Effect of Different Temperatures on the Degradation Rate and Half-Life of Termiticides in Tropical Soils under Laboratory Condition

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ABSTRACT

Soil termiticide treatment is a fundamental method to control termite population and infestation by creating a continuous barrier surrounding the structures. Dissipation of termiticides depends on half-life, degradation rate, leaching activity, and storage method. The use of termiticides without understanding their fate would lead to environmental contamination. This study determined the degradation rate and half-life of three commercially available termiticides (bifenthrin, fipronil, and imidacloprid) in soils having different textures, a sandy loam and loamy sand, under a laboratory setting. The remaining termiticides in the soils were extracted and analysed using an Ultra-Performance Liquid Chromatography (UPLC) system. It was found that bifenthrin had the highest half-life (166.88 days) and the lowest degradation rate (4.28ppm/day), compared to fipronil (56.05 days and 5.43 ppm/day) and imidacloprid (50.02 days and 5.46 ppm/day). On account of the high half-life, lower degradation rates, and good soil bonding capacities, fipronil and bifenthrin are recommended as termite control in this study. These features make fipronil and bifenthrin termiticides suitable for buildings environmental protection.

Key words: Degradation rate, half-life, imidacloprid, fipronil, bifenthrin

INTRODUCTION

Termites are destructive polyphagous insect pests, damaging household and finished products as well as plants such as sugarcane, millet, barley, and rice (Upadhyay *et al*. 2010). Of the 3106 identified termites, 363 are invasive species (Krishna *et al*. 2013). Termite infestation caused losses of between USD22 billion to USD40 billion in global property and annual losses of about USD400 million in Southeast Asia from 2007 to 2012 (Rust and Su 2012; Lee 2007). Subterranean termites attack accounted for 90% of total economic loss and 70% of building damages annually. (Kuswanto *et al*. 2015). Twelve different subterranean termite species are identified in Malaysia and Singapore comprising of genera such as *coptotermes, macrotermes, microtermes, globitermes, odontotermes,*

schedorhinotermes, and *microcerotermes*. They can be easily found inside and around buildings and structures (Lee *et al.* 2007).

Chemical termiticides, mainly soil termiticides, make up nearly 90% of all the subterranean control products, with bifenthrin and imidacloprid being significant products (Koehler *et al.* 2011). Properly applied soil-termiticide treatment should provide adequate protection for at least five years (Richman *et al.* 2006). The effectiveness of soil termiticide treatment can vary by locality (Ramakrishnan *et al.* 2000), and termiticide susceptibility can differ by species (Dhang 2011). Two critical factors in the determination of the efficacy of the soil treated with termiticides are toxicity and the mode of action.

Imidacloprid has a half-life of between 2.01 and 229 days (Anhalt *et al.* 2008; Fossen 2006; Oi 1999; Rouchaud *et al.* 1994; Sanyal *et al.* 2006; Sarkar *et al.* 2001). The half-life of fipronil ranges from 2 to 342 days (Connelly 2001; Lin *et al.* 2009a; 2009b; Ngim and Crosby 2001; Shuai *et al.* 2012; Ying and Kookana 2006). Fipronil may degrade to its primary metabolites in the form of sulfone oxidation, sulphide reduction, amide hydrolysis, and dessulfinyl photodegrade photolysis (Bobe *et al.* 1998; Hainzl and Casida 1996; Ngim and Crosby 2001; Ying and Kookana 2002). Meanwhile, previous studies report that the half-life of bifenthrin ranges from 122 to 390 days (Fecko 1999; Mulrooney *et al.* 2006), while bifenthrin is stable in soil with a high pH, and degrades at a slow rate (Kamble and Saran 2005).

Treatment of soil with termiticide can be performed during pre- and post-construction (Lee 2002), with post-construction treatment considered as dominant, representing 52% of the total treatment (Lee 2002). Soil treatment before construction involves the base of the concrete plate, whereas soil treatment after construction involves structural perimeter treatment (Peterson 2007). The minimum termiticide residue in the soil is between 2.85 years and 5.4 years, in both pre-and post-construction.

