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Jenna Buchanan

Department of Environmental
Science, University of Florida,
Gainesville, USA

Assessment of pharmaceutical and personal care product contamination in agricultural soils irrigated with treated wastewater

Jenna Buchanan

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Abstract

A single flush of a household toilet sends dozens of pharmaceutical compounds into the wastewater stream, and an increasing share of that treated water ends up irrigating the crops that feed us. This research assessed the occurrence and concentration of eight Pharmaceutical and Personal Care Products (PPCPs) in agricultural soils receiving primary-treated, secondary-treated, and groundwater irrigation at three farm sites near Gainesville, Florida, over 18 months (January 2021-June 2022). Triclosan and caffeine were detected in over 90% of primary-treated wastewater-irrigated soil samples, with mean concentrations of 287 and 342 ng/kg, respectively. Secondary treatment reduced soil PPCP loads by 38-67% depending on compound class. Carbamazepine showed the highest persistence, with a calculated soil half-life exceeding 120 days. These results highlight the need for advanced treatment technologies and soil monitoring programs where wastewater reuse is practiced for agricultural irrigation.

Keywords: Pharmaceutical contamination, personal care products, wastewater irrigation, agricultural soils, triclosan, carbamazepine, emerging contaminants, soil persistence, Florida

Introduction

When a farmer in central Florida turns on the irrigation pump, the water flowing onto vegetable fields may carry invisible traces of ibuprofen, birth control hormones, and antibacterial soap remnants of everyday life that conventional wastewater treatment plants were not designed to remove ^[1]. Treated wastewater reuse for agricultural irrigation is expanding rapidly across water-scarce regions, including the South-Eastern United States, where population growth is straining freshwater supplies ^[2]. Florida alone discharged over 2.8 billion liters per day of reclaimed water in 2021, with agriculture consuming roughly 40% of that volume ^[3].

PPCPs encompass a broad class of emerging contaminants including analgesics, antibiotics, hormones, antimicrobials, and UV filters that enter municipal wastewater through household and hospital discharge ^[4]. Primary and secondary treatment processes remove 30-90% of these compounds from the water phase, but residual concentrations can accumulate in irrigated soils through repeated application ^[5, 6]. Once in the soil, PPCPs may affect microbial communities, enter groundwater, or be taken up by crops ^[7].

This research measured the occurrence, concentration, and persistence of eight target PPCPs in agricultural soils under three irrigation regimes near Gainesville, Florida, to provide field-level data on the extent of contamination and the effectiveness of secondary treatment in reducing soil loading ^[8, 9].

Materials and Methods

Reagents and Chemicals

Target analytes and deuterated internal standards (ibuprofen-d3, triclosan-d3, caffeine-13C3, carbamazepine-d10, sulfamethoxazole-d4, diclofenac-d4, estrone-d4, oxybenzone-d5) were purchased from Toronto Research Chemicals (Ontario, Canada) at purity 98% or higher. LC-MS grade methanol, acetonitrile, and formic acid were from Fisher Scientific. Oasis HLB solid-phase extraction cartridges (6 mL, 200 mg) were from Waters Corporation ^[10].

Corresponding Author:

Jenna Buchanan

Department of Environmental
Science, University of Florida,
Gainesville, USA

Quality control and calibration

Method validation achieved recovery rates of 72-108% for all target analytes. Method detection limits ranged from 0.3 ng/kg (estrone) to 4.2 ng/kg (caffeine). Each analytical batch included a method blank, duplicate sample, and matrix spike. All batches with blank contamination exceeding 30% of the lowest sample concentration were re-extracted. Coefficient of variation for duplicate analyses was below 12% ^[11].

Research area description

Three commercial farms practicing wastewater irrigation were selected within 30 km of Gainesville, Alachua County, Florida (29°39'N, 82°20'W). Farm A received primary-treated effluent, Farm B received secondary-treated (activated sludge) effluent,

and Farm C used groundwater from the Floridan Aquifer as reference. All farms grew similar crop rotations on Arredondo fine sand with pH 5.8-6.4 and organic matter 1.2-1.8% ^[12].

Methods

Soil sampling was conducted bimonthly at 0-15 cm and 15-30 cm depths from six points per farm over 18 months. Samples were extracted using pressurized liquid extraction (ASE 350, Dionex), cleaned by SPE, and analyzed on an Agilent 1290 UHPLC coupled with a 6495C triple-quadrupole mass spectrometer in MRM mode. Soil persistence was modeled using first-order kinetics. Statistical comparisons used Kruskal-Wallis tests with Dunn post-hoc correction at $p<0.05$ in R ^[13, 14].

Results

Table 1: Mean PPCP concentrations (ng/kg), detection frequency (%), and soil half-life by irrigation source

Compound	Farm A	Farm B	Farm C	Det. A%	Det. B%	Det. C%	t1/2 (d)
Ibuprofen	214	82	12	87.3	62.4	18.6	21
Triclosan	287	118	8	91.2	48.3	12.4	87
Caffeine	342	213	34	94.6	71.8	34.2	14
Carbamaz.	183	121	47	78.4	64.2	41.3	>120
Sulfameth.	94	43	18	68.7	51.6	22.7	32
Diclofenac	127	67	11	72.1	47.3	14.8	28
Estrone	38	14	4	43.8	28.7	8.3	18
Oxybenzone	198	86	16	82.6	56.4	21.6	43

Mean PPCP concentrations (ng/kg dry soil) and detection frequencies were highest in Farm A and lowest in Farm C across all eight compounds (Table 1). Carbamazepine showed the

longest soil half-life (>120 days). Secondary treatment reduced soil concentrations by 38-67% compared to primary-treated irrigation.

