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Soil Dissipation and Leaching Behavior of a Neonicotinoid Insecticide Thiamethoxam

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Abstract Persistence and leaching of thiamethoxam in soil were studied under laboratory conditions. The persistence studies carried out at two fortification levels and under three moisture regimes revealed that thiamethoxam persisted beyond 90 days in all the treatments with half-life varying from 46.3 to 301.0 days. Under dry conditions, the dissipation was faster at 10 mg kg^{-1} level as compared to 1 mg kg^{-1}, whereas the reverse trend was observed under field capacity moisture and submerged conditions. The effect of moisture was prominent and longer persistence was observed under dry conditions ($t_{1/2}$ 200.7–301.0 days) followed by field capacity moisture ($t_{1/2}$ 91.2–94.1 days) and submerged condition ($t_{1/2}$ 46.3–75.3 days). The leaching experiment carried out under laboratory conditions showed that on leaching the soil column with water equivalent to 65 cm rainfall, 66–79% of the applied thiamethoxam was recovered from leachate and no residues were detected in soil. Results showed that thiamethoxam has a potential to leach down under heavy rainfall conditions.

Keywords Thiamethoxam · Dissipation · Moisture regimes · Leaching · Alluvial soil

Crop protection is an integral part of modern agriculture with pesticide application as a major component. After application, a pesticide finds its way into soil by spray drift, washing of plant surfaces by rain, etc. Pesticides in soil may be taken up by plants, degraded into other chemical forms, or leached downward, possibly, to groundwater. Many pesticides bind strongly to soil and are therefore immobile. If the pesticide is not readily degraded and moves freely with water percolating downward through the soil, the likelihood of it reaching groundwater is relatively high. Leaching of pesticides to ground water is a cause of concern as the groundwater is the major source of drinking water especially in the developing countries. To ensure the safety of groundwater it is necessary to study the dissipation and mobility behavior of pesticides before recommending them for agricultural use.

The insecticide thiamethoxam (3-(2-chloro-thiazol-5-yl methyl)-5-methyl-[1,3,5] oxadiazinan-4-ylidene-*N*-nitroamine) (Fig. 1) is a new nitromethylene derived neonicotinoid with contact, stomach and systemic activity. It has high water solubility (4.1 g L^{-1} at 25°C), low octanol water partitioning coefficient (0.74) and low vapor pressure (4.95×10^{-11} mmHg at 25°C). It is active against sucking and chewing insects like aphids, whiteflies, plant hoppers, thrips, beetles, etc. that attacks various crops like rice (Krishnaiah et al. 2003), maize (Drinkwater 2003), cotton (Satpute et al. 2001), vegetables (Misra 2002; Brian et al. 2004), mango (Patel et al. 2003), etc. Though much work has been carried out on its bioefficacy as seed treatment or foliar spray, little published information is available on its persistence and leaching behavior. Therefore, laboratory studies were conducted to assess persistence and leaching of thiamethoxam in soil.

Materials and Methods

For laboratory studies, soil was collected from the plough layer (0–15 cm depth) from the research farm of Indian Agricultural Research Institute, New Delhi, India, with no history of pesticide application. Soil was air-dried in the

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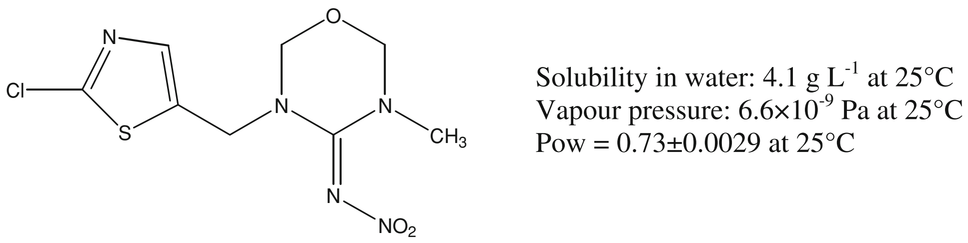


Fig. 1 Chemical structure and properties of thiamethoxam

Solubility in water: 4.1 g L^{-1} at 25°C
Vapour pressure: 6.6×10^{-9} Pa at 25°C
Pow = 0.73±0.0029 at 25°C

shade, ground, sieved through a 2-mm mesh screen. The physico-chemical properties of the soil (type inceptisol) were: pH 7.69, organic carbon 0.501%, clay 5.0%, sand 77.5%, silt 17.5%, texture sandy loam and 20% moisture content at field capacity.