Termites must be exterminated, and structures must be protected against termite invasion (Tsunoda 2005). Non-repellent termiticides are thus more favourable than repellent termiticides, which allow poisoned termites to transfer the toxic substance to members of the colony (Hu 2005). Soil treatment creates an environment barrier with no impact on the population of termites (Forschler and Jenkins 2000). Termiticides in soil may degrade overtime, and the rate of degradation depends on environmental factors such as humidity, temperature, and soil pH (Wiltz 2012). Thus, the estimation of degradation on different commercial termiticides under field conditions could provide further useful information on soil termiticide degradation rate.

Many researchers around the world have studied the fate of bifenthrin, fipronil, and imidacloprid but these subject areas have not been carried out in a tropical climate. Therefore, the objectives of this study were to determine the degradation rates and half-life of bifenthrin, fipronil, and imidacloprid in different soils (sandy loam and loamy sand) at different temperatures (30°C and 40°C).

MATERIALS AND METHODS

Soil Sampling
Soil samples were collected from two sites, i.e. Durian Valley, Universiti Sains Malaysia (USM; 5°21.35'N; 100°18.16'E) for sandy loam and Teluk Bahang, Penang (5°26.47'N; 100°13.04'E) for loamy sand. The two types of soil chosen were based on the suggestion of Ab Majid and Hassan (2013) that 44% of termites in Penang are found in loamy sand and 56% in sandy loam soil. Only two soil types, namely sandy loam and loamy sand, have been recorded with termites. The soils were taken from the top layer (A-horizon) at approximately 10 cm. The remaining debris such as stones, vegetation, and macrofauna were removed. The soils were air-dried at room temperature (20–25°C). The soils were then sieved through a 2-mm sieve, stored at ambient temperature, and were kept air-dried. The soils were subsequently analysed for particle size, pH, and organic matter content. Soil pH was determined using a pH meter (HANNA HI 8424, Romania). The soils were mixed with distilled water at a ratio of 1:2 and left overnight to obtain the pH value (Ab Majid and Hassan 2013).

Soil Properties
The soil samples were first analysed for particle size, pH, and content of organic matter. A method proposed by Bouyoucus (1962) was applied to the soil texture. Approximately 50 g of the soil tested was mixed in a 500 mL beaker with 100 mL of 6% hydrogen peroxide (H_2O_2). The mixture was left overnight at room temperature. The beaker was then placed on a hot plate at 90°C for 10 min, followed by the addition of 50 mL of 1M NaOH. The mixture was topped up with distilled water to 400 mL and left for 20 min. Next, the mixture was stirred using a magnetic stirrer for 10 min. The mixture was subsequently transferred into a 1L measuring cylinder, followed by the addition of 1L of distilled water. The mixture was allowed to reach thermal equilibrium, at which point the temperature was recorded. The measuring cylinder was gently tilted several times for the contents to be thoroughly mixed. The hydrometer was immersed in the mixture, and the reading was recorded after 40 sec. This step was repeated after 2 h. Readings from the hydrometer and thermometer were used to obtain the percentage of sand, silt, and clay. Lastly, soil texture was determined using the United States Department of Agriculture (USDA) textural triangle. Soil pH was also determined using a pH meter by mixing the soil with distilled water at a ratio of 1:2 and left overnight (Ab Majid and Hassan 2013). Organic matter content was obtained using a loss of ignition technique.

Termiticides
The termiticides used in this study are representatives of three chemical classes: chloronicotinyl (imidacloprid, Prothor), phenyl pyrazole (fipronil, Chalcid 5.0), and pyrethroid (bifenthrin, Maxxthor). Formulated products, i.e. Prothor 200 SC (Ensystex, MAlaysia Sdn. Bhd., Kuala Lumpur), Maxxthor 100 SC (Ensystex,

Malaysia Sdn. Bhd., Kuala Lumpur), and Chalcid 5.0 SC (Hextar Chemicals Sdn. Bhd., Selangor, Malaysia) were purchased from a local distributor.

