Science of the Total Environment 739 (2020) 139756

Contents lists available at ScienceDirect

Science of the Total Environment

journal homepage: www.elsevier.com/locate/scitotenv

Review

Reclaimed wastewater as a viable water source for agricultural irrigation: A review of food crop growth inhibition and promotion in the context of environmental change

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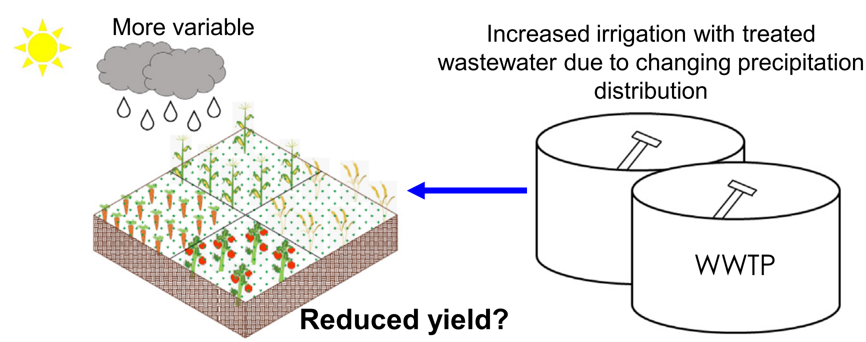
HIGHLIGHTS

- A shift from precipitative to irrigation watering due to climate change is expected.
- The impact of treated wastewater constituents on food crop growth was assessed.
- Nanoparticles and xenobiotics do not impact growth at typical concentrations.
- Irrigation management to reduce salt accumulation may be the most impactful.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 25 March 2020
Received in revised form 24 May 2020
Accepted 25 May 2020
Available online 01 June 2020

Editor: Jay Gan

Keywords:
Crop yield
Pharmaceuticals and personal care products
Soil chemistry
Nanomaterials
Phytotoxicity
Climate change

ABSTRACT

The geographical and temporal distribution of precipitation has and is continuing to change with changing climate. Shifting precipitation will likely require adaptations to irrigation strategies, and because 35% of rainfed and 60% of irrigated agriculture is within 20 km of a wastewater treatment plant, we expect that the use of treated wastewater (e.g., reclaimed wastewater) for irrigation will increase. Treated wastewater contains various organic and inorganic substances that may have beneficial (e.g., nitrate) or deleterious (e.g., salt) effects on plants, which may cause a change in global food productivity should a large change to treated wastewater irrigation occur. We reviewed literature focused on food crop growth inhibition or promotion resulting from exposure to xenobiotics, engineered nanoparticles, nitrogen, and phosphorus, metals, and salts. Xenobiotics and engineered nanoparticles, in nearly all instances, were detrimental to crop growth, but only at concentrations much greater than would be currently expected in treated wastewater. However, future changes in wastewater flow and use of these compounds and particles may result in phytotoxicity, particularly for xenobiotics, as some are present in wastewater at concentrations within approximately an order of magnitude of concentrations which caused growth inhibition. The availability of nutrients present in treated wastewater provided the greatest overall benefit, but may be surpassed by the detrimental impact of salt in scenarios where either high concentrations of salt are directly deleterious to plant development (rare) or in scenarios where soils are poorly managed, resulting in soil salt accumulation.

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https://doi.org/10.1016/j.scitotenv.2020.139756
0048-9697/

Contents

1. Introduction

A large fraction (69%) of global water withdrawals are used for agriculture (FAO, 2016) and the world is shifting towards irrigation rather than relying on precipitation for agricultural production, especially as the climate is becoming drier and more variable. Between 1961 and 2003, the amount of irrigated land approximately doubled, while the proportion of agricultural lands watered with precipitation decreased (Molden et al., 2007). Simultaneously, changes in spatiotemporal distribution of precipitation have already reduced global maize and wheat yields by 3.8% and 5.5%, respectively, between 1980 and 2008 (Lobell et al., 2011). Without change to current irrigation distribution, maize, wheat, and barley yields are expected to change by −14%, 1.6% (a slight increase due to increased CO_2 availability), and −1.8%, respectively, by 2030 (Tebaldi and Lobell, 2008). We expect that declining productivity of some crops due to reduced precipitation will further drive increases in irrigation over rainfed cultivation.

Re-purposing of wastewater treatment systems to produce water suitable for irrigated agriculture could be both cost-effective and sustainable because 35% of rainfed and 60% of irrigated croplands across the globe are near wastewater treatment plants (i.e., either urban or peri-urban, within 20 km of an urban area) (Thebo et al., 2014). Estimates of current direct wastewater irrigation and projections of future use are few and specific to certain regions (no global estimates available) (Jeroen et al., 2004; Scott et al., 2004). However, one recent study showed that 65% of irrigated croplands downstream from urban areas use urban wastewater for irrigation, de facto, based on their catchment wastewater flows (Thebo et al., 2017). Israel has been a leader in this field of irrigation with reclaimed wastewater, accounting for greater than ~40% of agricultural water flows being derived from reclaimed wastewater (Avgar, 2018).

We postulate that agricultural irrigation with wastewater will increase globally, replacing decreasing and more variable precipitation, due to urban proximity (i.e., wastewater availability) and the ever-increasing need to increase food production as the global population increases (Butt et al., 2005; Carleton and Hsiang, 2016; Haim et al., 2008). However, treated wastewater contains constituents that are known to inhibit plant growth, including salt and heavy metals, which may decrease yields. Treated wastewater also contains constituents that can promote growth, including nitrogen (N) and phosphorus (P) and other beneficial nutrients. The objective of this publication is to review the relevant constituents of wastewater that impact plant growth in the context of environmental change and increasing wastewater irrigation. This review is intended to be encompassing of broad facets of the impact of treated wastewater on crop growth, but primarily focuses on xenobiotics and engineered nanoparticles because an abundance of literature already exists related to nutrients, salt, and metals.

2. Xenobiotics' effects on crop growth

Various anthropogenic chemicals (referred to from the crop's perspective as xenobiotics) are present in wastewater (Ferrer and Thurman, 2012; Hanigan et al., 2015). Some xenobiotics are released to wastewater directly, such as hydrocarbons from industry, or *per*- and polyfluorinated chemicals from textile washing (Schellenberger et al., 2019). Others, such as pharmaceuticals, are released in human urine or feces. Quantities of pharmaceutical releases to wastewater vary widely between compounds and populations, but current data generally indicate that a large proportion of consumed pharmaceuticals are released to domestic wastewater. For example, two studies of the metabolism of 42 and 212 pharmaceuticals indicate that humans excrete, 67 ± 34% of the pharmaceutical mass ingested without alteration, while the remainder is partially metabolized (Lienert et al., 2007a; Lienert et al., 2007b).

