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Perfluorooctane sulfonate in surface soils: Effects on reproduction in the collembolan, *Folsomia candida*, and the oribatid mite, *Oppia nitens*

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HIGHLIGHTS

- Chronic toxicity of PFOS was determined for two soil invertebrates in two soil types.
- Toxicity was 2–4 times greater in the sandy loam soil, compared to clay loam soil.
- Oribatid mites were significantly more sensitive to PFOS, compared to collembolan.
- Application of *Oppia nitens* as a new standard soil test species was demonstrated.
- Results fulfill data gap for soil quality guideline derivation for PFOS in soil.

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ABSTRACT

Perfluorooctane sulfonate (PFOS) is a persistent organic pollutant, which has been detected at significant concentrations in soils at sites used for fire-fighting training operations. Recent ecotoxicological research has mainly focused on earthworms to assess the toxicity of PFOS in soil. However, the inclusion of other soil taxonomic groups allow for a more holistic estimate of contaminant risk, including the derivation of more comprehensive soil quality guidelines. The present study assessed the toxicity of PFOS using the collembolan, *Folsomia candida*, and the oribatid mite, *Oppia nitens*, in two types of soil: a coarse-textured sandy loam (VSL) and fine-textured clay loam (NRS). As a standard *O. nitens* reproduction test is being formalized, the results of the study were also used to compare sensitivity across test species. Effects were soil dependent, with test species being 2–4 times more susceptible to PFOS in VSL, relative to NRS, likely due to differences in organic matter and clay content. *Oppia nitens* was significantly more sensitive to PFOS, regardless of soil type, in comparison to *F. candida*. The IC50s for reproduction for *O. nitens* were 23 mg kg^{-1} (95% confidence interval: 17–32 mg kg^{-1}) in the VSL and 95 mg kg^{-1} (69–134 mg kg^{-1}) in the NRS, and for *F. candida* were 94 mg kg^{-1} (72–122 mg kg^{-1}) in the VSL and 233 mg kg^{-1} (177–306 mg kg^{-1}) in the NRS. The present study demonstrates the application and inclusion of the oribatid mite, *O. nitens*, for the risk assessment of contaminants in soil.

1. Introduction

Perfluorinated alkyl (PFA) compounds were extensively used for decades for the surface protection of paper and textiles, as a fume suppressant within the electronics industry (e.g., electroplating), as well as additives to improve lubricants, polymers, paints, and fire suppression materials (aqueous film forming foams) (3M Company, 2000; Moody and Field, 2000; Zhang et al., 2012). However, despite the perceived industrial and consumer benefits of these substances, the global detection across environmental media, as well as in humans and wildlife has since led to the combined voluntary and regulatory phase-out of specific PFAs to mitigate further environmental impacts (European Parliament, 2006; Government of Canada, 2009). Of the PFAs, perfluorooctane sulfonic acid (PFOS) has become one of the most studied, mainly due to its chemical stability, resistance to degradation, and its persistence within the environment and wildlife (Giesy and Kannan, 2001; Houde et al., 2011). In 2009, PFOS and related compounds were included in Annex B of the Stockholm Convention on Persistent Organic Pollutants (Stockholm Convention, 2009), resulting in restricted

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manufacture and use within the EU, with similar actions effected in North America (US EPA, 2000). However, manufacture and use under usage exemptions continue in some countries (Zhang et al., 2012), which have contributed to environmental releases not only within the country of origin, but within the global market through export and import activities.

The fate and effects of PFOS have been well-studied with evidence of bioaccumulation and biomagnification in wildlife (Müller et al., 2011; Greaves and Letcher, 2013; D'Hollander et al., 2014), and detection in environmental media spanning from highly-concentrated industrialized areas to rural and remote regions (Giesy and Kannan, 2001; Rankin et al., 2016; Chen et al., 2016). Within Canada, usage of PFOS in fire suppressant formulation for fire-fighting training activities has led to the contamination of many locations managed by the Federal government. In general, PFOS has been historically used as an effective fire suppressant for hydrocarbon-fuel fires, with applications at airport, oil refinery and military facilities (Moody and Field, 2000). Although not limited to Canada, this has led to the detection of high soil concentrations within training facilities and localized regions, as a result of surface soil contamination, run-off and long-term vertical translocation through the soil profile (Baduel et al., 2015; Xiao et al., 2015; Filipovic et al., 2015). Further inputs to the soil also occur from the degradation of PFOS precursors (Anderson et al., 2016), with recent concern resulting from the retention and accumulation within sludge (biosolid) material, and subsequent application of sludges to agricultural land (Higgins et al., 2005; Washington et al., 2010; Gottschall et al., 2017). Given the existing legacy of contaminated sites, and the potential influx from biosolid amendment, an evaluation of the ecotoxicological effects of PFOS on soil fauna are warranted.

