation was classified as slight degree of salinity, slight potential to affect rate of infiltration into the soil, no risk of Na, Cl, and B toxicity, and pH at a normal range.

Soil Sodicity and Salinity

Reclaimed wastewater irrigation increased soil salinity (EC) by about two to three times and soil sodicity (SAR) at all soil depths (Fig. 1). Soil pH was not significantly affected compared with WW irrigation. A higher concentration of Na in RWW than WW (Table 2) contributed to the increased soil sodicity and salinity (Fig. 1). Similar results were reported by Qian and Mecham (2005) after 5 yr of urban RWW irrigation of Renohill soil cultivated with ryegrass (*Lolium perenne* L.). However, they found higher values of SAR of 9.3 and EC of 4.3 dS m^{-1}, compared with the present study (Fig. 1).

Soil sodicity is usually evaluated with soil properties, such as pH, EC, and SAR, to identify the potential risk and negative effects on plant health and soil fertility. In general, if soil pH is <8.5, EC <4000 μS cm^{-1} with a SAR <13, the soil is considered suitable for agriculture (Brady and Weil, 2008). In this study, these three variables were lower than those critical values after 11 yr of irrigation with RWW for all soil depths (Fig. 1). Thus, there was a minimal influence of RWW on salinity and sodicity in the studied soil.

The salinization potential of a soil after long-term irrigation with RWW is also dependent on soil and climate conditions. An annual irrigation rate of 400 mm was reported to leach 80% of the salts in a loamy soil (Chesworth, 2008). The average annual precipitation in the studied area was 1372 mm yr^{-1} (NCDC, 2009); therefore, a large portion of salts from the RWW irrigation would be leached during the rainy season. Despite

the low salinization potential of RWW to a soil, further studies are needed to evaluate possible impacts on groundwater. The combination of high annual rainfall, sandy soils, and shallow water tables in Florida increases the risk of groundwater contamination (Syvertsen et al., 1993).

Macronutrients

Total organic C in the RWW-irrigated soils increased slightly (~2%) at the depth of 30 to 45 cm and no difference was observed in the other soil depths. This may be attributed to the following factors: (i) direct C input through RWW (Xu et

Table 2. Mean (± SE) values of variables in the well water (WW) (*n* = 5) and reclaimed wastewater (RWW) (*n* = 90). The reference limits to reuse reclaimed wastewater for irrigation (2001–2008) and predominant ionic species in RWW.