Removal of xenobiotics by conventional wastewater treatment plants occurs through sorption to the biosolids, chemical oxidation by added disinfectants, and/or through abiotic/biotic reactions. The degree of removal of xenobiotics in wastewater treatment plants is highly variable and depends on the compound, treatment processes employed, and oxidation-reduction conditions in those processes. Consequently, xenobiotic concentrations in wastewater effluents range from below detection limits (usually in the low ng/L range) to 100s of $\mu g\ L^{-1}$ (Dickenson et al., 2011; Oppenheimer et al., 2011), and surface waters that receive treated effluents have concentrations from below detection limits up to 10s of $\mu g\ L^{-1}$ (Dickenson et al., 2011; Ferrer and Thurman, 2012; Oppenheimer et al., 2011). Advanced treatment technologies (e.g., activated carbon, membranes, and advanced oxidation) are effective at removing some xenobiotics to below detection limits, but are expensive and energy intensive, currently limiting their broad application (Ganiyu et al., 2015; Homem and Santos, 2011; Roback et al., 2018).

2.1. Xenobiotic fate in agricultural systems

Irrigation with treated wastewater exposes crops to xenobiotics. Translocation and accumulation in crop compartments are primarily affected by xenobiotic physicochemical properties and soil sorption. Published literature indicates that xenobiotics primarily accumulate in the leaves and roots, and to a lesser extent in fruits, shoots, and stems. However, there is a lack of xenobiotic crop uptake data from field-scale agricultural systems, likely due to several factors such as a lack of well-controlled experimental fields (Al Nasir and Batarseh, 2008; Calderón-Preciado et al., 2012; Eggen et al., 2011; Mathews et al., 2014; Shenker et al., 2011; Tanoue et al., 2012; Wu et al., 2010; Wu et al., 2014). Here we briefly review the factors that influence plant uptake of xenobiotics.

Xenobiotics present in irrigated water first interact with soil. Increased soil sorption leads to decreased plant availability and thus soil sorption is an important factor in determining plant exposure to xenobiotics. Soil sorption primarily depends on pH, ionizable functional groups in the soil (i.e., soil organic matter and minerals such as clays), and ionizable functional groups of the xenobiotic compound. pH in the range of 4 to 8 tends to produce the greatest sorption potential, particularly at the lower end of this range, and overlaps significantly with environmental soil conditions in agricultural systems (Borgman and Chefetz, 2013; Gu et al., 2007; Kodešová et al., 2015; Kulshrestha et al., 2004; Kurwadkar et al., 2007; Park and Huwe, 2016; Zhang et al., 2014). Cation exchange is the dominant mechanism in acidic soils

Table 1
Plant uptake of xenobiotics from selected studies conducted in the field irrigated with treated wastewater. pKa were calculated with Marvin (Chemicalize.org 2019).

Compound (pK_a)	Concentration in irrigation water	Concentration in plant tissue (ng g^{-1} dry mass)
Bezafibrate (3.8)	1.2 μg L^{-1} (Malchi et al. 2014)	Carrot leaves (3–6), roots (2–6)
Caffeine (NA)	1.6 μg L^{-1} (Malchi et al. 2014)	Carrot leaves (4–8)
	0.25 μg L^{-1} (spiked) (Wu et al. 2014)	Sweet potato leaves (6), roots (1–2)
		Mature carrot root (0.4)
		Celery stem (0.2)
Carbamazepine (NA)	1.7 μg L^{-1} (Riemenschneider et al. 2016)	Arugula leaves (61), roots (38), shoots (8)
		Cabbage fruit (10), leaves (79), roots (61)
		Carrot leaves (61), roots (14)
		Eggplant fruit (32), leaves (78), roots (193), shoots (14)
		Parsley leaves (91), roots (41)
		Lettuce leaves (216), roots (27)
		Pepper fruit (8), roots (40), shoots (30)
		Potato leaves (173), roots (77), shoots (60)
		Tomato fruit (5), roots (27), shoots (41)
		Zucchini fruit (7), leaves (42), roots (69), shoots (9)
	0.25 μg L^{-1} (spiked) (Wu et al. 2014)	Mature cucumber fruit (0.02)
		Premature cabbage leaf (0.04)
		Premature celery stem (0.01)
		Premature and mature lettuce leaf (0.02–0.04)
		Premature and mature spinach leaf (0.01)
	1.4 μg L^{-1} (Malchi et al. 2014)	Carrot leaves (13—22), roots (6–8)
		Sweet potato leaves (3–5), roots (1)
Epoxy-carbamazepine (NA)	0.5 μg L^{-1} (Riemenschneider et al. 2016)	Arugula leaves (34), roots (22), shoots (7)
		Cabbage fruits (5), leaves (19), roots (13)
		Carrot leaves (54), roots (8)
		Eggplant fruits (12), leaves (45), roots (22), shoots (15)
		Lettuce leaves (89), roots (10)
		Parsley leaves (21), roots (25)
		Pepper roots (4)
		Potato leaves (138), roots (14), shoots (5)
		Tomato roots (3), shoots (12)
	1.4 μg L^{-1}as carbamazepine prior to plant metabolization (Malchi et al. 2014)	Zucchini fruits (7), leaves (26), roots (44), shoots (6)
		Sweet potato leaves (15–66), roots (0.1–3)
		Carrot leaves (15–66), roots (0.1–3)
Carbamazepine-dihydroxide (9.2)	6.4 μg L^{-1} (Riemenschneider et al. 2016)	Carrot leaves (20), roots (7)
		Lettuce leaves (42), roots (19)
		Parsley leaves (11), roots (25)
		Potato leaves (14), roots (5), shoots (3)
	1.4 μg L^{-1} as carbamazepine prior to plant metabolization (Malchi et al. 2014)	Carrot leaves (0.3–3)
		Sweet potato leaves (0.3–3)
Ciprofloxacin (5.6, 8.8)	0.3 μg L^{-1} (Riemenschneider et al. 2016)	Cabbage fruits (7)
		Carrot roots (12)
Clofibric acid (3.4)	0.9 μg L^{-1} (Malchi et al. 2014)	Carrot leaves (0.4–2), roots (<0.8)
		Sweet potato leaves (0.4–2), roots (<0.8)
Diethyltoluamide (NA)	0.25 μg L^{-1} spiked (Wu et al. 2014)	Premature carrot root (3)
Dilantin (8.5)	0.25 μg L^{-1} spiked (Wu et al. 2014)	Mature lettuce leaf (0.1)
		Spinach leaf (0.1)
Gabapentin (9.9, 4.6)	1.7 μg L^{-1} (Riemenschneider et al. 2016)	Arugula leaves (36)
		Carrot roots (10)
		Parsley leaves (22), roots (29)
		Potato leaves (32)
Gemfibrozil (4.4)	0.7 μg L^{-1} (Malchi et al. 2014)	Sweet potato leaves (0.1–0.5)
	0.25 μg L^{-1} spiked (Wu et al. 2014)	Not detected in any edible plant tissue
Lamotrigine (5.9)	1.5 μg L^{-1} (Malchi et al. 2014)	Carrot leaves (6–19), roots (3–8)
Metoprolol (9.7)	0.7 μg L^{-1} (Malchi et al. 2014)	Sweet potato leaves (0.4–0.5)
Primidone (11.5)	0.25 μg L^{-1} spiked (Wu et al. 2014)	Cabbage leaf (0.3)
		Lettuce leaf (0.1)
		Premature celery stem (0.5)
Sildenafil (11.1, 6.0)	0.5 μg L^{-1} (Malchi et al. 2014)	Carrot leaves and roots (0.2–2)
		Sweet potato leaves (0.5–1)
Sulfamethoxazole (6.2)	0.03–0.06 ng/L (Christou et al. 2017)	Tomato fruits (5)
	0.8 μg L^{-1} (Malchi et al. 2014)	Carrot roots (0.1–0.2)
		Sweet potato roots (0.1–0.2)
	0.25 μg L^{-1} spiked (Wu et al. 2014)	Not detected in any edible plant tissue
Sulfapyridine (6.2)	0.6 μg L^{-1} (Malchi et al. 2014)	Carrot leaves and roots (<0.5)
Trimethoprim (7.2)	0.02–0.07 ng/ L (Christou et al. 2017)	Tomato fruits (3)
	0.25 μg L^{-1} spiked (Wu et al. 2014)	Not detected in any edible plant tissue