Analytical grade thiamethoxam was supplied by Rallis India. Two commercial formulations of thiamethoxam *viz* Cruiser (70 WS) and Actara (25 WG) used in the experiment were purchased from the local market. Standard stock solution (1,000 μg mL^{-1}) of analytical grade thiamethoxam was prepared in HPLC grade acetonitrile. Solutions of lower concentrations were obtained by serially diluting the stock solution with acetonitrile. Similarly, fresh stock solutions (~1,000 μg AI mL^{-1}) of formulations were prepared in distilled water. Organic solvents like hexane, acetone and dichloromethane were glass distilled before use. Analytical grade sodium chloride and sodium sulfate were procured from Qualigens Fine Chemicals. HPLC grade solvents were procured from Merck India Ltd, filtered and de-gassed prior to use.

Persistence of thiamethoxam in soil under laboratory condition was studied using analytical grade material at two concentration levels (10 and 1.0 mg kg^{-1}) and three moisture regimes (submerged, field capacity and air dry condition). The treatment of soil with pesticide was carried out in such a way that there was uniform distribution of pesticide without adversely affecting the soil microbial activity. Initially, soil was fortified at 100 mg kg^{-1} level. Soil (100 g) was taken in a glass beaker and required quantity of standard stock solution (1,000 μg mL^{-1}) of thiamethoxam was added. Additional acetone was added to dip the soil, stirred with glass rod for uniform distribution of pesticide and then left undisturbed till complete evaporation of acetone. The dry soil was again mixed. This fortified soil was diluted with untreated soil in the ratio 1:9 to get 10 mg kg^{-1} fortification level. Part of the fortified soil at 10 mg kg^{-1} level was further diluted with untreated soil in the ratio 1:9 to get 1 mg kg^{-1} level of fortification. At each stage of dilution, the soil was mixed thoroughly. The homogeneity of treated soils was tested by randomly drawing three samples from each levels of fortification (10 and 1.0 mg kg^{-1}) and analyzing them. At 1 mg kg^{-1} level the replicated samples gave recovery of 90 ± 3% and at 10 mg kg^{-1} level recovery obtained was 93 ± 4%. Since there was not much variation among replicates, the treated soil was considered homogeneous. The treated soil (20 g) was transferred into beakers, which were divided into three sets for each fortification level. The soil in one set was brought to the field capacity moisture level by adding 4 mL water into each beaker. In the second set, enough water was added to completely submerge the soil and 2 cm water level above the soil surface was maintained through out the experimental period. The third set was maintained dry with no water addition. A set of untreated control soil samples for each moisture regime was also maintained simultaneously. All the beakers were weighed and kept in incubation chamber at 25 ± 2°C. Relative humidity inside the incubator was >95%. The water lost was replenished daily by weighing the beakers. Samples in triplicate for each treatment were drawn at different time intervals and analyzed for thiamethoxam residues.

Laboratory leaching experiments were carried out in triplicate in packed columns using analytical grade thiamethoxam and its two commercial formulations (Actara 25 WG and Cruiser 70 WS) under saturated flow conditions. Initially soil (10 g) was fortified with 1,000 μg of thiamethoxam separately for analytical grade material and the two formulations. PVC columns (50 cm long × 7 cm ID) were cut longitudinally into two halves and then rejoined using adhesive tape, which allowed easy separation and recovery of the intact soil core after the leaching experiment. One end of the column was capped with perforated polyethylene sheet. Dry soil (~1,250 g) was added slowly, tapping the column intermittently for compact packing up to the height of 25-cm. Soil of the column was wetted by dipping the lower end of the column in the water and allowing it to rise upward by capillary action. Next day the column was hanged vertically for 30 min to drain out the excess water. Fortified soil (10 g) was placed on top of the column and the leaching started. Water (about 2.5 L) was passed through the column under natural flow condition (~0.7 mL min^{-1}) and five leachate fractions (~500 mL) were collected. A constant water head (2 cm) was maintained above the soil surface throughout the leaching experiment. After leaching, the soil column was retrieved and divided into five cores of 5 cm each. All the soil from each core was processed and analyzed. Two untreated control columns were maintained simultaneously.