Soil Treatment
A stock solution for each termiticide was prepared in 1000 mL deionised distilled water. Three termiticide concentrations were prepared: high (2000 ppm), medium (1000 ppm), and low (500 ppm). Then, 100 mL of each termiticide from the stock solution was applied to 1000 g of soil (sandy loam and loamy sand) and were filled into two plastic containers (1 L). Each plastic container was placed in the oven at temperatures of 30°C and 40°C. Each concentration was replicated thrice. For control, 1000 g of the soil samples were treated with 100 mL of deionised water. The soil samples were collected during the first, third, ninth, and twelfth month for analysis.

Soil and Residual Analysis
Technical grade standards of bifenthrin (98.8%), fipronil (97.9%) (Sigma-Aldrich, Malaysia), and imidacloprid (99.5%, Chem Service) were used. Acetonitrile HPLC grade (Baker) was used as a solvent to dissolve all termiticides. HPLC grade acetonitrile and ultra-pure water from a Milli-Q (Millipore Asia Limited, Selangor, Malaysia) purification system were used as the mobile phase in the chromatographic analysis.

Soil samples (previously stored in a freezer below 0°C) were air-dried for 24 h. Ten g of the soil samples for each replicate was weighed in a 200 mL conical flask followed by the addition of 40 mL of acetonitrile. The flasks were covered with aluminium foil and placed in a shaker maintained at 20°C and agitated at 200 rpm overnight. The samples were allowed to stand for 1 h to allow soil particles to settle. Then, 1.5 mL of clear supernatant was filtered through a 3-cc glass syringe equipped with a 0.2 µm Acrodisc® Syringe Filter into a 2.0 mL microcentrifuge tube. Aliquots were centrifuged (Eppendorf centrifuge 5424) at 12 000 rpm for 20 min. One mLof the supernatant was transferred into a 2 mL auto-injector vial, sealed with a PTFE-lined screw cap after passing through a 3-cc glass syringe. Samples were analysed after the extraction.

Extracted termiticides were separately analysed using UPLC. Data collection and peak analysis were performed using the Waters Acquity UPLC detected by a photo-diode array (PDA) connected to a computer. A single UPLC BEH C18 column (1.7µm i.d, 2.1mm x 100mm) was used for the separation. Injection volume was set at 5.0 µL, and the flow rate at 1.0mL/min.

Degradation Rate and Half-life of Termiticides
The termiticide residue was measured following the California Department of Food and Agriculture (CDFA 2011) standard:

$$R= \frac{(Spl\ H)(Std\ C)(Std\ V)(V)}{(Std\ H)(Spl\ V)(Spl\ W)} ,$$

where, R is termiticide residue of part per million (ppm), *Spl H* is the sample peak height area, *Std C* is the standard concentration (ppm), *Std V* is the standard volume injected (µl), *V* is the volume after the injection (ml), *Std H* is the standard peak height area, *Spl V* is the sample volume injected (µl), and *Spl W* is the sample weight (g).

The half-life of a substance was calculated according to Ong *et al.* (2016) and the degradation rate of termiticides was calculated based on the decrease in concentrations between the sampling time (Lin *et al.* 2008; Mahiudddin *et al.* 2014). Statistical analysis was performed on IBM SPSS Statistics Version 22. In the degradation study, a 2-way ANOVA was performed with 99% confidence limit. A factorial analysis of variance (ANOVA) was performed using termiticide, concentration, temperature, and soil types as independent variables to determine the significant differences between the above independent variables and on termiticide residual (dependent variable).

RESULTS

Sandy loam had a higher percentage of clay (16.4%) compared to loamy sand (11%),while loamy sand had a higher percentage of sand (84.6%) and silt (4.4%) compared to sandy loam (Table 1). Sandy loam appeared to be slightly acidic (pH= 4.4) compared to loamy sand (pH= 4.8,Table 1).

The degradation rate of termiticides was more significant at a high level of concentration (2000 ppm) compared to the medium (1000 ppm) and low (500 ppm) concentrations. Meanwhile, the half-life of termiticides tested in the laboratory study showed no clear pattern (Table 2).