with low soil organic matter and leads to increased sorption of protonated xenobiotics onto silicates with pH independent negative charge (Ding et al., 2016; Gao and Pedersen, 2005; Jones et al., 2005; Kodešová et al., 2015; Kulshrestha et al., 2004; Kurwadkar et al., 2007; Park and Huwe, 2016; Wegst-Uhrich et al., 2014). Sorption through this mechanism is decreased in soils closer to neutral pH due to a lack of xenobiotic ionization.

Increasing soil organic matter (i.e., organic carbon) content results in reduced xenobiotic mobility, although this is highly dependent on soil pH and the xenobiotic pK_a. Xenobiotics that are negatively charged at the soil pH tend to be most mobile because they do not interact with negatively charged soil organic matter and do not sorb to silicates (Chefetz et al., 2008; Revitt et al., 2015; Zhang et al., 2014). However, ionization also results in electrostatic repulsion by cells in the roots of many crops and thus soil mobility does not necessarily indicate plant uptake (Carter et al., 2014; Edgington, 1981; Goldstein et al., 2014; Tanoue et al., 2012).

Biodegradation of xenobiotics also affects plant uptake; prior exposure of soil to xenobiotics contained in agricultural irrigation water derived from wastewater can result in priming the soil microorganisms leading to greater rates of biodegradation (Topp et al., 2016; Xu et al., 2009; Yang et al., 2009). However, some transformation products are formed from a mixture of physical, chemical, and biological processes and can be more toxic or stable than the parent compound. Most studies do not quantify transformation products and thus it is difficult to separate effects from parent vs. derived compounds (Dévier et al., 2011; Koba et al., 2017; Ternes et al., 2007). Agricultural systems may also promote product reversion to the parent compound, further obscuring the impact of individual compounds and their degradation products (Fu et al., 2018; Qu et al., 2013).

Selected recent data of crop uptake of xenobiotics are included in Table 1. Because the goal of this manuscript is not to review uptake of xenobiotics, but growth inhibition, this table only includes recent data from field experiments, and serves to show that some xenobiotics are translocated into the plant when irrigated with wastewater. We only include field studies here for brevity and because field and hydroponic experiments do not always agree due to the artificial conditions employed in hydroponic studies compared to field studies as they relate to soil organic matter and pH (Wu et al., 2010; Wu et al., 2014). For a more complete discussion of uptake and human health risks, we refer to literature solely focused on this subject (Al-Farsi et al., 2017; Carvalho et al., 2014; Christou et al., 2018; Christou et al., 2019; Colon and Toor, 2016; Du and Liu, 2012; Jjemba, 2002b; Madikizela et al., 2018; Pan and Chu, 2017; Wu et al., 2015).

2.2. Crop yield effects from xenobiotic exposure

Various effects have been observed when plants were exposed to xenobiotics (Bartha et al. 2010; Christou et al. 2016). Specific exposure effects are thought to generally manifest in reactive oxygen species and thus membrane lipid peroxidation (Christou et al. 2016; Sun et al. 2018). Our discussion is focused on growth inhibition because 1) it is easily measured, resulting in a large data set, 2) growth is representative of many endpoints because many effects also manifest in changes to growth rate, and 3) it is the most relevant endpoint for a discussion of global food security in the context of environmental change.