Table 2: Total PPCP loads, irrigation water concentration, and soil biological indicators by farm

Parameter	Farm A	Farm B	Farm C	p-value
Total PPCPs 0-15cm	1,483	744	150	<0.001
Total PPCPs 15-30cm	412	187	38	<0.001
Irrigation water (ug/L)	8.74	3.21	0.08	<0.001
Soil microbial resp.	3.12	3.84	4.47	0.012
Dehydrogenase act.	48.3	62.7	78.4	0.003

Total PPCP loads in the upper 15 cm were nearly ten times higher in Farm A than in groundwater reference soils (Table 2). Soil microbial respiration and dehydrogenase activity were

inversely related to contamination level.

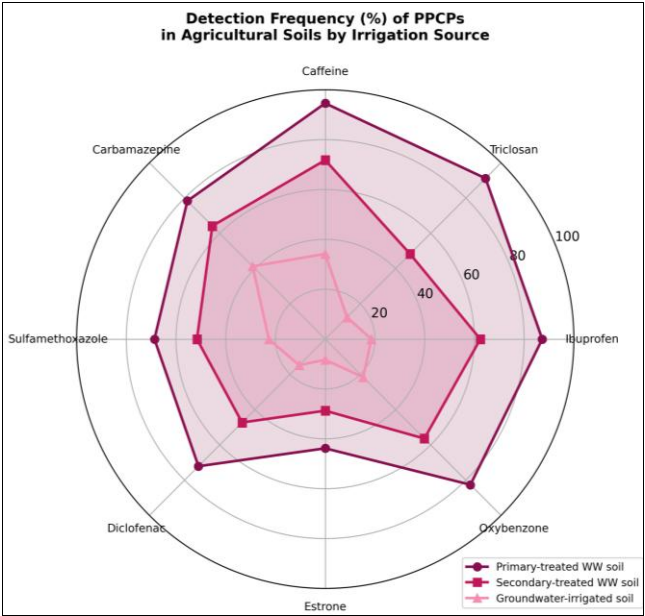


Fig 1 Radar chart comparing detection frequency (%) of eight PPCPs in soils under three irrigation sources

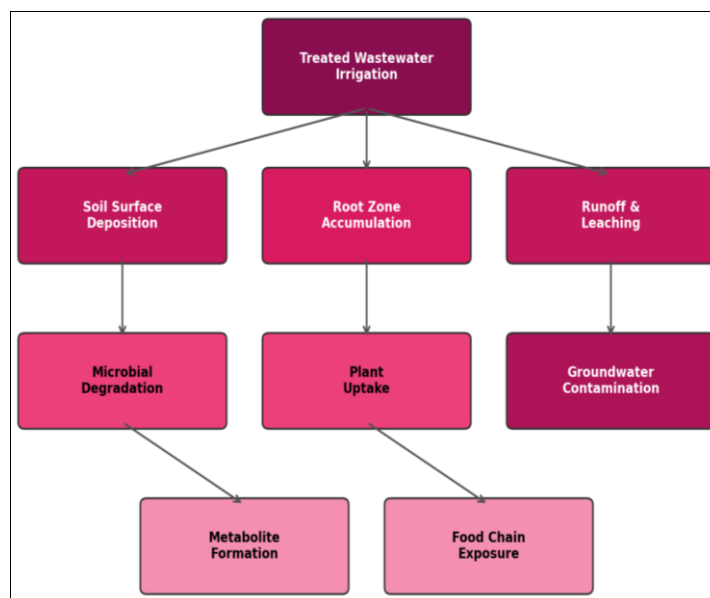


Fig 2: Conceptual diagram of PPCP transport and fate pathways in agricultural soils irrigated with treated wastewater

Comprehensive interpretation

The data paint a clear gradient: primary-treated wastewater delivers the highest contaminant loads, secondary treatment cuts them substantially but does not eliminate them, and even groundwater-irrigated soils carry measurable traces of the most persistent compounds ^[15, 16].

Discussion

The PPCP concentrations observed in Farm A soils are within the range reported from wastewater-irrigated sites in Israel and Spain, though direct comparison is complicated by differences in treatment level, irrigation frequency, and soil type ^[5, 6]. The 38-67% reduction achieved by secondary treatment falls short of the >90% removal needed to keep soil loads below proposed ecological screening values. Tertiary treatment technologies including ozonation and activated carbon adsorption can achieve such removal rates but remain expensive for agricultural-scale water reclamation ^[17].

The suppression of soil microbial activity in higher-contamination soils raises concerns about long-term soil health impacts. Triclosan is a known antimicrobial agent, and its accumulation at 287 ng/kg may alter microbial community structure, with downstream effects on nutrient cycling and disease suppression ^[7]. The inverse relationship between PPCP load and biological activity warrants further investigation using molecular community profiling approaches ^[18].

Conclusion

This 18-month field research confirmed that agricultural soils irrigated with treated wastewater accumulate measurable concentrations of pharmaceuticals and personal care products, with primary-treated effluent producing loads nearly ten times higher than groundwater-irrigated reference soils. Secondary treatment reduces but does not eliminate soil contamination. Carbamazepine and triclosan were the most persistent and frequently detected compounds, suggesting they should be priority targets for monitoring. The observed suppression of soil microbial activity indicates that PPCP accumulation carries ecological consequences. These findings support upgrading wastewater treatment infrastructure and establishing soil PPCP monitoring where reclaimed water irrigation is practiced.

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