TABLE 1
Soil characteristics for sandy loam and loamy sand used in this degradation study
(mean±standard deviation)

Characteristics	Sandy loam	Loamy sand
pH	4.4±0.05	4.8±0.04
Organic matter content (%)	11.4±0.14	8.5±0.45
Sand (%)	80.0	84.6
Clay (%)	16.4	11.0
Silt (%)	3.6	4.4

TABLE 2

Degradation rate and half-life of bifenthrin, fipronil and imidacloprid in the laboratory degradation study

Soil	Termiticide	Concentration level	Temperature (°C)	Degradation rate (ppm/day)	Half-life (days)
Sandy loam	Bifenthrin	High	30	5.34	70.33
			40	4.28	166.88
		Medium	30	2.67	71.54
			40	2.45	114.26
		Low	30	1.31	82.92
			40	1.26	100.51
	Fipronil	High	30	5.49	33.19
			40	5.43	56.05
		Medium	30	2.75	36.52
			40	2.71	57.12
		Low	30	1.37	40.60
	Imidacloprid	High	30	5.49	33.19
			40	5.46	50.02
		Medium	30	2.75	36.52
			40	2.74	45.25
		Low	30	1.37	40.60
			40	1.36	55.51
Loamy sand	Bifenthrin	High	30	5.35	69.41
			40	4.28	166.88
		Medium	30	2.65	75.42
			40	2.45	114.26
		Low	30	1.30	88.10
			40	1.26	100.51
	Fipronil	High	30	5.49	33.19
			40	5.44	54.10
		Medium	30	2.75	36.52
			40	2.72	55.59
		Low	30	1.37	40.60
			40	1.36	53.13
	Imidacloprid	High	30	5.49	33.19
			40	5.39	63.94
		Medium	30	2.75	36.52
			40	2.69	65.46
		Low	30	1.37	40.60
			40	1.35	62.38

The termiticide residuals in the laboratory study are depicted in Figure 1. Over time, the residual of all termiticides decreased. Bifenthrin in the sandy loam at 30°C (SLBH30) showed the highest residual in the first month (2327.94 ppm), while almost all imidacloprid indicated a low residual in the first month (average 32.06 ppm). Bifenthrin remained high compared to fipronil and imidacloprid during the 12 months. Factorial ANOVA analysis showed that the difference between the month and termiticide degradation was significant (F=77.38; df=3; P=0.00). The residues in the first month were higher, decreasing progressively until the twelfth month. There was also a substantial difference in the termiticides tested (F= 176.66; df=2; P=0.00). Bifenthrin residual in the soil remained high
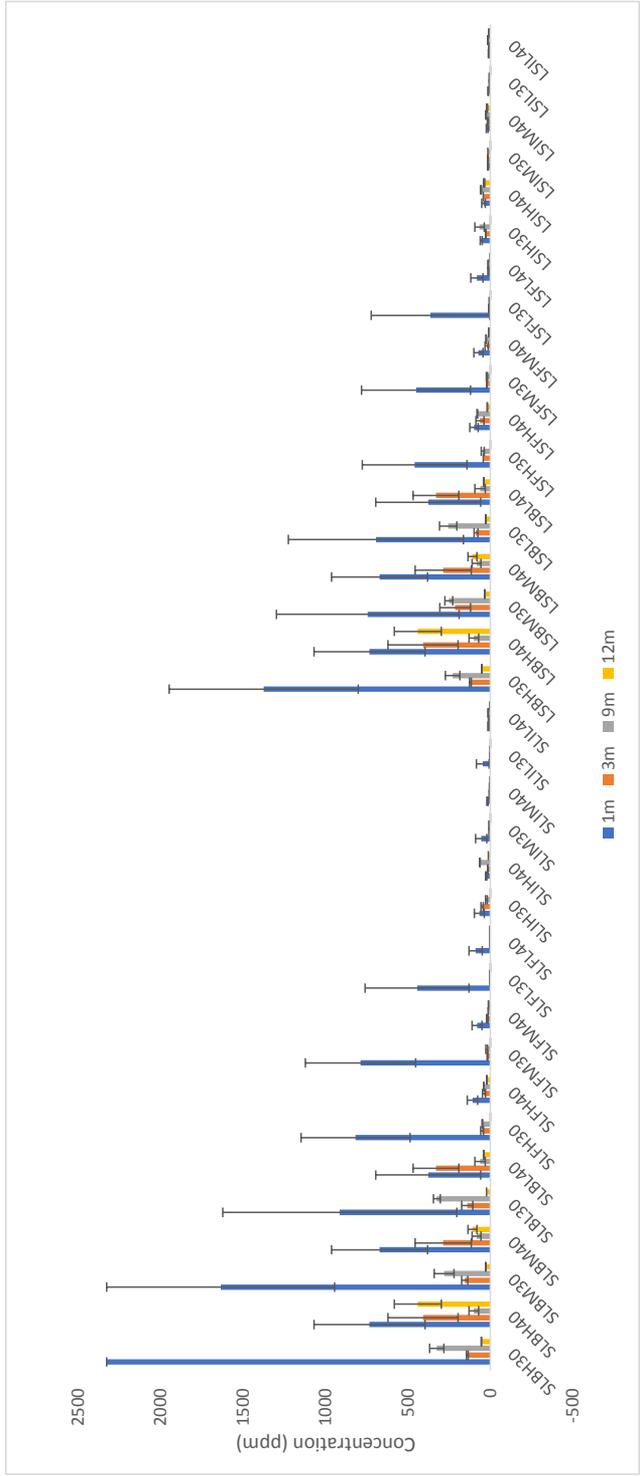
Figure 1. The degradation patterns of termiticides tested in different soil types (sandy loam and loamy sand) and temperature (30°C and 40°C) within 12 months for laboratory degradation study

