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Toxicity of fipronil to *Folsomia candida* in contrasting tropical soils and soil moisture contents: effects on the reproduction and growth

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Abstract

This study assessed the influence of three tropical soil types and soil moisture content on the toxicity and risk of the insecticide fipronil to collembolans *Folsomia candida*. Chronic toxicity tests were performed in a Tropical Artificial Soil (TAS), an Oxisol and an Entisol spiked with increasing concentrations of fipronil to assess the effects on the reproduction and growth of the species. The soil moisture contents were kept at 60% (standard condition) and 30 or 45% (water restriction) of their water holding capacity (WHC). The toxicity of fipronil on collembolans reproduction was about three times higher in Entisol compared to TAS or Oxisol. Higher toxicities were also found in the drier TAS ($EC_{50\ 30\%WHC} = 0.20$ vs $EC_{50\ 60\%WHC} = 0.70\ mg\ kg^{-1}$) and Oxisol ($EC_{50\ 45\%WHC} = 0.27$ vs $EC_{50\ 60\%WHC} = 0.54\ mg\ kg^{-1}$), while in Entisol lower impacts were found in the drier samples ($EC_{50\ 30\%WHC} = 0.41$ vs $EC_{50\ 60\%WHC} = 0.24\ mg\ kg^{-1}$). For all tested soils, the size of generated collembolans was reduced by the fipronil concentrations, regardless of soil moisture. However, the drier condition increased the effect on the growth in TAS and Entisol for some concentrations. A significant risk of exposure was found in TAS and Oxisol at drier conditions and, for Entisol, regardless of the soil moisture. The toxic effects and risk of fipronil on collembolans were higher in the natural sandy soil. The soil moisture content increase or decrease the toxicity of the insecticide for collembolans, depending on soil type.

Keywords Climate change · Collembolans · Pesticides · Seed dressing · Soil ecotoxicology

Introduction

The Brazilian territory is composed of a wide variety of soil types with different characteristics (EMBRAPA 2006). These particular soil characteristics, such as texture, pH, cation exchange capacity (CEC), clay and OM contents help to regulate the water availability and the pesticide bioavailability/toxicity to collembolans (Amorim et al. 2005; Chelinho et al. 2014; Ogungbemi and Van Gestel 2018; Bandeira et al. 2019). Some studies comparing the toxicity of pesticides in different types of soils indicate that the toxic effects may be enhanced in sandy soils when compared with fine-textured soils (Amorim et al. 2005; Domene et al. 2012; Bandeira et al. 2019).

In addition to the influence of soil types, climatic factors also play an important influence on the behavior and dynamics of pesticides in soils. According to the Intergovernmental Panel on Climate Change (IPCC), an increase in drought events is expected for tropical countries in the next years, as a consequence of climatic changes (IPCC 2014). Although the consequences may vary for Brazilian regions, a 20% reduction in rainfall regimes is expected by the year 2040, 35% between 2041 and 2070, and 50% between 2071 and 2100, for five of the six biomes that constitute the Brazilian territory (PBMC 2014).

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Drought events can promote changes in the environmental fate of pesticides, influence the life-cycle of soil fauna, as well as affect their responses to the toxicants (Domene et al. 2012; Bonmatin et al. 2015; Daam et al. 2019). In this way, some studies have reported that reductions in soil moisture increased the toxicity of some pesticides to soil invertebrates (Lima et al. 2011; Bandow et al. 2014b; Bandow et al. 2016; Hennig et al. 2020).

Fipronil (5 - amino - 1 - [2,6 - dichloro - 4 - (trifluoromethyl) phenyl] - 4 - (trifluoromethylsulfinyl) -1H –pyrazole - 3 - carbonitrile) is a broad-spectrum insecticide that belongs to the class of phenylpyrazoles, widely used in chemical seed treatment (Mohapatra et al. 2010; Bonmatin et al. 2015). Negative effects of fipronil on the collembolan's survival, reproduction and bioaccumulation are reported in the literature. San Miguel et al. (2008) observed that fipronil affected the collembolan's survival ($LC_{50} = 0.45\ mg\ kg^{-1}$) and reproduction (with EC_{50} varying between 0.335 and $0.500\ mg\ kg^{-1}$), also indicating that these non-target organisms are very susceptible to bioaccumulation. Alves et al. (2014) also found that reduction in collembolans reproduction can occur when exposed to increasing concentrations of fipronil ($EC_{20} = 0.12\ mg\ kg^{-1}$). Zortéa et al. (2018a, 2018b) found that both survival (with LC_{50} varying between 0.29 and $0.48\ mg\ kg^{-1}$) and reproduction (with EC_{50} varying between 0.14 and $0.29\ mg\ kg^{-1}$) of collembolans can be affected by exposure to fipronil in a veterinary medicine formulation.Collembolans are considered a representative group of soil fauna and are essential to the maintenance of soil quality (Culik and Zeppelini 2003). These organisms are suitable bioindicators due to their high sensitivity to contamination and environmental stress (Culik and Zeppelini 2003). The exposure of these organisms to drought scenarios negatively affect their metabolism, compromise their growth, survival and reproduction (Bursell 1970; Edney 1977; Van Gestel and Van Diepen 1997; Jänsch et al. 2005; Sławski and Sławska 2020). Although several studies have reported the effects of drought on collembolans, no studies were found describing the relationship between lower moisture in tropical soil and fipronil toxicity to these organisms.

