Effects of global change on the emission, fate, effects and risks of chemicals in aquatic ecosystems

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Deliverable D4.2 (D22): Crop uptake models and human exposure models for contaminants in water reuse systems

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INTRODUCTION

Application of treated wastewater (TWW) and biosolids to the agricultural system is a common practice in many arid and semi-arid regions. For example, in Israel more than 50% of the water used for crop irrigation is TWW (Fu et al., 2019). About 50% of the produced biosolids in the US and Europe are used for organic amendment to agricultural lands (Ben Mordechay et al., 2017). On a global scale, irrigation with TWW is expected to increase due to the looming water crisis. Increase in population demands more food production, and soil amendment with biosolids can help to increase the agricultural yield.

The irrigation with TWW and application of biosolids facilitates introducing of different pollutants of emerging concern such as pharmaceutical compounds (PCs) into the agricultural systems. Water reuse guidelines do not account for the presence of such pollutants and the potential risks these may pose. Once in the soil, PCs undergo sorption, degradation/transformation, and transport processes which determine their bioavailability.

Different crops that are exposed to PCs via TWW or biosolids have been shown to accumulate PCs from soil (Malchi et al., 2014; Goldstein et al., 2018). The PCs will be uptaken and accumulated in higher concentrations in crops grown in soils with low organic matter and clay content (Goldstein et al., 2014).

The presence of PCs and their metabolites in the agricultural environment raised concern due to the potential ecological and health risk associated with the human exposure to these pollutants. Even though the concentrations of PCs in the environment is low (ranges from µg/L to ng/L) and apparently will not cause acute toxicity for humans, the effect may build up over a long period. This risk can be more serious in countries where raw wastewater or semi- treated wastewater is used for irrigation (Carter et al., 2019).

A holistic risk framework for evaluating the impacts of PCs, present in wastewater reuse systems, on human and ecosystem health was proposed by (Carter et al., 2019). This Source–pathway–receptor analysis revealed that it is currently impossible to fully understand the risks of PCs in agricultural systems due to several significant knowledge gaps in the fate and behaviour of PCs entering the agricultural system.

To better understand the crop uptake and human health risks associated with reclaimed wastewater use and to develop valid models, the first task is to have better insights on the bioavailability of PCs in the root zone under changing environment. PCs behaviour in soils and their plant bioavailability are related to the presence of microplastics, which are pollutants of emerging concern. Hence, it is necessary to develop experimental datasets, which help to investigate the environmental fate of pharmaceuticals, and to evaluate the impact of microplastic pollution of soils on the plant exposure to PCs in agricultural systems.

PROJECT DESCRIPTION

Title:

Fate of pharmaceuticals in soils: Effect of microplastics and biopolymers.

Background of the study:

Microplastics in soils, and their impact on PCs fate in soils

The microplastic pollution is among the most important topics in environmental research in recent years (de Souza Machado et al., 2018, Xu et al., 2019). It is one of the most extensive and long-lasting anthropogenic changes on the earth surface (Barnes et al., 2009). The focus of research has been so far on microplastic pollution in the marine and freshwater environment.

The exposure and effect of microplastics in terrestrial systems have been largely unexplored even though the annual plastic release to land is estimated at 4-23 times of that released to oceans (Horton et al, 2017).

Microplastics accumulate within continental environments, especially in areas of high anthropogenic influence such as agricultural or urban areas (Horton et al., 2017). The contamination is high in agricultural areas, where green houses, mulches or silage films are applied (Piehl et al., 2018). Application of biosolids is another significant source of microplastic pollution in agricultural lands since 80% to 90% of the microplastics in domestic wastewater are entrained in the sludge (Talvitie et al., 2017). The treated sewage sludge also retain significant amount of microplastics, and the treatment processes may alter the surface properties according to the plastic type (Mahon et al., 2017). Microplastic fibers maintaining original characteristics as in sludge products have been found in agricultural fields up to 15 years after biosolids application (Zubris et al., 2005).

Only limited data is available for microplastic concentration in the soils. The microplastic concentration of up to 7% by weight was recorded in top soils close to roads and industrial areas in Sydney (Fuller and Gautam, 2016), and up to 60% concentrations in the top soil of contaminated areas could be realistic to occur in the environment (Huerta Lwanga et al., 2017). In general, polyethylene, polystyrene and polypropylene are the most common types of microplastics detected in soil (Piehl et al., 2018).

The tire wear particles are also the major contributors of microplastics in the environment. It is reported that 28% of all global emissions of microplastics are tire wear particles (Boucher et al., 2017). Tire wear particles can be transported from road by rainwater runoff into soils; the atmospheric deposition is another pathway of tire wear particle into terrestrial systems (Kole et al., 2017). Tire wear particles conveyed to wastewater treatment plants by urban runoff can also be expected to reach the soil system as described in case of other microplastics (Nizzetto et al., 2016). Little knowledge is available on the interactions of tire wear particles and organic pollutants.