*SL: sandy loam; LS: loamy sand; F: fipronil; B: bifenthrin; I: Imidacloprid; H: high concentration; M: medium concentration; L: low concentration

compared to imidacloprid and fipronil. Termiticides did affect the termiticide residual during the experiment. Termiticides concentration revealed a significant difference between termiticides (F= 19.06; df= 2; P= 0.00). The degradation rate of termiticides was higher at a high concentration level compared to medium and low concentrations levels.

The combination of month, termiticide and temperature significantly affected termiticide degradation (F= 5.69, df= 6, P= 0.00) although temperature did not affect the degradation individually (Table 3). A lower termiticide concentration at a higher temperature (40°C) took a more prolonged time (months) to degrade. Furthermore, the concentration of the termiticide was also dependent on the type of termiticide used. The concentration of bifenthrin was higher in the lower temperature (30°C) in the first month compared to fipronil and imidacloprid (Figure 1).

TABLE 3
Effects of month, termiticide and layer on degradation rate in the field degradation study

Source	df	Mean square	F	p value
Month	4	2190.81	985.73	**0.001**
Termiticide	2	5983.84	2692.36	**0.001**
Layer	1	0.24	0.11	0.74
Month * Termiticide	8	667.40	300.29	**0.001**
Month * Layer	4	2.18	0.98	0.43
Termiticide * Layer	2	2.38	1.07	0.35
Month * Termiticide * Layer	8	2.20	0.99	0.45