In this study, we hypothesize that a) the toxic effects and risk of fipronil via seed dressing formulations vary in contrasting soil types, being higher in natural sandy soil; b) the reduction of soil moisture content increases the fipronil toxicity to collembolans, depending on the tropical soil type. In this way, this study aimed to assess the influence of soil type and soil moisture content on the toxicity and risk of fipronil to collembolans *Folsomia candida*. For this, the effects of fipronil on the reproduction and growth of *F. candida* were assessed via chronic toxicity tests in three tropical soils (Tropical Artificial Soil – TAS, Entisol and Oxisol) under a standard (60% WHC) and a water restriction (30 or 45% WHC) conditions.

Material and methods

Test organisms

Collembolans *F. candida* were cultured in the laboratory according to ISO 11267 (ISO 2014). The organisms were kept in plastic containers containing a substrate composed of plaster of Paris, water, and activated charcoal (10:7:1, respectively). Twice a week, the culture medium received food (dry *Saccharomyces cerevisiae*) and had the moisture adjusted with a few drops of distilled water. The culture was kept in a room with a controlled temperature (20 ± 2 °C) and photoperiod (12 h). The species sensitivity was confirmed by using a positive control assay with boric acid in TAS ($EC_{50} = 171.55\ mg\ kg^{-1}$), according to Niemeyer et al. (2018).

Test soils

The ecotoxicological tests were performed in an artificial tropical soil (TAS) and two contrasting natural tropical soils.

The TAS (Garcia 2004) consisted of a mixture of fine sand, kaolin and powdered coconut husk (75:20:5, respectively). Calcium carbonate ($CaCO_3$) was used to adjust the pH to 6.0 ± 0.5 (ISO 2014). A clayey soil (Oxisol) was sampled in Palmitos (SC; 27° 04′S 53° 09′W) and a sandy soil (Entisol) was sampled in Araranguá (SC; 29° 00′S 49° 31′W). The soils were taken from the top layer (0–20 cm) of the soil profile in areas with no history of contamination by pesticides. The soil samples were sieved (#2 mm), defaunated through three freezing and thawing cycles, and air-dried (Alves et al. 2019).

For all test soils, the water holding capacity (WHC) and pH (1M-KCl) were determined according to ISO 11267 (2014), and the cation exchange capacity (CEC), soil organic matter (SOM), sand, clay, and silt contents (Table 1) were measured following Tedesco et al. (1995).

Test substance

Soils were spiked with a commercial formulation of an insecticide for seed dressing (Shelter®), which contains 250 g of fipronil L^{-1} as an active ingredient (a.i.).

Soil samples were spiked with increasing concentrations (Table 2), which were based on literature (Alves et al. 2014; Zortéa et al. 2018a) and preliminary tests (data not shown). A control treatment without contaminant (only with distilled water) was prepared for each soil.

Table 1 Mean values (±standard deviation; $n = 2$) of physical and chemical characteristics of Tropical Artificial Soil (TAS), natural sandy soil (Entisol) and natural clay loam soil (Oxisol), used in the ecotoxicological tests

Parameter	TAS	Entisol	Oxisol
pH (1M-KCl)	5.9 ± 0.1	4.2 ± 0.2	4.8 ± 0.1
SOM (%)	1.4 ± 0.0	2.2 ± 0.1	3.2 ± 0.8
CEC ($cmol_c\ dm^{-3}$)	3.3 ± 0.2	1.4 ± 0.1	10.8 ± 0.4
WHC (%)	46.3 ± 1.7	31.6 ± 1.1	53.0 ± 1.4
Sand (%)	67.2 ± 0.0	93.8 ± 0.4	31.5 ± 0.1
Clay (%)	14.3 ± 0.0	4.1 ± 0.2	35.5 ± 1.4
Silt (%)	18.5 ± 0.0	2.1 ± 0.1	33.0 ± 1.6
Soil texture	Sandy Loam	Sandy	Clay Loam

SOM Soil Organic Matter, *CEC* Cation Exchange Capacity, *WHC* Water Holding Capacity

Table 2 Nominal and real concentrations of the a.i. fipronil used in the chronic toxicity tests carried out in tropical artificial soil (TAS), entisol and oxisol with *Folsomia candida*