Variables	Unit	WW	RWW	Limits to RWW	Predominant ionic species in RWW†
		Mean ± SE			
pH	–	7.3 ± 0.1	8.1 ± 0.1	9‡	–
SAR§	–	0.9	3.7 ± 0.1	7.9¶	–
Na:Ca§	–	0.3	2.0	3#	–
Alkalinity (HCO_3^-)	mg L^{-1}	–	150.6 ± 3.0	700¶	HCO_3^- (95.3%); H_2CO_3 (1.5%)
BOD§	mg L^{-1}	–	13.9 ± 3.4	60‡	–
COD§	mg O_2 L^{-1}	–	64.4 ± 2.1	200‡	–
EC§	μS cm^{-1}	522.0 ± 26.1	995.0 ± 15.2	2000††	–
Turbidity	NTU§	1.1 ± 0.9	12.7 ± 0.9	50‡	–
TSS§	mg L^{-1}	–	17.5 ± 1.1	50‡	–
NO_3^--N	mg L^{-1}	0.3 ± 0.2	5.7 ± 0.3	50‡	NO_3^- (99.6%)
NO_2^--N	mg L^{-1}	<DL	0.5 ± 0.2	10¶	NO_2^- (99.9%)
NH_4^+-N	mg L^{-1}	0.1 ± 0.0	1.47 ± 0.2	40¶	NH_4^+ (93.6%); NH_3 (6.0%)
PO_4^{3-}–P	mg L^{-1}	<DL	2.2 ± 0.2	30‡	HPO_4^{2-} (63.0%); $CaHPO_4$ (15.7%)
SO_4^{2-}–S	mg L^{-1}	2.7 ± 0.0	24.47 ± 5.7	500‡	SO_4^{2-} (84.9%); $CaSO_4$ (10.6%)
TKN§	mg L^{-1}	–	4.1 ± 0.4	–	–
TP§	mg L^{-1}	–	2.9 ± 0.2	–	–
Ba	mg L^{-1}	–	0.01 ± 0.005	2‡	Ba^{2+} (92.5); BaDOM‡‡ (1.8%)
B	mg L^{-1}	0.1 ± 0.01	0.31 ± 0.01	0.75#	H_3BO_3 (92%); $H_2BO_3^-$ (7.5%)
Ca	mg L^{-1}	88.3 ± 2.8	60.6 ± 1.0	120¶	Ca^{2+} (88.7%); $CaSO_4$ (5.5%)
Cl	mg L^{-1}	28.0 ± 8.1	232.6 ± 14.9	360§§	Cl^- (99.5%)
Cu	mg L^{-1}	<DL	0.01 ± 0.005	0.2¶¶,‡,#	$CuCO_3$ (84.4%); CuOH (5.2%); $Cu(CO_3)_2^{2-}$ (4.4%); Cu^{2+} (1.8%)
Fe	mg L^{-1}	<DL	0.05 ± 0.005	5¶¶,#,‡	Fe^{2+} (83.5%); $FeHPO_4$ (6.0%)
K	mg L^{-1}	3.8 ± 0.1	17.1 ± 0.3	40¶	K^+ (99.4%)
Mn	mg L^{-1}	<DL	0.01 ± 0.005	0.2#,¶¶,‡	Mn^{2+} (65.5%); $MnCO_3$ (25.8%)
Na	mg L^{-1}	23.6 ± 0.06	122.1 ± 2.7	200‡	Na^+ (99.4%)
Zn	mg L^{-1}	0.01	0.06 ± 0.005	2¶¶,#,‡	Zn^{2+} (53.2%); $ZnOH^+$ (4.8%); $Zn(OH)_2$(6.9%); $ZnCO_3$ (24.1%)
Cd	mg L^{-1}	<DL	0.001	0.01¶¶,#	Cd^{2+} (56.7%); $CdCO_3$ (10.4%); $CdCl^+$(18.4%); $CdHPO_4$ (10.4%)
Co	mg L^{-1}	<DL	0.01 ± 0.005	0.05¶¶,#	Co^{2+} (73.2%); $CoCO^3$ (11.0%)
Cr	mg L^{-1}	<DL	0.01 ± 0.005	0.1¶¶,#	$CrOH^+$ (99.9%)
Li	mg L^{-1}	–	0.007 ± 0.002	2.5¶¶,#	Li^+ (99.5%)
Mg	mg L^{-1}	8.9 ± 0.2	11.1 ± 0.4	50‡¶	Mg^{2+} (91.1%); $MgSO_4$ (4.5%)
Ni	mg L^{-1}	<DL	0.008	0.2¶¶,#,‡	Ni^{2+} (67.7%); $NiCO_3$(18.3%); $NiHCO_3^+$ (11.54%); NiSO4 (3.4%); Ni DOM (1.9%)
Pb	mg L^{-1}	0.01 ± 0.00	0.04 ± 0.01	5#,¶¶	Pb DOM (40.4%); $PbCO_3$ (43.5%); $PbOH^+$ (3.7%); Pb^{2+} (1.6%)
Se	mg L^{-1}	–	<DL	0.02¶¶,#	–
Al	mg L^{-1}	1.2 ± 0.3	0.7 ± 0.1	5¶¶,#,‡	$Al(OH)_4^-$ (98.5%); $Al(OH)_3$ (1.4%)
Escherichia coli	100 mL^{-1}	<DL	<DL	–	–
Total coliforms	100 mL^{-1}	<DL	<DL	–	–

† Calculated with Visual Minteq 2.61; charge balance of 12.8%. <DL = below detection limit (NO_2–N 0.01, PO_4^3–P 0.01, Cu 0.005, Fe 0.006, Mn 0.001, Cd 0.0005, Co 0.0007, Cr 0.0006, Ni 0.001, and Se 0.01 mg L^{-1}).