The termiticide degradation pattern in sandy loam soil within 12 months is illustrated in Figure 2. In all the months tested, bifenthrin showed the highest concentration compared to imidacloprid and fipronil. The concentration of bifenthrin decreased substantially in the third month after treatment. During the first and last months of the experiment, the level of imidacloprid was the lowest compared tobifenthrin and fipronil. Loamy sand soil showed similar patterns with sandy loam in terms of termiticide degradation. During the first three months, bifenthrin showed a higher rate of degradation and gradually degraded over time. Bifenthrin also demonstrated an increased level of soil residue. In contrast, imidacloprid showed the lowest concentration from the first month to the twelfth month (Figure. 3).
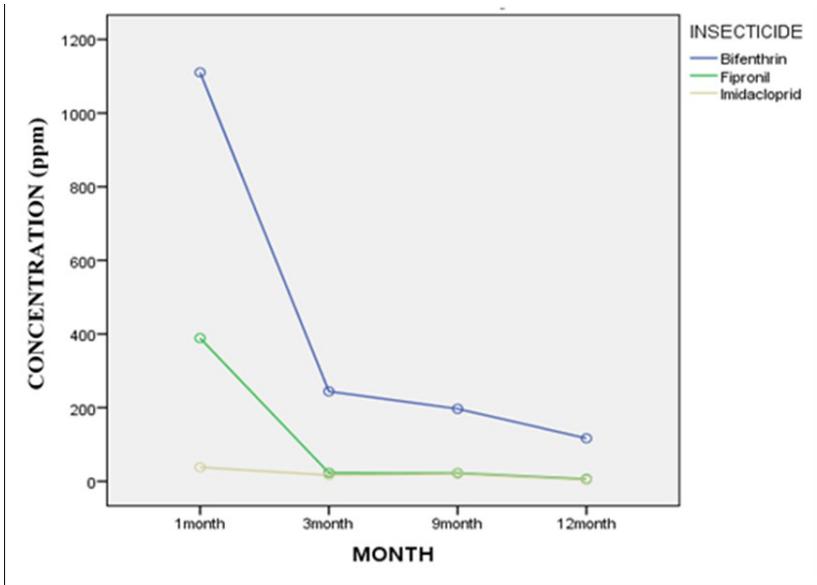
Figure 2. Degradation patterns of termiticides (bifenthrin, fipronil and imidacloprid) in sandy loam soil in the laboratory degradation study
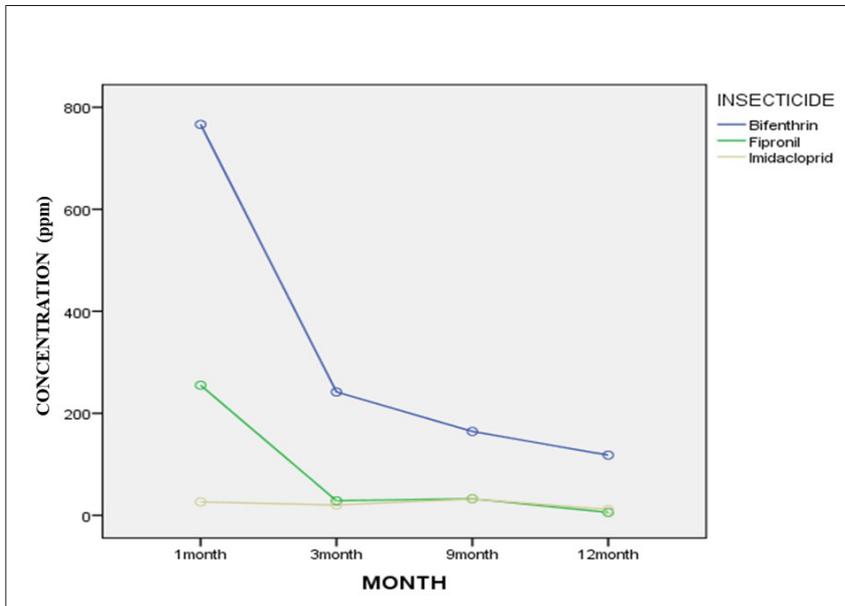

Figure 3. Degradation patterns of termiticides (bifenthrin, fipronil and imidacloprid) in loamy sand soil for laboratory degradation study

DISCUSSION

A laboratory degradation study found that bifenthrin had the highest concentration under all the conditions tested over time, compared to fipronil and imidacloprid. Bifenthrin was the most persistent termiticide as it decreased by less than 50% over the year (Horwood 2007). In previous studies, the half-life for field degradation was reported to be 122 to 345 days (Fecko 1999) and 13 months (Mulrooney et al. 2006). A study by Manzoor and Pervez (2017) in sandy loam and sandy clay loam soils following three years of application also reported bifenthrin as being stable over time. However, Su *et al*. (1999) and You *et al*. (2013) obtained contradictory results concerning bifenthrin. Bifenthrin was reported to possess less protection amongst other pyrethroids (Su *et al*. 1999), and dissipated at 84.6% for 30 days after treatment (You *et al.* 2013). Several factors affected the degradation process of pyrethroids such as the types of pyrethroids, pH and temperature, soil types, climate, and size of microbial populations (Gupta and Gajbhiye 2008; Tariq *et al.* 2017). Soil microbial activity has a vital role in the degradation of termiticides (Roy and Singh 2006; Singh *et al.* 2008). According to Sharma and Singh (2012), bifenthrin dissipates more quickly compared to sterile soil. The half-life values in the sterile and non-sterile soils were 330 and 147 days, respectively. This vast difference in the half-life values in both soils showed that microbial degradation was substantial in bifenthrin dissipation (Sharma and Singh 2012). Analysis of the HPLC showed that bifenthrin had a higher persistence than fipronil in sandy loam and sandy clay loam soils (Manzoor and Pervez 2017).

