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Fate and transport modeling of phthalate esters from biosolid amended soil under corn cultivation

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HIGHLIGHTS

- Investigation of transport of seven phthalates through a biosolid-amended soil under corn cultivation.
- Study of numerical simulation of phthalates leaching using HYDRUS-1D model.
- Increasing Log K_{ow} of PAEs (>5) resulted in lower transport from soil.
- A dual porosity two-site model predicts transport and adsorption of PAEs in soil.

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ABSTRACT

Phthalate esters (PAEs) are prevalent in the environment due to the broad range of industrial, agriculture and domestic applications. The ubiquitous use of PAEs has resulted in their potential to reach groundwater sources through application of agri-chemicals and municipal biosolids. A study was conducted to monitor the fate and transport of seven commonly detected PAEs in the environment including: dimethyl phthalate (DMP), diethyl phthalate (DEP), benzyl butyl phthalate (BBP), bis(di-ethyl hexyl) phthalate (DEHP), di-*n*-octyl phthalate (DnOP), dipentyl phthalate (DPP), and di-*n*-butyl phthalate (DnBP). Biosolids sourced from the Halifax Regional Municipality were applied at three rates on field-based lysimeter cells which were cropped to corn (*Zea mays*) for one growing season. In the present study, breakthrough curves (BTCs) were established for phthalates leaching from a corn-cultivated agricultural soil profile. The HYDRUS-1D model and a two-site sorption model were applied to predict transport parameters of PAEs using an inverse solution approach. Results of our research revealed that higher PAE adsorption was observed based on increasing carbon chain number. In addition, higher values of *F* (i.e. the fraction of type-1 sorption sites assumed to be in equilibrium with the solution phase) and lower values of *D* (i.e. dispersion coefficient) were observed for PAEs with large carbon chains which was validated both through the empirical dataset and the model simulations.

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1. Introduction

Phthalate esters (PAEs) are prevalent in the environment because of the broad range of use in industrial, agriculture and domestic applications. Although PAEs are mainly used as plasticizers to induce flexibility and workability of polymeric materials, they are also used to varying degrees in the manufacture of insect repellents, synthetic fibers, toys, paint pigments, medical products

and cosmetics. The annual worldwide production of PAEs exceeds 8 million metric tons [1]. The detection of PAEs in groundwater has been reported from land application of sewage sludge [2], irrigation using treated wastewater [3], leakage from stored drums at military and industrial facilities [4,5] or as a result of untreated leachate from landfills [6]. Some PAEs are suspected to be mutagens, hepatotoxic agents and carcinogens [7]. They are also included in the EPA list of priority pollutants [8] and are considered as Emerging Substances of Concern (ESOC) in the environment. As a result, several review studies have been conducted to highlight the factors that affect the transport and fate of these compounds in the envi-

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ronment [9–12]. Despite the recent emphasis on phthalates in the environment, significant gaps in knowledge still persist.

The current literature on PAE transport through soil has typically been conducted using batch reactors [13–15], disturbed soil columns [4], or small undisturbed soil columns [16] with no plant cultivation. Moreover, most of the studies on transport of ESOE in soils are carried out in water saturated soil columns. This can pose a challenge in transferring results from the laboratory to field-based conditions. Some of the existing challenges of using disturbed soil columns are reflected in unrealistic soil structure or pore geometry. In the field, preferential flow conditions, i.e. macropores resulting from root channels, soil macroorganisms, natural soil cracks, can bypass the soil matrix and increase solute mobility through the soil profile [17]. The presence of a crop rooting system can also have a direct, e.g. physico-chemical, or indirect, e.g. increase in microbial populations, effect on solute transport.

Modeling chemical transport through soils requires determination of specific soil parameters, such as bulk density and hydraulic conductivity, which can influence mobility and validation under field conditions. Fate and transport models can be applied to numerically solve different governing equations of solute transport through soil to provide a better understanding of the governing processes which promote chemical movement, including partitioning coefficients. For instance, HYDRUS-1D is an open-source water flow and solute transport model which has been extensively used with contaminants through soil [18]. The use of HYDRUS-1D has been well validated for a range of chemicals but has not been applied to the transport of PAEs from biosolids applied under agricultural cultivation.

The objective of this study was to simulate the governing processes in soil influencing the mobility of dimethyl phthalate (DMP), diethyl phthalate (DEP), benzyl butyl phthalate (BBP), bis(di-ethyl hexyl) phthalate (DEHP), di-*n*-octyl phthalate (DnOP), dipentyl phthalate (DPP), and di-*n*-butyl phthalate (DnBP) using the HYDRUS-1D model. The study was conducted under field conditions in a natural sandy loam textured soil under corn cultivation and with a history of alkaline treated biosolid applications.