We reviewed 83 publications and discarded data from 1) veterinary antibiotics or other compounds that we would not expect to be present in municipal treated wastewater, 2) non-crop plants such as duckweed because they are not relevant to human consumption, 3) experiments that did not include a growth endpoint that was comparable to a control, 4) experiments in which xenobiotics were applied simultaneously because it is impossible to determine which particular xenobiotic caused the effect, and 5) experiments that applied the xenobiotics indirectly through application of manure. Notably, the number of studies that applied multiple xenobiotics simultaneously is limited (n = 3) and an area ripe for further research based on the known agonistic and antagonistic mechanisms of toxicity (Hurtado et al. 2017; Marsoni et al. 2014; Sun et al. 2018). We did not remove select forage crops (e.g., alfalfa) as a significant declination in productivity would cause secondary effects on meat production, resulting in increased global food stress. Lowest observable adverse effect concentrations (LOAEC) of xenobiotics that caused growth inhibition were gathered from the remaining 32 publications and are shown in Fig. 1 (An et al. 2009a; An et al. 2009b; Azanu et al. 2016; Batchelder 1981, 1982; Boonsaner and Hawker 2010; Boxall et al. 2006; Calderón-Preciado et al. 2012; Carter et al. 2015; Cheng and Zhou 2002; D'Abrosca et al. 2008; Eggen et al. 2011; Hammad et al. 2018; Hillis et al. 2011; Jin et al. 2009; Jjemba 2002a; Jones-Lepp et al. 2010; Khan et al. 2018; Kong et al. 2007; Li et al. 2011; Liu et al. 2009; Michelini et al. 2012; Minden et al. 2017; Moore and Kröger 2010; Opriş et al. 2013; Osma et al. 2018; Pan and Chu 2016; Pino et al. 2016; Schmidt and Redshaw 2015; Shenker et al. 2011; Xie et al. 2010; Yang et al. 2010). We aggregated LOAEC rather than lethal or effective concentrations (LC_{50} or EC_{50}) because many publications did not report these statistical extrapolations, and in many cases the data are too coarse (i.e., lacking sufficient data between applied concentrations that caused effects) to support such an extrapolation. LOAECs were taken directly from raw data or interpolated from plots as the lowest applied concentration in which an effect was observed. No interpolation between applied concentrations was conducted. The complete data set is included as Table SI-1. The same data plotted as LOAEC vs xenobiotic is shown in Fig. SI-1, but we urge caution in the interpretation of this figure because of the loss in statistical power by fragmenting the dataset (i.e., each bar represents limited data).

Aqueous LOAECs ranged from 10^{-7} g/L water (ethinyl estradiol exposure reduced lettuce germination rate by 8% (D'Abrosca et al. 2008)) to 1 g/L (acetaminophen reduced grain weight of maize ~36% (Hammad et al. 2018)). Soil LOAECs ranged from 10^{-3} g/kg soil [carrot and lettuce exposed to oxytetracycline had a slight decrease in plant weight (Boxall et al. 2006) and zucchini exposed to carbamazepine had a ~45% decrease in root dry weight (Carter et al. 2015)] to 10 g/kg [soybean exposed to quinacrine, decrease also not quantified (Jjemba 2002a)]. Several xenobiotics did not affect specific crops even at very high exposure concentrations. For example, no growth effects were observed for cucumber exposed to 0.3 g/L sulfamethoxazole (aqueous), or 0.3 g/kg (soil) tetracycline (Liu et al. 2009). Although maize also appears to be tolerant to xenobiotic exposure, only three studies met our requirements for data relevance, with exposure to a total of only four compounds, and one study found no growth inhibition when exposed to 0.16 g/L chlortetracycline or oxytetracycline (Batchelder 1982). Given the global reliance on maize, a greater number of studies should be conducted before a conclusion is reached.

Exposure to xenobiotics nearly always resulted in a reduction in biomass compared to control samples, and rarely resulted in an increase, which may be concerning should concentrations of xenobiotics in treated wastewater effluent increase substantially. Such a scenario is concerning given that wastewater flows are likely to continue to decrease in response to decreased freshwater availability as a result of shifting climate, along with increasing xenobiotic use. Experiments that expose crops to multiple xenobiotics simultaneously are scarce, and thus the agonistic or antagonistic effects of a wastewater profile of xenobiotics are unknown and may exacerbate this issue. One study evaluating the agonistic effects of simultaneously applied xenobiotics found 13% reduced lettuce stem width and 27% increased stem height after exposure to 5×10^{-8} g/L of benzophenone, bisphenol A, butylated hydroxytoluene, caffeine, carbamazepine, methylparaben, 4-octylphenol, phenazone, triclosan, and tris(2-chloroethyl) phosphate (Hurtado et al. 2017). Another study found no growth effects on arugula when concentrations of the individual compounds was up to 2.5×10^{-5} g/L (Marsoni et al. 2014). The difference in these two studies may be that the former utilized hydroponic exposure (lack of soil and soil organic matter sorption) while the latter used a peat-based soil in which

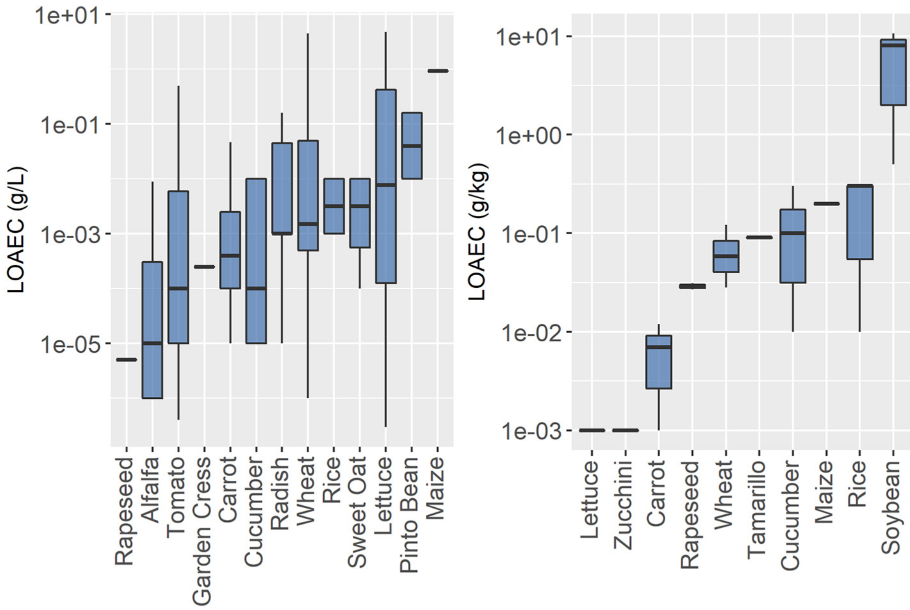


Fig. 1. Boxplot of lowest observable adverse effect concentrations for crops exposed to various individual xenobiotics from 25 publications using either hydroponics or watered soil (left), or 7 publications using mixed soil (right) as the exposure medium. Only publications with growth endpoints studying food crops or selected forage crops (e.g., alfalfa) exposed to municipal wastewater relevant xenobiotics are included. Experiments that did not result in statistically measurable phytotoxicity or exposed the plant to multiple xenobiotics simultaneously are not included in this figure but are shown in the raw data in the SI. Individual xenobiotics are typically present in treated wastewater at $<10^{-5}$ g/L treated water or $<10^{-6}$ g/kg soil that has been irrigated with treated wastewater.

some compounds may have been immobilized and/or degraded. Wastewater and soil concentrations required to cause an effect were found to be greater than would generally be expected to be currently present in treated wastewater ($<10^{-5}$ g/L) or soil irrigated with wastewater ($<10^{-6}$ g/kg). Regulatory guidance from the United States Environmental Protection Agency (US EPA) well reflects this, and suggests that the risk to crops of xenobiotics contained in reclaimed water used for irrigation is low (U.S. EPA 2012). We note, however, that the concentrations required to affect growth are reasonably low and thus future changes to wastewater flows or xenobiotic use may change this conclusion.