The half-life of termiticides varied according to various factors. The half-life for fipronil was reported to be within 2 to 342 days (Connelly 2001; Lin *et al*. 2009a, 2009b; Ngim and Crosby 2001; Shuai *et al*. 2012; Ying and Kookana 2006). Initial fipronil concentrations influenced the rate of fipronil degradation. Fipronil rapidly dissipated at a higher concentration and disappeared completely at a lower concentration (Manzoor and Pervez 2017). Therefore, the application of fipronil at the highest label rate caused low degradation rates (Manzoor and Pervez 2017; Saran and Kamble 2008). Fipronil tested in the sandy loam and sandy clay loam soils had a higher half-life, i.e. 270 to 555 days (Manzoor and Pervez 2017). Accordingly, Zhu *et al*. (2004) have also noted a slower degradation of fipronil in clay loam soil.

The fipronil half-life decreases with increasing temperature (Ying and Kookana 2002; Zhu *et al*. 2004). An extensive study has revealed that fipronil degradation is faster at 35°C than 25 °C (Zhu *et al*. 2004). Temperature could, therefore, affect fipronil degradation. Another factor in the degradation of fipronil in the soil is microbial activities (Ying and Kookana 2002; Zhu *et al*. 2004). Soil microbes consume fipronil as a source of energy, carbon, and/or nitrogen during the microbial degradation process. Inhibition of microbial soil activities may, therefore, prolong fipronil persistence at high application rates (Ying and Kookana 2006).

Imidacloprid was less persistent compared to fipronil and bifenthrin. Horwood (2007) demonstrated the least persistence in imidacloprid, decreasing

by about 95% by the end of the experiment. The half-life report was between 2.01 and 229 days (Anhalt *et al*. 2008; Fossen 2006; Oi 1999; Rouchaud *et al.* 1994; Sanyal *et al*. 2006; Sarkar *et al*. 2001). Gold *et al*. (1996) have reported that over time, termiticides have become less effective. Gold *et al*. (1996) further demonstrated that in 180 days, concentrations of all termiticides (chlorpyriphos, imidacloprid, and deltamethrin) in the trials decreased significantly (wt:wt basis in soil).

In this study, bifenthrin was persistent compared to fipronil and imidacloprid. The above mentioned two termiticides showed slightly different half-life values, with bifenthrin being between 122 to 345 days (Fecko 1999), and fipronil between 2 to 342 days (Connelly 2001; Lin *et al*. 2009a, 2009b; Ngim and Crosby 2001; Shuai *et al.* 2012; Ying and Kookana 2006). Therefore, the different results achieved between field studies and laboratory studies are determined by the half-life of the termiticide. Furthermore, the method used for termiticide treatment also influenced the termiticide concentration. For the field study, termiticides were treated by using the trenching method at 2500 ppm, while in the laboratory study, the termiticide was treated in a small amount and placed in plastic containers at 2000 ppm, 1000 ppm, and 500 ppm.

CONCLUSION

In the laboratory study, bifenthrin had a higher half-life and a lower rate of degradation than fipronil and imidacloprid. Thus, fipronil and bifenthrin were persistent compared to imidacloprid. Soil types and temperature did not have a significant impact on the degradation and bioavailability of the termiticides tested in this study. In this study, the percentage of clay in sandy loam did not show much difference from the loamy sand. Therefore, a study on other types of soils with a higher difference in clay percentage might provide new information concerning termiticide degradation. Besides, a degradation study should also be done on other commercially available termiticides such as chlorantraniliprole and chlorpyrifos. In addition, the bioavailability of termiticides should be tested on other beneficial insects, for instance, bees, ants and butterflies rather than only on termites.

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