	Fipronil concentrations ($mg\ kg^{-1}$)			
Soil type	Treatment	Nominal	Actual	% Nominal
TAS	Control	0	0	–
	C1	0.25	0.21	84
	C2	0.5	0.44	88
	C3	1	0.9	90
	C4	2	1.62	81
	C5	4	2.89	72.2
Entisol	Control	0	0	–
	C1	0.125	0.17	136
	C2	0.25	0.25	100
	C3	0.5	0.57	114
	C4	1	1.03	103
	C5	2	1.71	85.5
Oxisol	Control	0	0	–
	C1	0.25	0.27	108
	C2	0.5	0.46	92
	C3	1	0.72	72
	C4	2	1.28	64
	C5	4	2.53	63.2

The spiking occurred through an aqueous solution, with volume sufficient to reach 30 and 60% of the WHC in TAS and Entisol, and 45 and 60% of the WHC in Oxisol, since the species did not reproduce in Oxisol at 30% WHC. The soil moisture was checked at the beginning and the end of each assay (Supplementary Information 1).

The time-weighted average Predicted Environmental Concentrations at 28d (PEC) for fipronil were estimated based on the methodology proposed by the European and Mediterranean Plant Protection Organization (EPPO 2003). The software ESCAPE (2013) was used to calculate the PEC based on a soybean crop scenario. It was considered a sowing density of 60 kg of seeds per hectare (ha) (EMBRAPA 1988) at a depth of 0–5 cm (Alves et al. 2013) and soil densities of 1.0 $g\ cm^{-3}$ for TAS and Oxisol, and 1.5 $g\ cm^{-3}$ for Entisol (Souza et al. 2005; Bandeira et al. 2020). A single Shelter® application dose of 12 g a.i. per 60 kg of seeds (lowest manufacturer's recommendation), over a single planting cycle, with an interception rate of 5% by the plants (Jackson et al. 2009) and a dissipation half-life of 31 days (EFSA 2006) were assumed.

Chemical analysis

For all tests, soil samples from each treatment were taken at the start of the test for the quantification of the real concentration in soils. The extraction of fipronil was based on the modified QuEChERS acetate, without the cleaning step (Gebrehiwot et al. 2019). For all treatments, 10 g wet soil was used. Then, 10 mL ultrapure water and 10 mL acetonitrile acidified with 1% acetic acid (v/v) were added. The mixture was manually shaken vigorously for 1 min. Afterwards, 6 g anhydrous magnesium sulfate ($MgSO_4$) and 1.5 g anhydrous sodium acetate (CH_3COONa) were added to the mixture and, then, it was manually shaken for 1 min. Finally, the tube was centrifuged at 9000 rpm for 5 min. Subsequently, 1 mL supernatant was taken out and diluted in 10 mL methanol. The final diluted extract was filtered through a 0.2 µm polyvinylidene fluoride Millex syringe filter.

The fipronil was analyzed by LC-ESI-MS (LC MS 2020 Shimadzu®, Japan). $LC^{separation}$ was carried out in C18 ec analytical column. The method was validated (single-laboratory validation; INMETRO 2020), attending linearity, accuracy (in terms of recovery), repeatability and intermediate precision (in terms of relative standard deviation, RSD) and limits of quantification ($LOQ = 0.01\ mg\ kg^{-1}$). The results of chemical analyzes were converted to mg a.i. per kg of dry soil, based on the soil moisture measured at the beginning of the ecotoxicological test (Table 2).

Test procedures

The chronic toxicity tests were performed according to ISO 11267 (ISO 2014) at the temperature of 25 °C and 12 h-photoperiod.

The experimental units were constituted by glass containers with airtight closure caps that received 30 g of spiked or control soil. Ten organisms with synchronized age (10–12 d) were inserted into each container. On the 1st and 14th day of the test, food was offered to the organisms (≈2 mg of dry *Saccharomyces cerevisiae*). Weekly, the containers were opened to allow gas exchange and for soil moisture adjustment with distilled water (by weight difference). At the end of the test (28 d), the content of each replicate was transferred to another vessel that received water and a few drops of black ink to allow the flotation and to facilitate the visual contrast of organisms with the water. The surviving adults were visually counted. The tests in each soil occurred individually, however, both moisture treatments were tested simultaneously with the same batch of animals. All treatments were performed in five replicates plus an extra replica, which was used to assess the soil moisture and pH at the end of the bioassays.

To evaluate the reproduction and growth of collembolans, each vessel was photographed from a superior view in high resolution. The photos were analyzed through ImageJ® software (Alves et al. 2014), where all juveniles from each experimental unit were counted through the manual counting tool. In addition, the length (from the front head to the end of the abdomen) of generated collembolans was mensurated on the images by using the same software. For this, at least 30 juveniles of each replicate were randomly selected. For the replicates with <30, all juveniles were measured. In each replicate, the collembolans were classified in three distinct body sizes: small (≤3 mm), medium (>3 and ≤4.5 mm) and large (>4.5 mm). The results were presented in percentage of each size class.