3. Engineered nanoparticles present in treated wastewater and their effect on crop growth

3.1. Occurrence of engineered nanoparticles in wastewater effluent

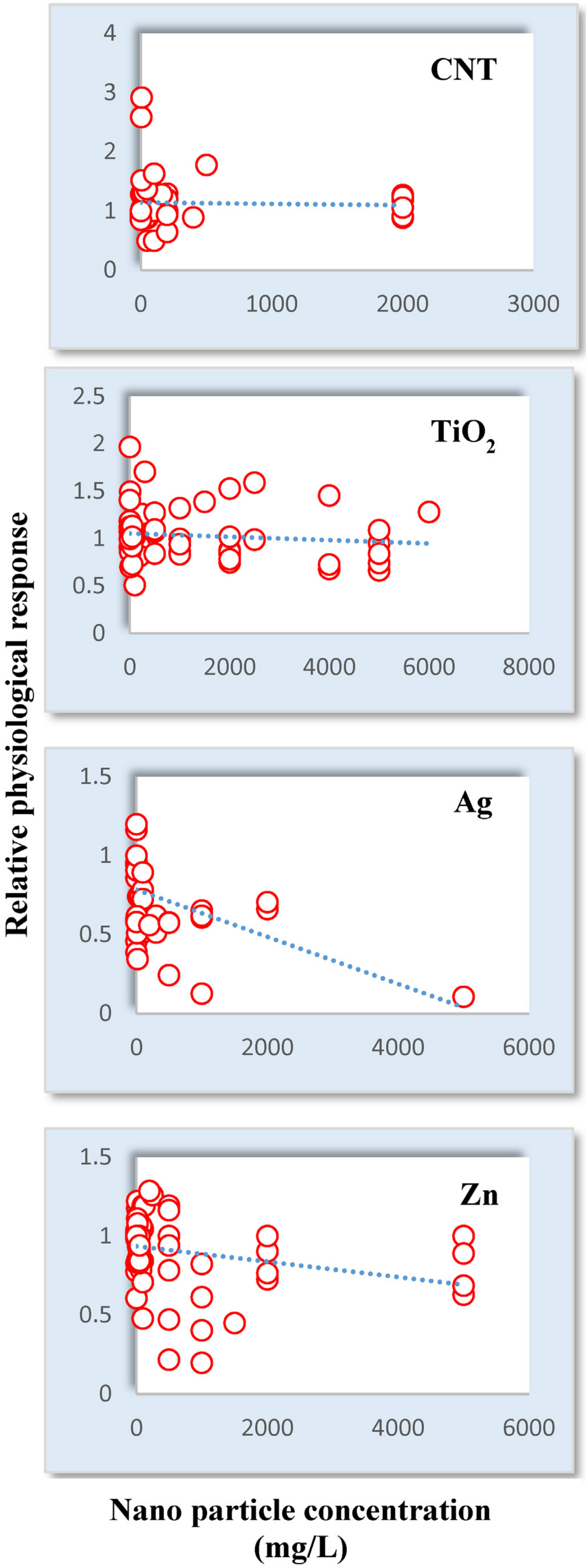
Due to the rapid development and wide application of nanotechnology, a range of engineered nanoparticles (ENPs) make their way into the wastewater system (Badireddy et al. 2012; Gottschalk and Nowack 2011; Jarvie et al. 2009; Keller et al. 2013; Levard et al. 2012; Limbach et al. 2008; Tuoriniemi et al. 2017; Venkatesan et al. 2018). Release of ENPs from consumer products is a major contributor of ENPs in wastewater (Koponen et al. 2011; Mitrano et al. 2015). For instance, silver (Ag)-based ENPs are released from certain clothes, wastewater treatment effluents, personal care products, medical devices, and electronics (Benn and Westerhoff 2008; Cervantes-Aviles et al. 2019). Nano-sized titanium (Ti) dioxide is used in paint, consumer textiles, and other products, and also can be mobilized into domestic wastewater by washing and other use (Kaegi et al. 2008; Kiser et al. 2009; Shi et al. 2016; Weir et al. 2012; Windler et al. 2012).

Modelling studies have been conducted to estimate the release of ENPs into wastewater systems. Keller et al. (2013) estimated that a majority of ENPs would be disposed of in landfills worldwide, and only a small fraction of ENPs would enter natural water systems. Globally, >30,000 tons of zinc- (Zn) based ENPs are produced annually, and 170–2985 tons/year are estimated to enter receiving water bodies. For carbon nanotubes (CNTs), only 33 tons were estimated to end up in water systems, equivalent to approximately 1% of CNTs produced annually worldwide (Keller et al. 2013). Using dynamic probabilistic modelling, Sun et al. (2016) estimated the concentrations of Ti, Ag, and CNT ENPs in wastewater effluents in European Union to be 44.4 μg L^{-1}, 2.65 ng/L, and 8.58 ng/L, respectively.

Limited experimental efforts have been conducted for direct analysis of the fate of ENPs in the wastewater treatment processes. Shi et al. (2016) found 74–85% of Ti-ENPs were removed by activated sludge and Ti-ENPs concentrations were 27–43 μg L^{-1} in the effluent of a wastewater treatment plant in China. Cervantes-Aviles et al. (2019) detected 0.5–13.5 ng/L Ag-ENPs in the effluents of different treatment processes in a wastewater treatment plant in California and determined that 76–96% of spiked Ag-ENP was removed by the secondary and tertiary treatment processes. Another study suggested that eight ENPs were generally amenable to aqueous removal by partitioning to other phases (soil, micelles, and sewage sludge) (Kidd et al. 2018).