‡ Fatta et al. (2004).

§ SAR = sodium adsorption ratio; Na:Ca = ratio of sodium to calcium; BOD = biochemical oxygen demand; COD = chemical oxygen demand; EC = electrical conductivity; NTU = nephelometric turbidity units; TSS = total suspended solids; TKN = total Kjeldahl nitrogen; TP = total phosphorus.

¶ Feigin et al. (1991).

USEPA (2004).

†† James et al. (1982).

‡‡ Dissolved organic matter.

§§ Reboll et al. (2000).

¶¶ FAO (1992).

al., 2010) due to the presence of dissolved organic matter (cell fragments and macromolecules) (Adrover et al., 2012); and (ii) RWW promotes higher biomass production (roots, trunk, and canopy) of citrus in Florida (Parsons et al., 2001), thus increasing C stock in the soil.

Irrigation with RWW increased TN and extractable NH_4^+-N and NO_3^--N, mainly at the surface layer (Fig. 1), as a consequence of NH_4^+ and NO_3^- inputs in RWW (Table 2). Leaching of NO_3^- is a concern in RWW irrigation (Sophocleous et al., 2009), particularly when associated with heavy applications of N fertilizers (USEPA, 2004). However, N addition from RWW seems insignificant based on the annual irrigation rate and total Kjeldahl nitrogen concentration in RWW (Table 2). The RWW added ~21 kg of N ha^{-1} yr^{-1}, compared with ~180 kg of N ha^{-1} yr^{-1} applied in fertilizers, which accounted for only ~10% of the total N inputs. The total N value is still within the recommended level of N fertilizers for citrus in Florida, which is 156 to 291 kg^{-1} of N ha^{-1} yr^{-1} (Obreza and Morgan, 2008).

Mehlich-3 extractable P in the soil irrigated with RWW increased in the 0- to 40-cm layer (~500% higher), reaching >56 mg kg^{-1} (Fig. 1), which is considered high, according to the citrus nutrition guideline for Florida (Obreza and Morgan, 2008). However, M-3 extractable P remained below the environmental threshold concentration of 150 mg kg^{-1} (Sims et al., 2002). Increased P concentration in the soil irrigated with RWW was expected. Usually the amount of P provided by RWW is higher than that removed by plants (USEPA, 2006). Palacios-Diaz et al. (2009) also reported higher concentration of extractable P (Olsen–P) in the soil irrigated with RWW, 22 to 50 mg kg^{-1}, compared with 14 to 22 mg kg^{-1} in the WW-irrigated soil. After 12 yr of RWW irrigation, Jaiswal and Elliott (2011) reported that M-3 extractable P in a 0- to 15-cm layer of an Ultisol remained ~110 mg kg^{-1} but with a minimal impact on groundwater quality.

Extractable Mg increased in the 0- to 15-cm soil depth and SO_4^{2-} increased at both the 15- to 30-cm and 45- to 90-cm soil depths with RWW compared with WW irrigation (Fig. 1). Magnesium and SO_4^{2-} were predominant in RWW (Table 2) and their inputs to the soil were up to 57 and 126 kg ha^{-1} yr^{-1}, respectively.

Mehlich-3 extractable Ca was higher with RWW in the deeper soil layers (>75 cm) (Fig. 2). The high Ca concentrations were probably attributed to the parent material of marine origin common in Riviera series. These bedded parent materials were moved to the subsurface during land preparation, resulting in high Ca at that depth.