Data analysis

The normality and homoscedasticity of the data from chronic toxicity tests were checked through the *Kolmogorov-Smirnov* and *Bartlett* tests, respectively. When necessary, data logarithmic transformations were used to meet the analysis of variance (ANOVA) assumptions. For reproduction and growth data, the differences between treatments were tested by the one-way ANOVA ($p < 0.05$). When statistical differences were detected in reproduction data, the treatments containing fipronil were compared with the control by using the post-hoc *Dunnett's* test ($p < 0.05$), allowing to determine the non-observed effect concentration (NOEC) and lowest observed effect concentration (LOEC). Differences between the average percentages of small, medium and large juveniles at 30% (or 45%) and 60% WHC, in the same soil type and concentration, were checked via *Tukey's* test ($p < 0.05$).

The effect concentrations causing a reduction in the collembolan's reproduction or growth in 10 or 50% (only reproduction), in relation to the control (EC_{10} or EC_{50}, respectively), were estimated by using non-linear regression models (Environment Canada 2007). The EC values were estimated based on measured concentrations (Table 1). Significant differences between EC_{50} values (reproduction data) from different soil types (at 60% WHC) or moisture contents (within the same soil), were assumed when their 95% confidence intervals did not overlap (Jegede et al. 2017).

A two-way ANOVA was used to evaluate the interaction between the soil moisture and fipronil concentrations, for each soil, on the effects caused on the reproduction and growth of juveniles collembolans. All statistical analyzes were performed using the Statistica® software, version 13.5.0.17.

The risk estimation was performed through the toxicity-exposure ratio (TER) approach (EC 2002), by dividing the EC_{10} of each soil moisture scenario by the respective PEC value ($TER = EC_{10}/PEC$). A significant risk for *F. candida* was considered when $TER < 5$.

Results

The validity criteria for chronic toxicity tests with *F. candida* (ISO 2014) were met for all performed tests (Supplementary Information 2).

Fipronil caused a reduction in the number of *F. candida* juveniles in all exposure scenarios, however, the magnitude of the effects varied according to the soil type and soil moisture (Fig. 1).

At 60% WHC, the fipronil toxicity (EC_{50}-based) did not differ between TAS and Oxisol. On the other hand, higher toxicity was found in Entisol, when compared with the other tested soils. In TAS and Oxisol, the dry condition (30 and 45% WHC respectively) increased the toxicity of fipronil to collembolans, in comparison to 60% WHC (Table 3). For TAS, EC_{50} values indicated toxicity more than three times higher in the dry soil (30% WHC). For Oxisol, the toxicity at 45% WHC was twice higher than at 60% WHC. On the other hand, in Entisol, the toxicity at 60% WHC was higher when compared with 30% WHC (Table 3). The two-way ANOVA (Supplementary Information 3) showed a significant interaction between the soil moisture content and concentrations of fipronil on the collembolan's reproduction and growth for all tested soils.

In general, while the percentage of small juveniles increased in all soils with increasing fipronil concentrations, the number of juveniles with large size decreased (in TAS and Entisol) at these conditions. When comparing the different soil moisture contents (Fig. 2) for the same fipronil concentration (i.e., at 1 and 2 $mg\,kg^{-1}$), the percentage of small collembolans was significantly higher at 30% WHC than at 60% WHC in TAS and Entisol. On the other hand, no significant influence of soil moisture content on the fipronil effects on juvenile's growth was observed in Oxisol, except at the concentration of 1 $mg\,kg^{-1}$, in which the percentage of small organisms was significantly higher at 45% WHC compared to 60% WHC. For TAS, Entisol and Oxisol, the toxic effects on the juvenile's growth (based on EC_{10}) were, respectively, about 2.2, 2.4 and 2.7-fold lower at 60% WHC when compared to the respective dry condition (Table 3).