The leachate fractions were transferred to separatory funnel and 50 g sodium chloride was added and dissolved. The aqueous phase was then partitioned with dichloromethane (3 × 30 mL). Combined dichloromethane phase was dried by passing through anhydrous sodium sulfate. Soil samples collected from different experiments were taken in beaker and enough acetone was added to dip the soil. The samples were stirred with glass rod and kept for 30 min with intermittent shaking. The contents were filtered and soil was transferred back and re-extracted two more times. Acetone extracts were pooled and concentrated using rotary evaporator. The concentrated extract was transferred to separatory funnel, diluted with saline solution (200 mL, 10%) and partitioned thrice using dichloromethane (3 × 30 mL). The dichloromethane phases were combined, passed through anhydrous sodium sulfate and evaporated to dryness.

Extracts obtained from different studies were concentrated to dryness using rotary evaporator and the residues were re-dissolved in HPLC grade acetonitrile. Residues of thiamethoxam were estimated by high-pressure liquid chromatography (Waters) equipped with RP-18e column (100 × 4.6 mm, 3.5 μm, Chromolith^{TM}, Merck), variable UV-visible detector (model 484), binary pump (model 501) and Rheodyne injector (20-μL loop). The operating parameters were: λ 242 nm and acetonitrile-water gradient solvent system (Table 1). Under these conditions the retention time of thiamethoxam was 4.23 min. The minimum detection limit was 0.1 μg mL^{-1} (2 ng) with linearity range 0–100 μg mL^{-1} (0–2,000 ng).

The half-life values were calculated based on first order dissipation kinetics, $C_t = C_0e^{-Kt}$, where C_t = concentration after time t, C_0 = apparent initial concentration and K = rate constant. The fitness of the data to first order kinetics was confirmed by testing the statistical significance of correlation coefficient. The half-life values were calculated from dissipation constant calculated from regression analysis. The percent dissipation for different time intervals was calculated from persistence data. Differences among treatments were ascertained by subjecting transformed (angular transformation) percent dissipation data to statistical analysis by Analysis of Variance (ANOVA) for factorial experimental design.

Table 1 Solvent gradient conditions used for the analysis of thiamethoxam

Time	Flow (mL min^{-1})	% Water	% Acetonitrile
0	1.0	80	20
6	1.0	65	35
12	1.0	50	50
15	1.0	80	20

Results and Discussion

The recoveries of thiamethoxam from water samples fortified at 0.01 and 0.1 μg mL^{-1} varied from 92% to 98%. Recoveries from soil fortified at 0.1 and 1.0 μg g^{-1} were 97–100% for air dry and field capacity samples and 90–93% for submerged samples.