Micronutrients

Compared with WW-irrigated soil, extractable B increased ~121% in the RWW-irrigated soil and was higher at the surface but decreased with soil depth (Fig. 2). Boron enrichment and potential phytotoxicity is one of the major concerns regarding RWW irrigation (Toze, 2006; Reboll et al., 2000). In the present study, B concentrations in the soil (Fig. 2) were below the critical level (1.3 mg kg^{-1}) of B toxicity to citrus plants (Aucejo et al., 1997), even after 11 yr of irrigation. Reboll et al. (2000) observed a similar trend where B concentration increased by 0.4 mg kg^{-1} after 3 yr of irrigation with RWW. Boron increased from 0.32 to 0.59 mg kg^{-1} in the surface and 0.18 to 0.45 mg kg^{-1} in the subsurface soil due to RWW irrigation. In the worst-case scenario, if soil extractable B increased at a mean annual rate of 0.025 mg kg^{-1} yr^{-1} [(0.45–0.18)/11 = 0.0245] at current RWW irrigation rates, it will take 52 yr (1.3/0.025 = 52) to reach the critical level of 1.3 mg B kg^{-1} soil. In the same area of study, Pereira et al. (2011b) found higher B concentrations on citrus leaves irrigated with RWW, but no toxicity symptoms were observed in field. Therefore, a B phytotoxicity problem is not likely to occur until RWW irrigation is extended for >50 yr. Qian and Mecham (2005) also reported that B accumulated in the soil, but no phytotoxicity occurred after 33 yr of irrigation with RWW.

Table 3. Mean concentration of total recoverable metals (USEPA 3050B) in surface soil (0–15 cm) irrigated with reclaimed wastewater (RWW) or well water (WW). Common concentration ranges of metals in soils.

Elements	RWW mean	WW mean	Common concentration ranges of metals in soil
	mg kg^{-1}		mg kg^{-1}
Co*	1.4	0.4	5.5–29.9†
Cu*	298	236	20–30‡
Mn*	89	50	10–9000‡
Mo*	1.2	0.4	0.1–7‡
Ni*	4.1	1.7	0.2–450‡
Zn*	63	39	10–300‡
Cd^{ns}§	0.3	0.3	0.06–1.1‡
Cr^{ns}	8	5.1	2–1100‡
Pb*	20	10	0.69–42‡

* Significant difference at $P \leq 0.05$ of total recoverable metals concentration between soil irrigated with RWW and WW according to the *t* test.

† Kabata-Pendias and Mukherjee (2007).

‡ Chen et al. (1999).

§ ns = no significant difference based on *t* test.

Chloride concentration was increased by ~260% in surface soil and consistent along the soil profile. The concentration of Cl in the RWW-irrigated soil (10.1 mg kg^{-1}) (Fig. 2) was less than the range of 20 to 900 mg kg^{-1}, commonly found in soils (Heckman, 2006); therefore, the risk of Cl phytotoxicity is minimal, even though it is the predominant micronutrient in RWW. After 12 mo of irrigation with RWW, Pedrero and Alarcón (2009) reported >402 mg Cl kg^{-1} in the soil. However, no toxic symptoms were observed on citrus leaves.

Extractable Fe, Mn, Zn, Ni, Cu, and Co in the soils were greater with RWW than WW irrigation plots (Fig. 2). Major enrichment of these elements occurred at the 15- to 30- and 30- to 45-cm soil layers. According to the ISP analysis of RWW, these elements were mostly found as free ionic species, ~83% as Fe^{2+}, ~65% as Mn^{2+}, ~53% as Zn^{2+}, ~67% as Ni^{2+}, and ~73% as Co^{2+} (Table 2). The free species added through RWW irrigation contributed to the increased M-3 extractable concentrations of these elements in soil. Consequently, RWW also increased the total recoverable concentrations of these nutrients, compared with the WW-treated plots (Table 3). Extractable Mo was below the detection limit, regardless of water sources, but its recoverable concentration increased by 200% (Table 3).

Although irrigation with RWW supplemented a significant amount of Co, Mn, Mo, Ni, and Zn, their total recoverable concentrations in the irrigated soil were within the common concentration range found in soil, according to Chen et al. (1999) and Kabata-Pendias and Mukherjee (2007). Therefore,