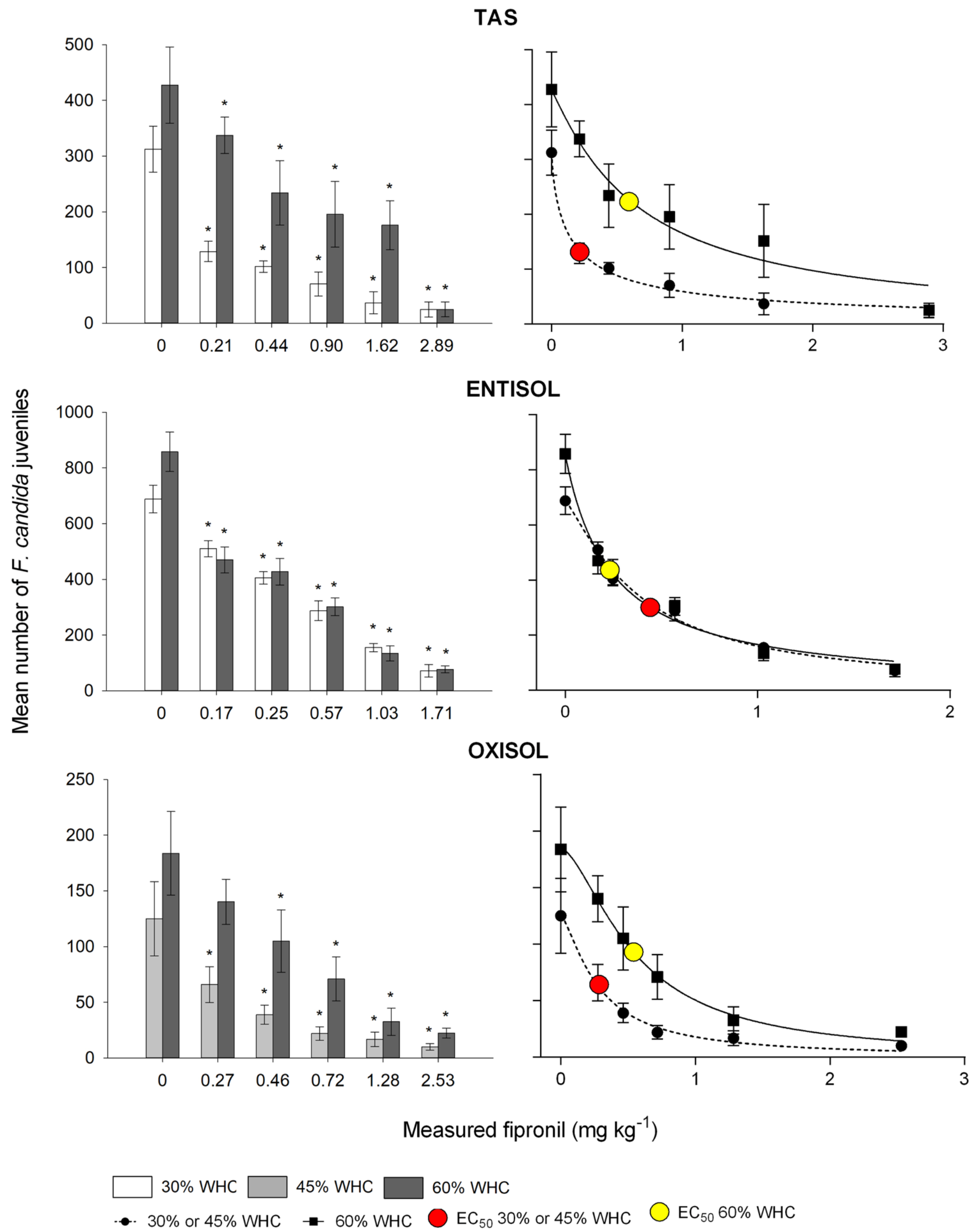

Fig. 1 Mean number (bars ± standard deviation, $n = 5$) and dose-effect curves (with effect concentrations of 50% - EC_{50}) of juveniles *Folsomia candida* generated after 28 d of exposure to different concentrations of fipronil in Entisol and Oxisol under regimes of 30 or 45 and 60% of soil water holdong capacity (WHC). Asterisks (*) represent significant reduction in the number of juveniles compared to the respective control (*Dunnett's* test, $p \leq 0.05$). In dose-effect curves, the EC_{50} values were plotted as red and yellow circles for the exposures at 30 or 45 and 60%, respectively

The estimated PEC was close to EC_{10} $TAS_{30\%\ WHC}$ and $Entisol_{60\%\ WHC}$. The TER values indicated a risk for collembolans in TAS and Oxisol only at the dry condition (TER < 5). In Entisol, however, a significant risk was found at both soil moisture contents (TER < 5; Table 4).

Discussion

Fipronil was toxic to *F. candida* even at lower concentrations, in all soils tested (Fig. 1). A decrease of 50% in the number of generating collembolans occurred at low fipronil concentrations (Table 3), depending on the soil type. The reduction in collembolans reproduction caused by the exposure to fipronil is reported in other studies (San Miguel et al. 2008; Alves et al. 2014; Zortéa et al. 2018a), corroborating our findings.

These effects are probably due to the mode of action of the insecticide on organisms that, in addition to cause the inhibition of GABA neurotransmitters, can affect the reproductive functions of collembolans. Fipronil is able to deregulate the endocrine system linked to sexual maturation and ecdysis in arthropods, causing reduced egg-laying (Cary et al. 2004; Gaertner et al. 2012). Regarding the effects of fipronil on the size of *F. candida* (Fig. 2), it was probably due to changes in physiological or biochemical processes into organisms (Ribeiro et al. 2001), which are expected because the organisms expend more energy on

Table 3 Ecotoxicological parameters for reproduction (NOEC, LOEC, EC_{50} and EC_{10}) and juvenile's growth (NOEC, LOEC and EC_{10}) from chronic toxicity assays with *Folsomia candida* exposed to increasing concentrations of fipronil in Tropical Artificial Soil (TAS), Entisol and Oxisol under two soil moisture contents (30 or 45%, and 60% of the soil water holding capacity—WHC)