The persistence of thiamethoxam in soil was studied at two concentration levels under three moisture regimes. The average initial deposits of thiamethoxam following treatment of soil at 1 and 10 mg kg^{-1} level varied from 0.90 to 1.13 μg g^{-1} and 9.30–11.04 μg g^{-1}, respectively. The residues of thiamethoxam persisted beyond 90 days and the dissipation in different treatments varied from 20.5% to 77.8% (Table 2). The dissipation of residues was biphasic with very fast dissipation during initial period followed by slower loss (Fig. 2). However, each phase followed first order kinetics (Correlation coefficient (*r*) varying from 0.88 to 0.99). In general, pesticide dissipation from various substrates like soil, plant, water follow single phase first order rate kinetics (rate of dissipation dependent on pesticide concentration at that time), if environmental conditions like temperature, moisture, microbial activity and ratio of pesticide molecule in adsorbed and free form remains constant during experimental period. However, it seldom happens under field condition. Slight changes in environmental conditions may not have significant effect on rate of dissipation but wide variations for longer period will certainly affect it. This results in multiphasic dissipation with first order dissipation within a phase. Multiphasic dissipation can also occur under controlled laboratory experiments, where the environmental conditions are properly maintained, due to adsorption process. After application of pesticide in soil, major amount is in free form. With passage of time, pesticide molecules get adsorbed on soil particles and after equilibrium a fixed ratio between concentration of adsorbed and free form of pesticide is maintained. However, adsorption is slow process, achieving equilibrium may take from hours to few days. Thus during initial period, major portion of the applied pesticide remains in free form resulting in higher rate of dissipation. Once the adsorption equilibrium is achieved, the rate of dissipation is controlled by desorption process. Thus slower adsorption can result in biphasic dissipation of pesticides. The half-life of thiamethoxam in different treatments varied from 16.1 to 115.5 days during initial fast phase and 60.2–376.3 days during the slower second phase. The half-life values calculated from forced single phase first order rate dissipation kinetics (overall in Fig. 2) varied from 46.3 to 301.0 days. Thus it is evident that single-phase first order kinetics underestimates the rate of dissipation during initial period and overestimates it during later period. Karmakar et al. (2006) have conducted a laboratory study on the persistence of thiamethoxam at 1.0 μg g^{-1} fortification level and field capacity moisture regime for 30 days period. They have reported the half-life of 13.1–25.5 days in four different soils of India based on single-phase first order dissipation kinetics. In sandy loam soil of Delhi, the reported half-life of 16.9 days is quite close to 16.1–17.1 days which we observed under field capacity and submerged condition, respectively, during initial period. However, during later period we observed that dissipation of thiamethoxam slowed down considerably, thus increasing its persistence beyond 90 days under all the moisture conditions. Karmakar et al. (2006) in their study have underestimated the persistence of thiamethoxam in soil because of the short study period. The biphasic dissipation has also been reported for acetochlor by Ma et al. (2004) and endosulfan by Whitmyre et al. (2004).

Moisture plays an important role in dissipation of pesticides in soil, as it tends to increase the rate of all loss processes. Under field condition during cropping season the normal moisture regime is expected to be field capacity moisture. Under field capacity moisture regime, thiamethoxam was found to be moderately persistent with overall half-life of 91.2–94.1 days (16.1–28.7 days during initial 10 days and 136.8 days during later period). Further increase in moisture level to submerged condition increased the rate of dissipation. The overall half-life values decreased from 91.2 to 94.1 days under field capacity to 46.3–75.3 days under submerged condition (Fig. 2). Under air-dry condition, the rate of dissipation was very slow and only 20–27% loss was recorded on 90th day. The overall half-life values varied from 200.7 to 301.0 days. Thus, the trend of persistence among different moisture regime was dry > field capacity > submerged and the differences were statistically significant (Fig. 3). This type of trend could be attributed to effect of moisture on the major processes involved in the loss. It seems that microbial degradation is a major route of loss of thiamethoxam in soil. Under dry condition, where microbial activity is expected to be negligible, the thiamethoxam dissipation was slowest. Slower dissipation of pendimethalin (Kulshrestha and Singh 1992) and chlorothalonil (Choudhury and Awasthi 2000) from dry soil has been reported. Under adequate moisture conditions (field capacity and submerged) the microbial activity is expected to be optimum and therefore the thiamethoxam dissipation was faster. The differences among field capacity and submerged moisture condition could be attributed to the type of microbes involved in degradation. The microbial population is expected to be primarily aerobic under field capacity moisture and anaerobic under submerged condition. It seems that anaerobic microbial population is more efficient in degradation of thiamethoxam than the aerobic microbial population. Similar observations have been reported for pendimethalin (Kulshrestha and Singh 1992), and chlorothalonil (Choudhury and Awasthi 2000).