3.2. Effects of wastewater associated ENPs on crop growth

Engineered nanoparticles can stimulate or inhibit agricultural crop growth through various mechanisms, including improving disease resistance, altering membrane transport, stimulating the generation of free radicals, and changing the delivery of nutritional elements (Kranjc and Drobne 2019; Su et al. 2019; Vithanage et al. 2017). Previous studies well documented promoting effects of CNTs, especially at low concentration, on germination, plant growth, and development (Alimohammadi et al. 2011; Lahiani et al. 2013a; Villagarcia et al. 2012). The enhancement has been linked to the CNT-derived upregulation of genes related to water channel and cell division (Villagarcia et al. 2012). However, ENPs, especially metallic ENPs, have been shown to result in phytotoxicity, through enhanced generation of reactive oxygen species, which damages the photosynthetic system, as well as phytotoxicity through other mechanisms (Barhoumi et al. 2015; Dewez and Oukarroum 2012; Khan et al. 2017; Rao and Shekhawat 2014). In particular, Ag NPs can penetrate plant cells and affect cell division, leading to cell disintegration, chromatin bridging, metaphase interruption, and other impairment (Kumari et al. 2009).



The plant uptake and toxicity of Ag NPs can also be affected by its speciation and aggregation (Stegemeier et al. 2015).

Many studies have been devoted to analyzing the effects of ENPs on agricultural crop growth, mostly in the perspective of potential application of ENPs in agricultural and food systems as well as their potential implications on agricultural productivity and human health. In Fig. 2 and Tables SI-2 through SI-5, the effects of selected ENPs on agricultural plant growth have been compiled. For our analysis, we selected data from 36 publications: **CNTs** - (Canas et al. 2008; Khodakovskaya et al. 2012; Lahiani et al. 2013b; Lin and Xing 2007; Lin et al. 2009; Mondal et al. 2011; Pandey et al. 2018; Tiwari et al. 2014; Tripathi et al. 2011; Wang et al. 2012) **Ti** - (Ahmed et al. 2019; Boonyanitipong et al. 2011; Castiglione et al. 2011; Daghan 2018; Feizi et al. 2012; Gao et al. 2008; Hong et al. 2005; Jiang et al. 2017; Kibbey and Strevett 2019; Larue et al. 2012; Liu et al. 2019; Raliya et al. 2015; Song et al. 2013; Su et al. 2007) **Ag** - (Abbas et al. 2019; Kaveh et al. 2013; Lee et al. 2012; Liu et al. 2019; Mirzajani et al. 2013; Nair and Chung 2014; Qian et al. 2013; Song et al. 2013; Vannini et al. 2014) **Zn** - (Ahmed et al. 2019; Bandyopadhyay et al. 2015; Boonyanitipong et al. 2011; Dimkpa et al. 2012; Du et al. 2011; Kim et al. 2011; Kouhi et al. 2014, 2015; Rao and Shekhawat 2014; Raskar and Laware 2014; Yoon et al. 2014; Zhang et al. 2015). CNT, Ti, Ag, and Zn ENPs were selected based on their relatively high use and release to the wastewater (Sun et al. 2016). A range of agricultural crops including rice, wheat, maize, tomato, bean, and others were studied. A suite of growth-related physiological responses (e.g., fresh weight/dry weight of shoot/root/fruit, root elongation, seed germination, and other parameters) was aggregated from published studies, and relative response compared to a control without input of ENPs was used for integrative analysis of data. This calculation was used to normalize measurements for different biological endpoint effects of nanoparticles.

Most experiments with CNTs reported positive effects of CNTs on crop growth (e.g., increased fresh biomass, shoot weight, root length and others) even at concentrations as high as 2000 mg/L in hydroponic and soil culture systems. However, negative or harmful effects of metallic ENPs on agricultural crops were observed. The concentration of Ag ENPs was significantly correlated with physiological response (Pearson correlation coefficient, $r = -0.49$, $p < 0.01$) and Zn ENPs ($r = -0.27$, $p = 0.02$), but not with Ti ENPs ($r = -0.10$, $p = 0.31$) or CNTs ($r = -0.037$, $p = 0.78$). In addition, there was significant correlation between growth and concentration of Ag ENPs (Growth $= -1.5 \times 10^{-4}$ Ag $+ 0.80$, $p < 0.01$, where Ag is the concentration (mg/L) of Ag ENPs) and Zn ENPs (Growth $= -4.5 \times 10^{-5}$ Zn $+ 0.92$, $p < 0.01$, Zn is the concentration (mg/L) of Zn ENPs), but not with CNTs or Ti. The inhibitory effects were strongest for Ag ENPs, and the average relative response was 0.77, 0.76, and 0.51 for exposures of Ag ENPs of ≤10, 10–100, and >100 mg/L, respectively, representing an overall inhibitory effect of Ag ENPs at these relatively high concentrations. It should be noted that the concentrations used in these studies far exceeded concentrations present in wastewater. At concentrations currently expected in treated wastewater, growth inhibition from ENP exposure is likely to be negligible for these four ENPs. In terms of risk to the food supply, it is appropriate that US EPA guidance focuses more on bulk dissolved elements than nanomaterials (U.S. EPA 2012).

Despite an abundance of studies, there are several significant knowledge gaps regarding the effects of ENPs in treated wastewater on agricultural crops. In the majority of these publications, no additional experiments were conducted to distinguish effects of the heavy metal compared to the nanoparticle itself, which is an area of ongoing study. Additionally, very few studies have been conducted with ENPs and experimental conditions relevant to wastewater reclamation and reuse

Fig. 2. Relative physiological growth response (e.g., fresh weight/dry weight of shoot/root/fruit, root elongation, seed germination) of 24 agricultural crops vs. the concentration of engineered nanoparticles (ENP) in applied water (mg/L) for crop growth. Raw data is presented in Tables SI-2 to SI-5.

for agriculture (Kouhi et al. 2015). The lowest concentration of ENPs used for crop growth experiments was 10^{-4} and 2×10^{-5} g/L for CNT and Ag ENPs, respectively, while their concentrations in wastewater treatment effluents are estimated to be in the range of 10^{-9} g/L. The lowest concentration of Ti ENPs used was 7×10^{-5} g/L, similar to the estimated concentration in wastewater effluents, but this was not the case for the majority of publications, with concentrations above 10^{-2} g/L. Further, most of existing results are based on short-term exposures and data is rare for long-term or multi-year exposures, which are important for evaluating the effects of ENPs on agricultural crop growth and their implications in agricultural systems. We expect that reduced experimental concentrations will result in limited effects and that increasing duration of exposure will not significantly change uptake and therefore growth. However, reduced wastewater flows and increased ENP use in the future might significantly increase crop exposure, but it is unlikely that concentrations will exceed those in the reviewed literature, which had limited or no impact on various physiological endpoints. Further experimentation is needed to confirm these hypotheses.