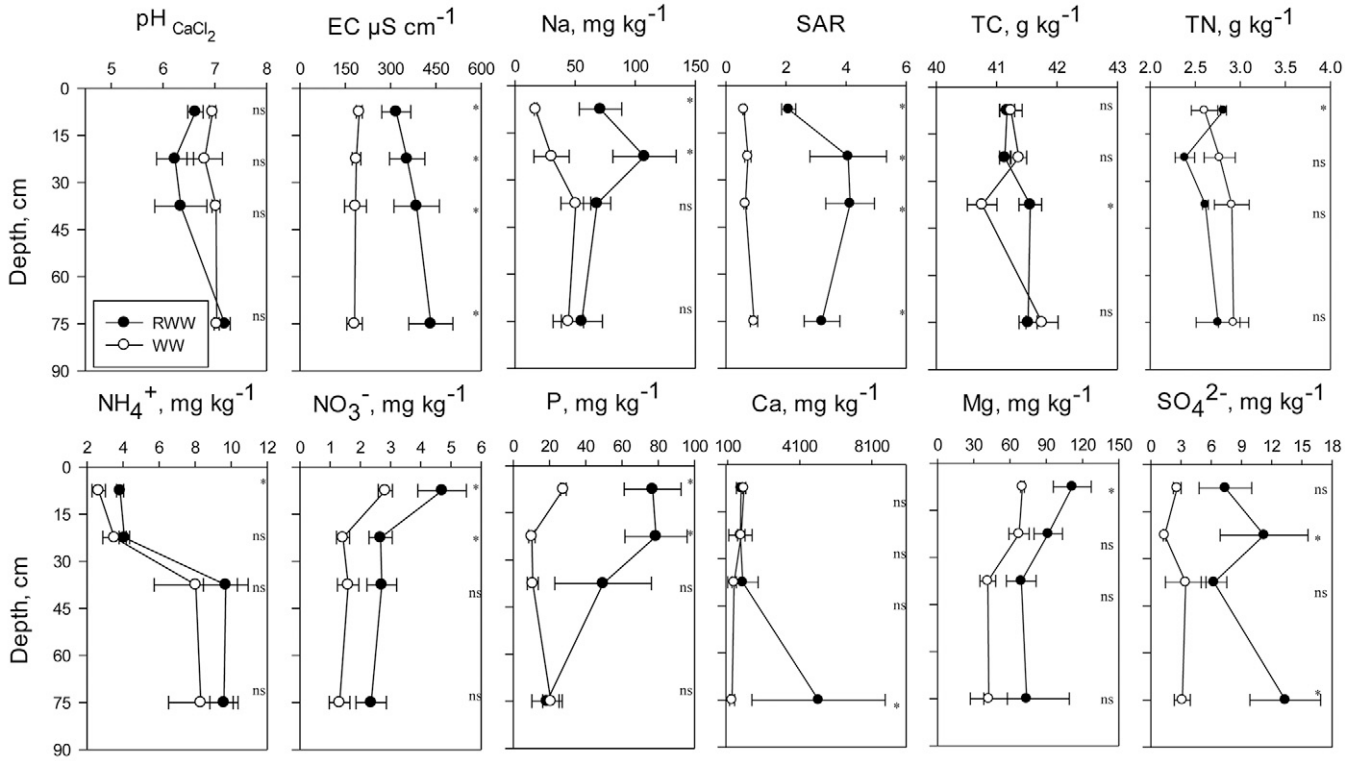

Fig. 1. Mean (n = 20) values of pHCaCl$_2$, electrical conductivity (EC), Na, sodium adsorption ratio (SAR), total carbon (TC), total nitrogen (TN), and macronutrients (NO_4^+, NO_3^-, P, Ca, Mg, and SO_4^{2-}) along the soil profile irrigated with reclaimed wastewater (RWW) and well water (WW) at different depths. The Na and SO_4^{-2} extracted by saturated paste; NO_3^- and NH_4^+ extracted with KCl, 2 mol L^{-1}; P, Ca, and Mg extracted with Mehlich-3 solution. * indicates significant difference $P \leq 0.05$ according to the t test; ns indicates no significant difference to the t test.