Table 2 Persistence of thiamethoxam in soil fortified at 1.0 and 10.0 mg kg^{-1} levels and maintained at dry, field capacity and submerged moisture regimes under laboratory conditions

Moisture regime	Treatment (mg kg^{-1})	Residues ± SD[a] (μg g^{-1})							
		0 days	3 days	7 days	15 days	30 days	45 days	60 days	90 days
Dry	1	1.13 ± 0.05	–[b]	1.05 ± 0.01 (7.1)[c]	1.03 ± 0.01 (9.1)	1.00 ± 0.03 (11.6)	0.99 ± 0.04 (12.2)	0.96 ± 0.02 (15.4)	0.90 ± 0.02 (20.5)
	10	11.04 ± 0.51	–	10.46 ± 0.25 (5.3)	10.22 ± 0.05 (7.4)	9.53 ± 0.35 (13.7)	9.00 ± 0.24 (18.5)	8.73 ± 0.09 (20.9)	8.02 ± 0.29 (27.4)
Field capacity	1	0.98 ± 0.03	0.79 ± 0.02 (18.6)	0.72 ± 0.02 (26.5)	0.65 ± 0.02 (32.9)	0.59 ± 0.04 (39.6)	0.55 ± 0.03 (43.4)	0.48 ± 0.02 (51.1)	0.45 ± 0.06 (54.1)
	10	10.05 ± 0.18	9.24 ± 0.32 (8.2)	8.44 ± 0.14 (16.0)	7.55 ± 0.26 (24.9)	6.69 ± 0.44 (33.5)	5.86 ± 0.21 (41.7)	5.48 ± 0.13 (45.5)	5.14 ± 0.23 (48.9)
Submerged	1	0.90 ± 0.03	0.75 ± 0.02 (16.8)	0.67 ± 0.03 (25.4)	0.48 ± 0.03 (47.1)	0.41 ± 0.02 (54.1)	0.37 ± 0.02 (58.6)	0.32 ± 0.02 (64.2)	0.20 ± 0.01 (77.8)
	10	9.30 ± 0.41	7.05 ± 0.12 (24.2)	6.17 ± 0.31 (33.7)	5.70 ± 0.10 (38.7)	5.23 ± 0.09 (43.8)	4.69 ± 0.27 (49.6)	3.91 ± 0.27 (57.9)	3.49 ± 0.05 (62.5)