Measured endpoint	Parameter	Fipronil concentration (mg kg^{-1})					
		TAS		Entisol		Oxisol	
		30% WHC	60% WHC	30% WHC	60% WHC	45% WHC	60% WHC
Reproduction	NOEC	<0.21	<0.21	<0.17	<0.17	<0.27	0.27
	LOEC	0.21	0.21	0.17	0.17	0.27	0.46
	EC_{50}	0.20	0.70	0.41	0.24	0.27	0.54
	Limits (95%)	(0.15–0.25)	(0.42–0.94)	(0.36–0.46)	(0.20–0.29)	(0.20–0.34)	(0.42–0.70)
	EC_{10}	0.01	0.08	0.04	0.01	0.07	0.12
	Limits (95%)	(0.00–0.02)	(0.00–0.16)	(0.03–0.06)	(0.00–0.02)	(0.04–0.10)	(0.05–0.20)
Growth	NOEC	<0.21	<0.21	<0.17	<0.17	<0.27	<0.27
	LOEC	0.21	0.21	0.17	0.17	0.27	0.27
	EC_{10}	0.27	0.65	0.08	0.14	0.10	0.22
	Limits (95%)	(0.07–0.47)	(0.22–1.09)	(0.04–0.12)	(0.03–0.26)	(–)	(0.10–0.33)

(–) Limits could not be estimated

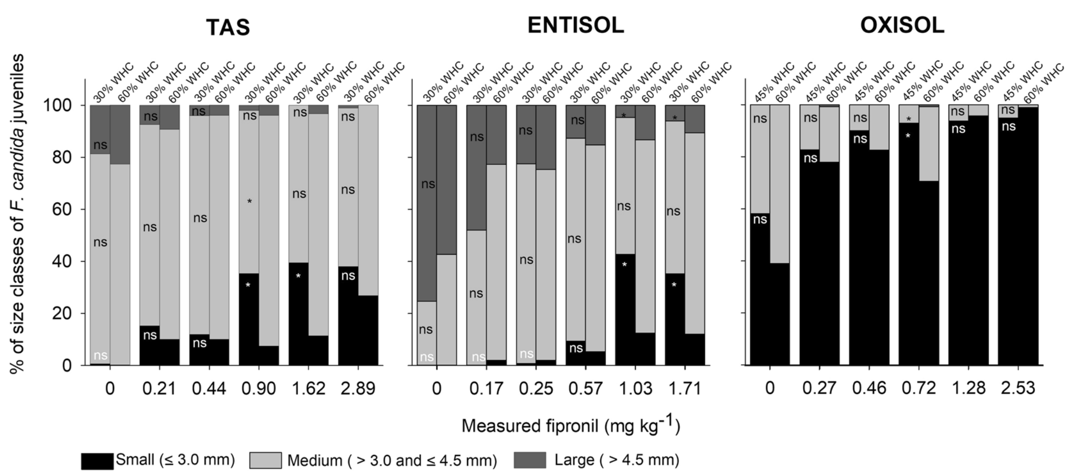

Fig. 2 Number of small (≤3 mm), medium (>3 and ≤4.5 mm) and large (>4.5 mm) *Folsomia candida* juveniles (expressed as the % of the total sample) found after 28 days of exposure to different concentrations of fipronil in TAS, Entisol and Oxisol, under 30 or 45%, and 60% of soil WHC. Asterisks (*) indicate a significant difference between treatments with the same fipronil concentration but distinct soil moisture contents, and 'ns' indicate no significant difference (*Tukey's* test - $p \leq 0.05$)

Table 4 Predicted environmental concentration (PEC) and toxicity exposure ratio (TER) for the exposure of *Folsomia candida* to fipronil in TAS, Entisol and Oxisol under different soil moisture contents (30 or 45%, and 60% of the soil water holding capacity—WHC)

Soil	WHC (%)	EC_{10} (mg kg^{-1})	PEC (mg kg^{-1})	TER
TAS	30	0.01	0.017	**0.59**
	60	0.08	0.017	5.30
Entisol	30	0.04	0.014	**2.90**
	60	0.01	0.014	**0.73**
Oxisol	45	0.07	0.017	**4.13**
	60	0.12	0.017	7.07

TER values lower than 5 (in bold) indicate significant/unacceptable risk

detoxification mechanisms, thus reducing their resources for growth (Sousa et al. 2000; Ribeiro et al. 2001, Hoffmann et al. 2003, Brodeur et al. 2017).