Finally, in addition to affecting plant growth directly, ENPs transfer through the food chain. Compared to the effects to plant growth, relatively limited studies have been conducted regarding the fate of ENPs in the agriculture food chain, i.e., transfer from fresh products to humans or to grazing animals and then to humans. A significant data gap exists regarding human exposure to ENPs through agricultural products and the associated risk and implications to human health from increasing the proportion of treated wastewater irrigation.

4. Influence of plant available nutrients present in treated wastewater

Plant available major nutrients, N, P, and K are commonly present in treated wastewater and have the most pronounced effect on soil characteristics and plant growth when irrigated with treated wastewater. The same nutrients can also increase microbial activity involved in carbon metabolism and in general, increase soil microbial metabolism. Often, N and P levels in treated wastewater could be present in concentrations that exceed metabolic needs of the plants grown in the irrigated soils, leading to their potential release into the environment. The degree of treatment and regulations for treated wastewater reuse for irrigation are the determining factors for nutrient releases into the environment. Release of N and P into receiving waters causes eutrophication or algal blooms. More importantly, treated wastewater irrigation could lead to high N inputs into soils, transformation of reduced N forms into nitrate, followed by leaching of the nitrate into groundwater. One study demonstrated that poorly denitrified wastewater containing ~40 mg/L NH_4^+-N resulted in increased NO_3^- concentrations in the underlying aquifer similar to that of agricultural fertilizer use (Kass et al. 2005). However, Candela et al. (2007) found no increase in nitrate in the groundwater as a result of using treated wastewater to irrigate a golf course in an urban area and determined that the application of N from the treated wastewater was lower than the agronomic requirements. Anane et al. (2014) found that reclaimed wastewater was not an influencing factor on groundwater contamination by nitrate, but high cropping density was more likely to cause elevated nitrate levels. Therefore, prolonged irrigation of crops with treated wastewater may not cause groundwater nitrate contamination problems as long as the application rates and agronomic uptake rates are balanced. The overwhelming outcome of treated wastewater irrigation results in beneficial impacts of nutrients in terms of soil microbial activity and plant growth and yields (Jaramillo and Restrepo 2017). Guo et al. (2017) found that increased N and P in treated wastewater irrigation increased abundance of certain microbial groups such as Verrucomicrobia and Gemmatimonadetes. On the other hand, P input from reclaimed wastewater water into irrigated soils has not been reported to be released into the groundwater, but surface waters releases are possible as is the case with agricultural runoff.

5. Changes to soil induced by irrigation with treated wastewater

5.1. Salt and heavy metal accumulation

Several recent reviews on the effects of heavy metals on plants have been written including Chibuike and Obiora (2014), Tangahu et al. (2011), Gjorgieva Ackova (2018), and Singh et al. (2016). For the purposes of this paper we summarize the key points from these reviews and other sources. Plant-available heavy metals are present as soluble components in the soil solution or as compounds that can be solubilized by root exudates (Blaylock and Huang 2000). Plants require specific heavy metals to function optimally, but excessive amounts of metals can become harmful. Excess heavy metals can have both direct and indirect negative effects on plants. Direct effects include inhibition of enzyme activity, metabolic processes, and photosynthesis as well as oxidative stress (Van Assche and Clijsters 1990). Metals may indirectly affect plants when non-essential metals compete with essential metals for cation exchange sites in soils and plants (Taiz and Zeiger 2002). In addition, high heavy metal concentrations may adversely affect beneficial soil microorganisms. As a result, organic matter decomposition may decrease thereby decreasing soil nutrient availability.

The effect of heavy metal toxicity on the plant growth depends on the particular heavy metal as well as their concentration in the soil. For metals that are not required by plants such as Pb, Cd, Hg, and As, adverse effects have been recorded at very low concentrations. For instance, Kibra (2008) observed significant reduction in height and tiller production of rice plants growing on a soil contaminated with 1 mg/kg Hg. Similarly, shoot and root growth in wheat plants was reduced when Cd in the soil solution was as low as 5 mg/L (Ahmad et al. 2012). For metals that are beneficial or essential to plants, low soil concentrations typically improve plant production. However, even for these metals, plant functioning can be damaged at higher concentrations. Several studies have shown that growth, nutrient content, and enzyme activities in tomato, radish and mung bean increased at soil Co concentrations between 0 and 50 mg/kg but decreased at Co concentrations exceeding 100 mg/kg (Jayakumar et al. 2007; Jayakumar et al. 2008; Jayakumar et al. 2013). Similarly, growth and physiology of cluster beans increased at soil solution Zn concentrations up to 25 mg/L but decreased at concentrations above 50 mg/L (Manivasagaperumal et al. 2011). Often, soils are polluted with more than one heavy metal. Ghani (2010) examined the combined effect of Cd, Cr, Co, Mn, and Pb on the growth of maize and observed that the combined effect of two or more heavy metals was typically determined by the most toxic heavy metal. Given that heavy metal concentrations in wastewater effluent are typically limited by discharge limits to much less than the thresholds for toxicity described above, and metals overall are present at <750 μg L^{-1} for Al, Ni, Fe, and Zn, and <100 μg L^{-1} for others (Carletti et al. 2008; Karvelas et al. 2003; Metcalf and Eddy 2013), metals, including heavy metals, likely have limited to no impact on crops irrigated with treated wastewater.