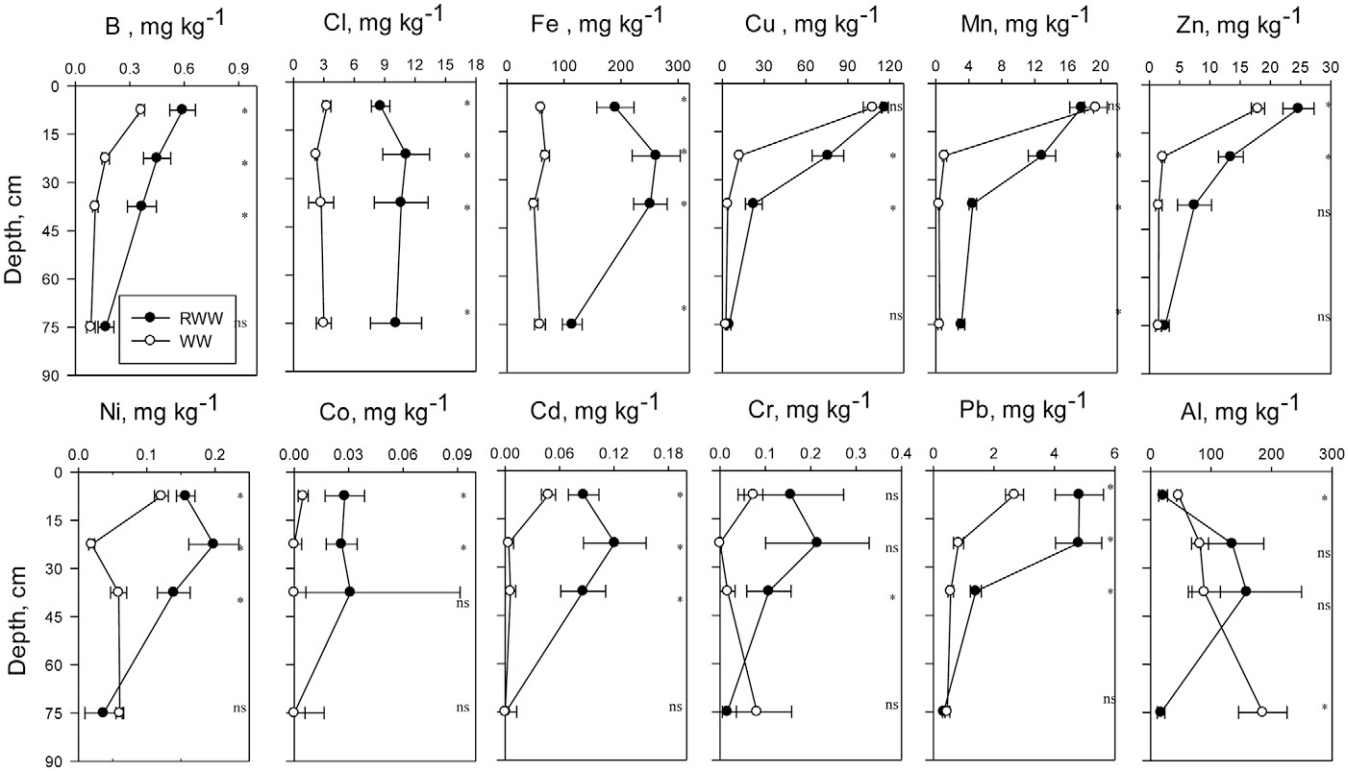

Fig. 2. Mean (n = 20) values of soil micronutrients (C, Cl, Fe, Cu, Mn, Zn, Ni, and Co) and nonessential elements (Cd, Cr, Pb, and Al) along the soil profile irrigated with reclaimed wastewater (RWW) and well water (WW) at different depths. B extracted with hot water; Cl extracted with saturated paste; Fe, Cu, Mn, Zn, Ni, Co, Cd, Cr, Pb, and Al extracted with Mehlich-3 solution. * indicates significant difference at $P \leq 0.05$ according to the t test; ns indicates no significant difference to the t test.

RWW irrigation appears beneficial to citrus production in soils with poor contents of those micronutrients.