Based on the toxicity results, our first work hypothesis could be confirmed, since the effects of fipronil on the reproduction of *F. candida* (at 60% WHC) in Entisol (natural sandy soil) were greater than those found in TAS and Oxisol (Table 3). The reason for these findings is probably due to the influence of the adsorptive characteristics of tropical soils on the environmental fate of fipronil (Masutti and Mermut 2007; Scorza and Frando 2013). Among the main factors that influence the bioavailability and consequently the ecotoxicity of pesticides in the soil, are the clay and OM contents, the reactivity of the mineral surfaces, and the sorption equilibrium of pesticide between pore water and solid phases of the soil (Smit and Van Gestel 1998; Domene et al. 2010; Domene et al. 2012; Mandal and Singh 2013). TAS contains 1.4% OM (Table 1) based on coconut fiber, which has very rich adsorptive properties such as porous morphology, many carboxylic, hydroxylic, and carbonyl sites (Da Silva et al. 2012), with high WHC and the ability to adsorb lipophilic pesticides, such as fipronil (Tingle et al. 2003; Gunasekara et al. 2007). Oxisol has undergone intense weathering processes, having significant clay contents, high WHC (Table 1), and mineral surfaces with high amounts of iron oxy-hydroxides (Massuti and Mermut 2007), that favor chemical bonds with electronegative atoms of fipronil (F^-, Cl^-, O^{2-} and N^{3-} - Singh et al. 2014; Singh et al. 2016). Entisol has low WHC, low clay and silt contents (Table 1) and, consequently, fewer binding sites and probably low pesticide adsorption capacity, favoring greater bioavailability of fipronil in soil pores for collembolans uptaking (Van Gestel 2012; Peijnenburg et al. 2012).

Similar to our findings, Zortéa et al. (2018a; 2018b) assessed the effects of a veterinary medicine containing fipronil to collembolans and found high toxicity in Entisol when compared to TAS and Oxisol. Bandeira et al. (2019) found that the toxicity of an imidacloprid-based seed dressing formulation to *F. candida* was lower in TAS and Oxisol, compared to Entisol. Other studies evaluating the toxicity of swine manure to soil invertebrates also found that the deleterious effects are greater in Entisol compared to Oxisol (Segat et al. 2015; Maccari et al. 2016). Despite testing different contaminants, these studies also corroborated our first hypothesis, that natural sandy soils, like Entisol, enhance the ecotoxicological effects of fipronil to collembolans.

The adsorption of fipronil in the solid matrix depends on the number of adsorptive sites but also on the soil water content. According to Bobé et al. (1997), the adsorption of the fipronil on soil increases as the ratio soil/water decreases. Thus, the increase in soil moisture content may have provided an increase in the adsorption of fipronil to the soil particles (especially to clay and silt), reducing the bioavailability and consequently the toxicity for collembolans. In this way, the decrease in soil moisture content may have provided a reduced adsorption of fipronil to the soil, becoming more bioavailable and toxic for organisms in the soil pores. This could partially explain the increase in the fipronil toxicity on collembolan's reproduction in TAS and Oxisol with the decrease of soil moisture.

In addition to the relationship between fipronil bioavailability and soil water content, the lowest available water content in TAS and Oxisol may have contributed to a higher species sensitivity, because under drought conditions collembolans may have their metabolic activities reduced, which may affect their growth, compromise the survival and reproductive performance (Bursell 1970; Edney,1977; Van Gestel and Van Diepen 1997; Jänsch et al. 2005; Sławski and Sławska 2020). Some studies have indicated that the optimum soil moisture for metabolic maintenance and guarantee of the existence of the species is about 60% WHC (Van Gestel and Van Diepen 1997; Crouau et al. 1999; Bandow et al. 2014a; Bandow et al. 2014b). This may also be the explanation for the poor reproductive performance in controls at the dryer scenarios, in all tested soils (Fig. 1; Supplementary Information 2).

On the other hand, sandy soils with lower clay and silt contents, such as Entisol, present lower sorption capacities compared to soils with greater contents of these minerals (Spomer and Kamble 2010). In the presence of high available water content, the toxicity of pesticides in sandy soil depends not only on their ability to bind to adsorption sites but also on the molecule's ability to compete with water for binding sites (Harris 1964; Fishel 2003). In their study, Harris (1964) concluded that in sandy soil the high water content available in the soil pores may compete for the binding sites with the pesticides heptachlor, DDT, diazinon, V-C13 and parathion, increasing their bioavailability and, consequently, their toxicity for insects.

Collembolan size has been considered as a promising toxicity endpoint that can also be used for assessing long-term effects of contaminants (Guimarães et al. 2019; Szabó et al. 2020). Our experiments indicated that the dry condition increased the incidence of small juveniles compared to 60% WHC, in TAS and Entisol at fipronil concentrations of 1 and 2 mg kg^{-1} (Fig. 2). In addition to the possible metabolic stresses, the drier scenario e may have caused a reduction in the thickness of the water layer that surrounds the soil particles, negatively affecting the habitat of collembolans and the access to food and water, compromising their development (Coleman et al. 2004; Singh et al. 2019). From these inferences, the problem of reduction in soil moisture content becomes evident on an ecological scale for collembolans, since smaller collembolans can be more susceptible to stress and predation, resulting in an additional risk for these organisms (Guimarães et al. 2019).