A second concern associated with use of treated wastewater for irrigation is increasing soil salinity as salts contained in the water may accumulate in soils. Build-up of salts in the soil causes chemical and physical changes to the soil that reduce crop yields or can prevent crop production all together. High concentrations of Ca^{2+} and other polyvalent ions reduce the ability of plants to take up water as high salt concentrations decrease the osmotic potential across the crop's roots. Crops must then use resources to correct for that potential difference or suffer from reduced water uptake, resulting in wilting (Munns and Tester 2008). In addition, high salt concentrations can reduce plant growth due to specific-ion toxicities and nutritional imbalances (Läuchli and Epstein 1990; Munns and Tester 2008). Salinity can negatively affect photosynthesis by decreasing CO_2 availability due to diffusion limitations (Flexas et al. 2007) and a reduction in photosynthetic pigments (Ashraf and Harris 2013; Delfine et al. 1999). Salt accumulation in spinach inhibits photosynthesis (Di Martino et al. 1999) primarily by decreasing stomatal and mesophyll conductance to CO_2 (Delfine et al. 1998) and reducing chlorophyll content, which can affect light absorbance (Alvino et al. 2000; Delfine et al. 1999). The presence of high fractions (>15%) of exchangeable sodium (Na^+) relative to bi- or trivalent cations in soil can cause a breakdown of soil structure due to clay dispersion. Dispersed clays lead to reduced infiltration and hydraulic conductivity, thereby limiting root growth, and seedling establishment. Clay dispersion also increases soil erosion and clay swelling (Minhas et al. 2019). An increase in pH is associated with high Na^+ content (alkalization), caused by the presence of HCO_3^- and CO_3^{2-}. The Exchangeable Sodium Percentage, or fraction of the cation exchange complex occupied by Na^+ typically shows a linear relationship with soil pH (Sumner and Naidu 1998). Increasing pH can cause deficiencies and/or toxicities of plant nutrients. Sodium in the soil also competes with Ca^{2+}, K^+, and other cations. As a result, high amounts of Na^+ can cause a reduction in these other cations to reduce their availability to crops. In summary, soils with high levels of exchangeable Na^+ may impact plant growth by deterioration of soil structure, nutrient deficiencies or imbalances, and specific toxicity to Na^+-sensitive plants.

The addition of salts contained in wastewater tends to affect the surface horizons more than the subsurface horizons of the soil, and salt accumulation is often greater as clay content of soils increases (Chen et al. 2013). Palacios-Díaz et al. (2009) observed high electrical conductivity values in a soil when municipal wastewater was used for irrigation. The levels exceeded the threshold of salinity damage of alfalfa and could cause plant mortality. Chen et al. (2013) conducted a study using reclaimed wastewater and found that the average soil salinity plots irrigated with the reclaimed water was 19.2% higher than those irrigated with tap water, but the growth of the grasses in the experiment (Bluegrass and Buffalograss) was not impacted.

As global wastewater flow decreases due to water conservation, increases in salinity, organic matter, and metal concentrations can be expected. However, given that both salts and metals have a known detrimental impact to receiving streams, effluent permitting is expected to drive wastewater treatment processes in controlling their concentrations. However, if effluent concentrations continue to increase, more salt-tolerant crops such as barley, camelina, rye, safflower, sunflower, sugar beets, amaranth, and quinoa may become favored in scenarios where wastewater is applied for irrigation. Given that most crops are sensitive to heavy metals, increasing concentrations are likely to negatively impact crop production.

5.2. Soil microbiome

Treated wastewater irrigation has been shown to have beneficial effects or no effects on soil microbiome in terms of microbial diversity and inter-species connection, and on plant growth in terms of yield compared to groundwater irrigation (Li et al. 2019). The focus here is on how contaminants in treated wastewater affect soil microbiome and in turn, the productivity of soils in terms of crop growth. Engineered nanomaterials including those commonly occurring in treated wastewater have adverse effects on soil microbiota (Liu et al. 2018). The significant broader effects included decreased microbial diversity and shifts in microbial communities, although specific effects on individual microorganisms or genus were minimal. The predominant ENPs that induced these effects were suspected to be TiO_2, ZnO, and Ag particles, but it was difficult to separate effects of ENPs present in the wastewater and dissolved metals.

The intermediate role of rhizosphere interactions of the soil microbiome on root uptake of PPCPs by the plant not been directly studied or reported in the literature. The soil microbiome may play an important role in the bioaccumulation of persistent contaminants (not amenable to biodegradation) such as perfluoroalkyl substances, carbamazepine, and antibiotics in treated wastewater used for irrigation and hence promote the plant uptake at the higher concentrations in the rhizosphere (Li et al. 2013; Pullagurala et al. 2018). There is much discussion and work on biodegradation of xenobiotic input from reclaimed water and sludge into soils, but not on the bioaccumulation by the soil microbiome. If the soil microbiome attenuates leachability of recalcitrant trace contaminants, they become available for plant uptake and hence deserve significant research to understand this phenomenon. This is particularly the case with soils irrigated with treated wastewater because the nutrients present in the water may stimulate microbial growth. The conferring of antibiotic resistance to the soil microbiome by antibiotic resistance genes in treated wastewater used for irrigation is also known. Han et al. (2016) found a significant divergence of soil microbial community structure following reclaimed water irrigation, however, it is not clear if such a resulting microbiome affects plant growth in those soils.

6. Conclusions

We expect that treated wastewater will increasingly contribute to irrigation supplies for food crops. We critically reviewed literature related to the impacts of wastewater constituents, particularly, trace contaminants and ENPs on crop yield and found that:

1) At concentrations much greater than are expected in treated wastewater, xenobiotics generally cause reduced growth in a broad range of food crops.
2) CNTs and Ag, Ti, and Zn ENPs also generally caused declinations in plant growth, but again the impacts were only observed at concentrations much greater than are expected in treated wastewater.
3) The presence of N and P presents one of the greatest benefits of irrigation with treated wastewater over more typical sources and there is an abundance of evidence that indicates that yields are increased and nitrate and P releases to groundwater and surface waters are small or negligible.
4) Salt accumulation likely presents the greatest threat to crop yields and may result in significant decreases in crop yield if soils are poorly managed. Metals were detrimental to growth but only at concentrations greater than present in treated wastewater.
5) Future changes to wastewater flow due to climate change or increasing use of the chemicals or particles may change the conclusion that they cause little or no phytotoxicity, and this is especially apparent for xenobiotics, of which, some are present in wastewater effluent at concentrations within an order of magnitude of their LOAEC.

Together, the reviewed literature suggests that the overall impact of treated wastewater constituents on crop growth is likely to be beneficial as long as salts concentrations in the treated wastewater are well controlled, and the soil managed to limit salt accumulation. Further, should the concentrations of ENPs, metals, salts, or xenobiotics significantly increase, the beneficial impact of N and P present in treated wastewater may be overcome, resulting in a net decrease in yield.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

This research was supported by AFRI grant no. 2017-69007-26309 from the USDA National Institute of Food and Agriculture and partially supported by the National Science Foundation grant no. 1804255 and no. 1808372.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.scitotenv.2020.139756.

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