Independent of irrigation water sources, total and M-3-extractable concentrations of Cu in the soil was high compared with the common concentration range found in soils (Table 3, Fig. 2). The enrichment of Cu and also Zn in the citrus soil is likely attributed to the spraying of chemicals. According to He et al. (2005), chemical sprays can supplement 1 to 2 kg Cu ha^{-1} yr^{-1} and 5 to 9 kg Zn ha^{-1} yr^{-1} to the citrus grove. Considering the annual irrigation rate and concentrations of Cu and Zn in RWW, the RWW irrigation annually adds only ~0.04 kg Cu ha^{-1} and ~0.30 kg Zn ha^{-1} to the soils. The input from RWW should be minimal, compared with the annual application of Cu- and/or Zn-containing fungicides and fertilizers. Nevertheless, it may be necessary to monitor Cu and Zn availability in the soils with long-term RWW irrigation, particularly under citrus production due to multiple pollution sources.

Unexpectedly, fruit yield between RWW (228 ± 57 kg $tree^{-1}$) and WW (243 ± 115 kg $tree^{-1}$) blocks from 1997–1998 to 2007–2008 were similar. Although significant ($P < 0.05$), effects of RWW on some soil variables were observed. These changes may not be enough to raise citrus yield. Generally, the soil fertility of both RWW and WW blocks did not show any limiting factors for citrus production.

Nonessential Elements

Reclaimed wastewater irrigation increased extractable concentration (Fig. 2 and Table 3) of Cd, Cr, and Pb, and also the total recoverable concentration of Pb, compared with WW irrigation. The higher concentration of these elements in soil irrigated with RWW is attributed to their relatively higher concentrations in RWW compared with WW (Table 2).

Although Pb total recoverable concentration increased in soil after 11 yr of irrigation with RWW, it was within the common concentration range found in Florida soils (Table 3) and still below the soil cleanup target levels (SCTLs, 400 mg Pb kg^{-1}) of Florida Department of Environmental Protection (FDEP, 2005). The effect of RWW irrigation on the concentration of nonessential elements in soil is inconsistent in the literature. Rusan et al. (2007) did not report any change in Cd and Pb concentrations in the soil profile after 10 yr of irrigation with RWW, whereas van Oort et al. (2008) observed significant accumulation of Cd in soil after long-term irrigation of RWW.

Based on the average concentrations of Cd, Cr, and Pb in RWW (Table 2), the annual rate of irrigation (ARI) at 19,420 L $tree^{-1}$ yr^{-1} and the plant population (PP) of 250 trees ha^{-1}, the total annual pollutant load for each metal (APLR) is estimated as: APLR = (element concentration in RWW) × ARI × PP. The calculated APLR for Cd, Cr, and Pb is ~5 g ha^{-1} yr^{-1}, ~77 g ha^{-1} yr^{-1}, and ~208 g ha^{-1} yr^{-1}, respectively. These quantities are negligible compared with those standards (1900 g ha^{-1} yr^{-1}, 154,000 g ha^{-1} yr^{-1}, and 15,000 g ha^{-1} yr^{-1}, respectively, for Cd, Cr, and Pb) for biosolids application (USEPA, 1995).

Conclusions

Most nutrients in the studied RWW are present as free ions and readily available (~53–99%), whereas more than about 80% of Cu, Cr, Pb, and Al in RWW are complexed with CO_3^-, OH^-, and/or dissolved organic matter with reduced bioavailability.

The RWW irrigation generally increased total recoverable and extractable amounts of nutrients, except for Fe in the soil compared with the WW treatment, but no difference in citrus yields was measured. Reclaimed wastewater irrigation tended to increase soil sodicity and salinity, as well as B accumulation in the soil, but it unlikely results in any significant salinization/sodification of the soil or B toxicity to the plant unless the RWW irrigation is extended beyond 40 to 50 yr.

Long-term irrigation with RWW potentially promotes the accumulation of Cd, Cr, and Pb in soil, but the risk of soil pollution with these elements is minimal because of their low concentrations in RWW.

Acknowledgments

This work was, in part, supported by a fellowship from FAPESP (São Paulo Research Foundation) awarded to B.F.F. Pereira (process# 06/56419-6) and the University of Florida. The authors would like to thank Douglas J. Banks for his assistance with the sampling and ICP–OES analysis of soil, plant, and water samples, and Yuangen Yang, Qin Lu, and Jinghua Fan for their assistance with chemical analyses. We would like to also thank Jamie Gamiotea for providing the analytical data of RWW.

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