In Oxisol, soil texture seems to be the main factor that influenced the collembolans growth, given the predominance of small juveniles in the control treatment, regardless of the moisture regime (Fig. 2). This probably occurred because in clayey soil, fewer porous spaces are available to be occupied by collembolans, which can impair the colonization and body development due to limited access to water and food resources (Domene et al. 2011). This assumption can also be demonstrated through the number of juveniles generated and through the proportion of medium and large-size juveniles in the control treatments of each soil (Supplementary Information 2; Fig. 2).

In the same way as our study, Hennig et al. (2020) observed that the toxicity of imidacloprid for *F. candida* in an Oxisol was greater at 45% WHC when compared to 60% WHC, while in Entisol there was no clear influence of soil moisture content (30 and 60% WHC) on the toxicity. The authors attributed their findings to the adsorptive properties of natural soils and the consequent bioavailability of the contaminant to collembolans. Other studies conducted in artificial soils also agrees with our findings in TAS. Bandow et al. (2014b) observed that the toxicity of pyrimethanil on the reproduction of *F. candida* and *Sinella curviseta* in OECD soil (sandy loam soil - 5% OM) was greater at 30% WHC compared to 50% and 70% WHC. Højer et al. (2001) observed that the toxicity of phenols to collembolans increased with the reduction of moisture content in a LUFA 2.2 soil (Loamy Soil - 6% OM). For both studies, the observed effects were related to a synergistic or additive effect of drought stress with contaminant toxicity, as well as to the adsorptive properties of the soil and the consequent bioavailability of the contaminant for collembolans, as reported in the present study.

Our estimated PECs (0.014–0.017 mg kg^{-1}) fitted in the range of fipronil concentrations found in agricultural soils by some authors (Li et al. 2015) in peanut fields. Ying and Kookana (2006) found fipronil concentrations between 0.01 and 2.06 mg kg^{-1} in field soils after three years of application. Mandal and Singh (2013) reported fipronil concentrations of 1.90 and 5.50 mg kg^{-1} in two different soils 120 d after the application. Similarly, Silva et al. (2016) found about 3.5 mg kg^{-1} of fipronil in two soils 40 d after pesticide application. These values led us to suppose that fipronil can be persistent in soils and, in some cases, soil invertebrates may be exposed to environmental levels higher than those estimated in our study.

The TER approach indicated no risk for *F. candida* exposure to fipronil at the lowest recommended dose of the commercial formulation in TAS and Oxisol under the standard soil moisture condition. However, a significant risk of fipronil was observed in Entisol, revealing that *F. candida* populations can be affected when exposed to sandy agricultural soils (Table 4). This is also probably due to the soil properties like low clay and OM content as observed to mancozeb (Carniel et al. 2019) and imidacloprid (Bandeira et al. 2019) in Brazilian soils by other authors, which seems to be a relevant finding for Brazil, since the environmental risk assessment of pesticides does not consider the soil type in the risk estimation (IBAMA 1996).

Based on the risk calculated in this study, considering the influence of abiotic factors such as soil type and soil moisture content in prospective risk assessments of pesticides in the soil seems to be essential to avoid underestimation in future scenarios.

Conclusions

Our results showed that the toxic effects of fipronil on *F. candida* reproduction are dependent on the tropical soil type, and are enhanced in the sandy soil, compared to a sandy loam artificial soil and natural clay soil. The reduction of soil moisture may increase or decrease the toxic effects of fipronil on *F. candida* reproduction, depending on the soil type. Soils with higher WHC and adsorptive properties tend to increase fipronil toxicity when dry, while those with less WHC and less favorable adsorptive properties can reduce a.i. toxicity. In all tested soils, the percentage of small juveniles increased with fipronil concentration regardless of soil moisture, but more small juveniles were generated at dry scenarios compared to the standard soil moisture in TAS and Entisol. Disregarding the climatic variables, the risk of fipronil seem to depend on the soil texture. When considering the reduction of soil moisture, an increased risk is found in soils with higher WHC.

Data availability

The data that support the findings of this study are available and should be requested by e-mail.

Code availability

ImageJ, RRID:SCR_003070; STATISTICA, RRID:SCR_014 ?213; SCAPE, RRID: Not available.

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Author contributions TBH: Conceptualization, Data curation, Formal analysis, Investigation, Writing—original draft. PRLA: Conceptualization, Formal analysis, Funding acquisition, Investigation, Project administration, Resources, Supervision, Writing—review & editing. TT: Investigation, Formal analysis. FOB: Investigation, Data curation, Formal analysis. WES: Investigation. LCC: Investigation, Data curation, Formal analysis. IKG: Investigation. DB: Resources, Supervision, Writing—review & editing.

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Compliance with ethical standards

Conflict of interest The authors declare no competing interests.

Ethical approval All procedures performed in studies involving animals were in accordance with the ethical standards of the institution or practice at which the studies were conducted (Brazilian regimentation for the scientific use of animals - Law no. 11.794).

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