Despite the regulatory phase-out, PFOS contamination is still being detected due to its persistence and attenuation within soils (Houtz et al., 2013; Xiao et al., 2015; Rankin et al., 2016). As this compound is highly stable in soil, there is a significant risk of adverse effects to terrestrial organisms. There are a limited number of studies that evaluate the impacts of PFOS to soil fauna, with the majority focused solely on earthworm toxicity and bioaccumulation (Joung et al., 2010; Zareitalabad et al., 2013; Xu et al., 2013; Das et al., 2013; Zhao et al., 2013; D'Hollander et al., 2014; Rich et al., 2015; Wen et al., 2015; Navarro et al., 2016). The effects of PFOS to other soil fauna are unknown, and therefore, the objective of this study was to widen the scope and understanding of PFOS ecotoxicological effects on two additional soil fauna: the collembolan, *Folsomia candida*, and the oribatid mite, *Oppia nitens*. Collembola and oribatid mites are micro-invertebrates that comprise a significant portion of soil fauna globally, and are critical to the maintenance and preservation of soil structure through their contributions to decomposition and nutrient cycling (Seastedt, 1984; Singh et al., 1996). The use of *F. candida* is prevalent in ecotoxicity testing, with established standards for their use in the assessment of contaminants in soils (EC, 2014; OECD, 2009; ISO, 2014). Although the need for consideration of oribatid mites in ecotoxicity test has been recognized (Lebrun and van Straalen, 1995; Huguier et al., 2015), it is only recently that *O. nitens* was introduced as a potential test species (Princz et al., 2010). However, since this time, studies have demonstrated the value of the inclusion of this test species for substance and contaminated land effects testing (Princz et al., 2010; Princz et al., 2012; Owojori and Siciliano, 2012; de Lima e Silva et al., 2017; Jamshidian et al., 2017). As a result, efforts are progressing to formalize an *O. nitens* reproduction inhibition test as a new standard test method for soil ecotoxicity testing (ECCC, 2018; ISO, 2018). Therefore, the objectives of this study included a comparison of effects between the proposed test species, *O. nitens*, to the standard test species, *F. candida*. Furthermore, given that varying soil properties, such as organic carbon, can also play an influential role in the sorption and subsequent bioavailability of PFOS (Higgins and Luthy, 2006; Milinovic et al., 2015), ecotoxicity testing was conducted in two contrasting field surface soils.

2. Materials and methods

2.1. Test soils

Two soils were used to represent fine and coarse soil types (CCME, 2007). The fine soil (NRS) was collected from a fallow agricultural field in Ontario and characterized as a clay loam. Due to its clay-rich composition, clumps of air-dried NRS were passed through a soil grinder in order to facilitate processing. The coarse soil (VSL) was collected from Alberta and characterized as a sandy loam; the soil was air-dried and sieved through a 4-mm mesh. Subsequent to processing, the soils were air-dried, homogenized and stored in opaque pails at room temperature until use in tests. After air-drying, the soil moisture contents of the NRS and VSL were 21% and 3.7%, respectively; no indigenous fauna were observed throughout the tests or at time of test processing (i.e., via flotation or heat extraction), indicating that the soil processing method was sufficient to defaunate the natural soils. A summary of soil characteristics is provided in Table 1, with a more detailed description provided in Table S1.

2.2. Preparation of test soils

Perfluorooctane sulfonate (PFOS) potassium salt (≥98%, Sigma-Aldrich) (CAS 2795-39-3) was introduced to the test soils using acetone as a carrier; internal tests demonstrated that PFOS was soluble in acetone up to 42 g L^{-1}. Initial range-finding tests were conducted with the collembolan to narrow the effective concentration range for the reproduction tests. The results from the collembolan range-finder tests were also used to derive the test concentrations for the oribatid mite reproduction test in the VSL; however, the results of the VSL test demonstrated increased sensitivity of the species to the contaminated soil, and therefore, the test concentration range for the NRS was adjusted accordingly. In all cases, test concentrations were increased sufficiently in order to induce a toxic effect so that corresponding inhibitory concentrations (e.g., at the 10, 25 and 50% level) could be calculated, contributing to the soil guideline derivation process (CCME, 2007; ECCC, 2017). The final nominal test concentrations for the collembolan reproduction tests in both soils were 0, 21, 35, 60, 102, 173,

Table 1
Summary of soil characteristics for field soils used for toxicity tests. All values were obtained from single homogenized samples with the exception of soil pH and moisture content, which were averaged from multiple samples.

Parameter	Unit	VSL	NRS
Soil Texture		Sandy Loam	Clay Loam
Soil Type[a]		Coarse	Fine
Sand (>0.050 mm)[b]	%	75.2	39.8
Silt (>0.002–0.050 mm)[b]	%	16.2	28.3
Clay (≤0.002 mm)[b]	%	8.6	31.9
$pH_{0.01M\ CaCl2}$		5.4 ± 0.2	6.8 ± 0.1
Organic matter content (at 350 °C)[c]	%	2.6	15
Soil water holding capacity[d]	%	54.0	76.8
Optimal water holding capacity of soil[d]	%	42.5	52.5
Moisture content of test soil at test start[d]	%	19.8 ± 1.3	36.0 ± 1.3

[a] As defined in CCME (2007).
[b] Particle size distribution (filter candle system).
[c] Loss on ignition.
[d] EC (2014).