The presence of microplastics in soil can affect the fate of organic pollutants (Huffer et al., 2019). The migration of pollutants from soil to groundwater and freshwater reservoirs and the bioavailability of pollutants can be affected by the presence of microplastics. Both microplastics and PCs co-exist in soils. PCs can be adsorbed on microplastics (Li et al., 2018; Magadini et al., 2020; Puckowski et al., 2020). Hence, PCs can be either blocked in soil pores with microplastics or transported with microplastics (Xu et al., 2019). The sorption efficiency of microplastics and its effect on mobility of contaminants depends on properties of both sorbent and sorbate (Huffer et al., 2019). Sorption capacity of different microplastics depends on its structure, composition, surface characteristics, glass transition temperature and degree of crystallinity (Fred-Ahmadu et al., 2020).

Microplastics can affect the transport of organic compounds in soil since their sorption affinities towards microplastics are significantly different from that of the natural organic matter and minerals in soils (Teuten et al., 2007). The presence of polyethylene reported to increase the mobility of atrazine and 4-(2, 4-dichlorophenoxy) butyric acid in soils (Huffer et al., 2019). The information on the effects of microplastics on the transport of PCs in soil is lacking.

The aging of microplastics have potential to alter its surface characteristics and its interactions with co-occurring chemical pollutants. The UV- accelerated aging of polystyrene reduced the sorption of nonpolar organic compounds (Huffer et al., 2018), whereas it increased the sorption of hydrophilic organic compounds on polystyrene and polyvinyl chloride (Liu et al., 2019). The reports on PCs sorption on aged microplastics are limited. The PCs sorption on tires and the impact of the tire presence in soil on PCs transport of PCs is has not been investigated yet.

Biopolymers as natural soil sorbents and their impact on fate of PCs in soils

One of the most important precursors for soil organic matter are plant cuticles, and their major component biopolymers cutin, and cutan are considered as efficient sorbents of organic pollutants (Shechter et al., 2010). Cutin is a polyester-like macromolecule that can be depolymerized and solubilized upon saponification, while cutan is a non-saponifiable aliphatic biopolymer with higher preservation potential than cutin (Deshmukh et al., 2003; Boom et al., 2005). High sorption affinity of cutin and cutan for organic compounds emphasize their sorption potential as natural sorbents in soils, which can influence the mobility of organic contaminants in soil. The impact of biopolymers on PCs transport in soil has not been studied yet.

Soil organic matter (SOM) content can affect the sorption of PCs in soil and hence their mobility in the soil matrix. High SOM content in soil will usually cause greater retardation of organic contaminants in the soil profile (Borgman et al., 2013). Thus, the presence of high level of biopolymers will increase SOM content and may decrease PCs mobility. The structural composition of biopolymers affects their biodegradability in soils (Olshansky et al., 2015). Sorptive affinity of biopolymers towards organic pollutants is affected by degree of biopolymer decomposition (Stimler et al., 2006; Shechter et al., 2008). Though sorption of different organic pollutants on biopolymers were studied, the effect of biopolymers and their decomposition on transport of PCs in soils are yet to be elucidated.

Research objectives:

The overall goal of this research is to investigate the effect of anthropogenic and natural micro-polymers (i.e., microplastics and cutin/cutan) on fate of PCs in soils. Specific aims are: (1) To study and compare the effect of microplastics and biopolymers on transport of PCs in different soils. (2) To elucidate the impact of aging of microplastics on their contribution on transport of PCs in soils. (3) To examine the effect of decomposition of biopolymers on their role on transport of PCs in soils.

Working hypothesis:

1) Microplastics can affect the transport of PCs in soil since their adsorptive capacity are significantly different from that of soil organic matter and minerals. Different type of microplastics will thus have different effect on mobility of PCs in soil.
2) The effect of microplastics on mobility of PCs will be more prominent with aged microplastics since aging can lead to significant changes in surface properties of microplastics due to its oxidation and formation of localized microcracks, and therefore increase its sorption capacity.
3) Amendment with biopolymers will reduce the mobility of PCs in soils since it will act as an efficient adsorbent. Cutin and cutan will influence the mobility of PCs in soil differently since they exhibit different sorption characteristics due to their difference in structural properties.
4) Despite cutin's polyester and cutan's polyethylene-like structures, the impact of these biopolymers on PCs transport will differ from that of microplastics due to dissimilarity of nature and properties of microplastics and biopolymers, such as crystallinity and glass transition temperature.

Detailed description of the research plan:

Choice of soils: The experiments will be conducted on sandy and clayey soils. The properties of selected soils are presented in Table 1.

Table 1**:** Selected properties of the studied soils.