2. Materials and methods

2.1. Site description and experimental design

A study was conducted using nine field-based lysimeter cells with a Truro Association (Tormentine), Ortho-Humic Podzol soil at the Bio-Environmental Engineering Centre (BEEC), Faculty of Agriculture, Dalhousie University, located in Bible Hill, Nova Scotia, Canada (45°23' N, 63°14' W), from May 2014–November 2014. The soil was a highly acidic reddish brown sandy loam till with a soil particle size distribution of 58.9% sand, 30.9% silt, and 10.3% clay [19]. The sandy loam soil had an initial pH of 5.2, electrical conductivity of 0.1 mS cm⁻¹, soil bulk density of 1.39 mg cm⁻³, organic matter content of 3.4%, total C of 1.4%, total N of 0.1%, and water content at 33 and 1500 kPa of 0.25 and 0.07 (w/w), respectively.

The lysimeter study site was established in 2009 and allowed to settle for two years prior to application of any soil treatments. The lysimeter cells were arranged in a completely randomized design with three replicates, allowing for a control and two treatments. Each lysimeter cell had total dimensions measuring 5.79 m × 3.35 m × 0.6 m with a geotextile membrane-lined inner cell with dimensions of 1.2 m × 1.1 m × 0.6 m in the centre of each lysimeter, established directly over a subsurface drain. The subsurface drainage pipe was installed beneath each lysimeter cell, with a drain in the centre, to a depth of 0.60 m and covered with gravel and a permeable landscape cloth. Each sub-surface drain was connected to a tipping bucket system and an ISCO 6712 Portable

Sampler (Teledyne ISCO, Lincoln, NE) for water sample collection during flow events. The tipping bucket assembly for each lysimeter cell was also connected to a Campbell Scientific CR-100 datalogger to measure discharge rate for every flow event over the entire year.

In the spring 2012, alkaline treated biosolids originating from the N-Viro Systems Canada Biosolids Facility operating in the Halifax Regional Municipality were applied in the inner cell of each lysimeter at rates of 0, 7, and 28 Mg ha⁻¹ (w/w). Phthalate concentrations of the alkaline treated biosolids (ATB) are shown in Table 1 and additional chemical characteristics of the ATB are presented in Price et al. [20]. The biosolids were incorporated into the top 10 cm of the soil and planted with annual ryegrass (*Lolium multiflorum*). The same rates of biosolids were applied in 2013 to the lysimeter plots. In the study year, 2014, the biosolid rates were increased to ensure phthalate esters in the discharge water would be at the method limit of detection; therefore, lysimeter cells previously amended with 7 Mg ha⁻¹ received 28 Mg ha⁻¹ and the 28 Mg ha⁻¹ treatment cells received 42 Mg ha⁻¹. The current agronomic recommendations in Nova Scotia for growing corn using the ATB product is 14 Mg ha⁻¹ (w/w). Dekalb RoundUp Ready (DK2678/DK2679) variety of corn (*Zea mays* L.) was planted in the inner and outer cells in the late spring (June) 2014 with a spacing allowing for a total of six corn rows per lysimeter cell, and two corn rows within the inner cell. A nitrogen fertilizer was applied (27-0-0) at 25 kg N ha⁻¹ at planting and as a sidedress three weeks after germination. The corn was harvested at full maturity by hand in the fall (late October) and separated into stover (biomass) and grain. Fresh weight at harvest was taken of the corn biomass and grain and subsequently dried at 45 °C for five days. The dried crop material, corn biomass and grain, was fully ground to pass through a 2 mm sieve using a Wiley Mill. Three 10 g subsamples of corn biomass and grain for each lysimeter cell were taken for analysis of phthalates.

2.2. Soil and discharge sampling and laboratory analysis

Soil sampling of each lysimeter cell was conducted over three time periods during the growing season. In each lysimeter cell, a composite of five soil cores collected from the inner cell was made using an Oakfield soil probe with a diameter of 2.5 cm and to a depth of 0–15 cm. Initially, soils were sampled in early spring (May) prior to application of biosolids to determine background concentrations of phthalates. Subsequently, soil samples were collected one week (June) and one month (July) after application of the biosolid treatments. Soil samples were passed through a 2 mm stainless steel sieve and stored at –20 °C. Prior to extraction, the soil samples were freeze dried and two 10 g subsamples were taken for analysis. The extraction and analytical procedures by Accelerated Solvent Extraction (ASE) and GC–MS are described in Khosravi and Price [21].

The HYDRUS-1D model uses specific soil characteristics including: soil bulk density, soil particle analysis, saturated hydraulic conductivity (Ks), and moisture curve parameters to provide estimates on chemical transport. Wherever parameters were not determined empirically they can be predicted using the Rosetta program included in HYDRUS-1D. Field measurements were determined in each lysimeter cell for soil bulk density [22], soil particle distribution [23], organic matter content [24], soil moisture release by pressure plates at 10, 33, and 1500 kPa [25], and saturated hydraulic conductivity by Guelph permeameter, at the 0.15 and 0.45 m depths [26]. The HYDRUS-1D model was initially run for each treatment separately using two models: 1) equilibrium advection–dispersion (CDE) and 2) the physical (two-region) nonequilibrium advection–dispersion models. In equilibrium CDE, all the soil is supposed to be composed of one type of sorbent while the two-site sorption concept assumed that the sorption sites could be divided into two fractions. Sorption was assumed

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