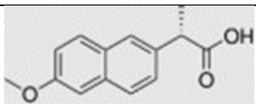
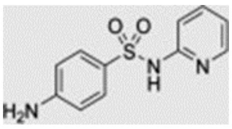
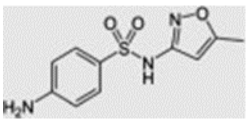
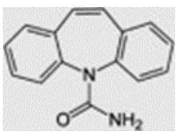
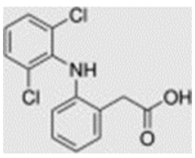
	Nir Oz	Ein Hashlosha	Sa'ad
Clay %	13	15	40

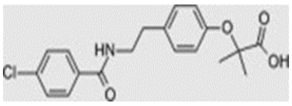
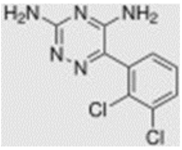
Silt %	8	12.5	12.5
Sand %	79	72.5	47.5
Organic Carbon %	0.81 ± 0.02	1.3 ± 0.04	0.48 ± 0.02
pH	7.87 ± 0.15	7.8 ± 0.19	7.81 ± 0.02

Choice of microplastics and biopolymers: Most commonly present microplastics polyethylene, polypropylene, polyester, polystyrene and tire wear particles will be used for the study. The experiments will be conducted with two different sizes of microplastics, 300 nm and 1 mm. Plant cuticular materials will be used as biopolymers for the study. The cutin and cutan will be isolated from the fruits of tomato and leaves of *Agave Americana* as in (Shechter et al., 2010). The tomato cuticle is a cutan-free with major structural component being cutin, whereas for *Agave Americana*, cutan is the major constituent together with cutin (Shechter et al., 2006).

Choice of PCs: The experiments will be conducted with wide range of PCs that are persistent in soil and known to pose environmental risk. The physicochemical properties of selected pharmaceuticals are presented in Table 2.

Table 2: Physicochemical properties of selected PCs.

Name and formula	Structure	Molecular weight (g/mol)	pK_a	Log K_{ow}	Water solubility (mg/L)
Naproxen $C_{14}H_{14}O_3$		230.3	4.45	3.18	15.9 at 25°C
Sulfapyridine $C_{11}H_{11}N_3O_2S$		249.3	pKa1=2.28 pKa2=8.4	0.35	268 at 25°C
Sulfamethoxazole $C_{10}H_{11}N_3O_3S$		253.3	pKa1 =1.6 pKa2 =5.7	0.89	610 at 37°C
Carbamazepine $C_{15}H_{12}N_2O$		236.3	pKa1 = −0.5 pKa2 = 14.4	2.45	18 at 25°C
Diclofenac $C_{14}H_{11}Cl_2NO_2$		296.2	4.14	4.51	2.37 at 25°C

Bezafibrate $C_{19}H_{20}ClNO_4$		361.8	3.3	4.25	1.55
Lamotrigine $C_9H_7Cl_2N$		256.1	5.7	2.57	170 at 25°C

Characterization of microplastics and biopolymers: The sorbents will be characterized by high-resolution scanning electron microscope (HR-SEM) to study surface topography and the BET technique to measure specific surface area. The microplastics will be characterized by X-ray powder diffraction (XRD) analysis to check crystallinity. The biopolymers will be characterized by Fourier Transform Infrared Spectroscopy (FTIR) to understand functional groups present in the cuticular matrix.

Aging of microplastics and decomposition of biopolymers: Microplastics will be artificially aged in a custom-made aging chamber as in (Huffer et al., 2018). In brief, the particles will be treated with H_2O_2 for sorbent surface oxidation. The samples will be then exposed to UV light. The aged particles will be characterized prior to their use in the experiments.

The cutin and cutan will be incubated in soil in plastic containers as in (Shechter et al., 2010) and will be sampled after 1, 3, 6 and 12 months of incubation. The samples will be characterized prior to the experiments.

Transport experiments: The mobility of PCs in soil can be studied using column transport experiments. The effect microplastics, cutin and cutan have on fate of PCs in soil will be studied for 5% and 10 % (w/w) of soil amendment. Transport of PCs applied as a mixture will be studied in: (i) soils, (ii) soil mixed with pristine microplastics, (iii) soils mixed with biopolymers, (iv) soils mixed with aged microplastics, and (v) soils mixed with decomposed biopolymers. The column experiments will be initiated by saturating the column from the bottom with a background solution of 5 mM $CaCl_2$. Then pulse solution containing mixture of PCs mentioned above and the tracer (10 mg/L KBr) will be continuously applied to pertinent soil columns. All transport experiments will be performed at two different flow rates to evaluate the effect of flow rate on the transport of PCs (Seutnjens et al., 2001).

The concentrations of PCs will be measured for periodically collected eluents using HPLC and LC-MS. The concentrations of bromide in eluents will be measured by ICP-AES. After the contaminant leaching experiment, the input solutions will be switched to 5 mM $CaCl_2$ to evaluate the desorption process of PCs from the columns. Breakthrough curves for each pharmaceutical will be constructed using concentrations obtained in their eluents and initial solutions. All experiments will be performed in triplicate.

Using Hydrus 1D (Seutnjens et al., 2001; Chotpantarat et al., 2011; Park and Huwe, 2016), water and solute transport will be simulated by applying chemical non-equilibrium model. Hydrodynamic dispersion coefficient will be evaluated using data obtained for transport of non-reactive (Br) tracer. Using inversion method (available in Hydrus 1D), retardation factor, the parameters of sorption isotherm, and the reaction rate coefficients will be calculated. As in (Seutnjens et al., 2001), the rate coefficients at two flow rates will be compared to infer whether mass transfer between surface sites and solution is limited by diffusion or by kinetic sorption.

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