[a] Standard deviation

[b] Samples not drawn

[c] Figures in parentheses shows percent dissipation

Fig. 2 Dissipation of thiamethoxam from soil under laboratory conditions

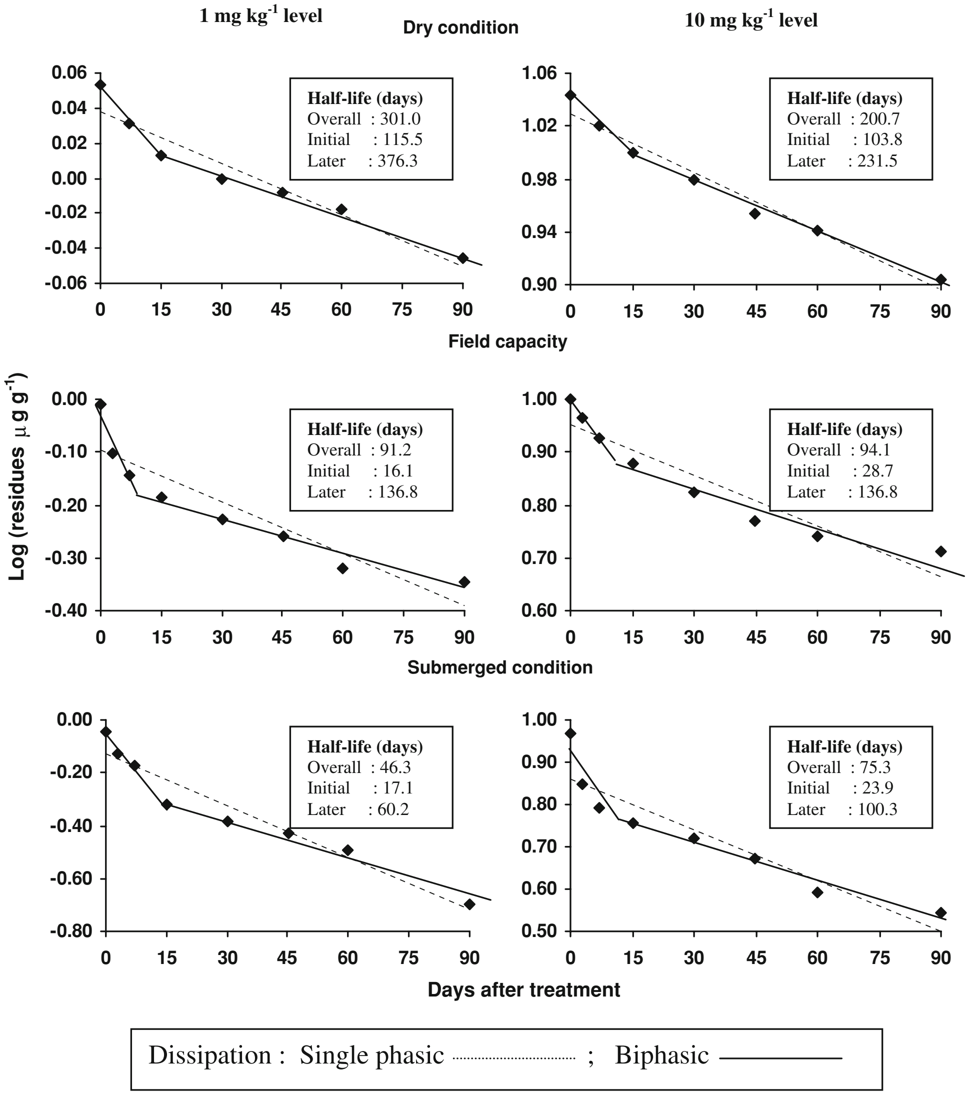
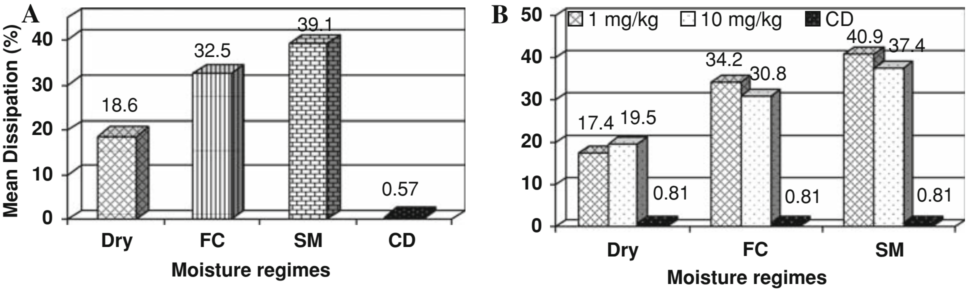


Fig. 3 Effect of (**A**) moisture regimes and (**B**) level of treatment on dissipation of thiamethoxam from soil under laboratory condition



Dissipation : Arc sin transformed values.
Dry : Air dry, FC : Field capacity, SM : Submerged;
CD : Critical difference at 0.1%.

The effect of the level of treatment (rate of application) on the dissipation of thiamethoxam in soil varied among the moisture regimes and the differences were statistically significant. Under field capacity and submerged condition, the dissipation in both the phases were faster at low than high level of treatment (Fig. 2) and the differences in dissipation were statistically significant (Fig. 3). The observed overall half-life values varied from 46.3 to 91.2 days (first phase 16.1–17.1 days and second phase 60.2–136.8 days) at 1 mg kg^{-1} levels as compared to 75.3–94.1 days (first phase 23.9–28.7 days and second phase 100.3–136.8 days) at 10 mg kg^{-1} levels under field

Table 3 Distribution of thiamethoxam in leachate fractions in laboratory leaching study

Fr. No	Volume (mL)	Cumulative volume (mL)	Mean concentration ± SD[a] (µg mL^{-1})			Cumulative amount recovered (µg)		
			AG[b]	Cruiser[c]	Actara[c]	AG	Cruiser	Actara
1	500	500	0.540 ± 0.062	0.471 ± 0.042	0.446 ± 0.020	270.0	235.5	223.0
2	350	850	1.316 ± 0.097	0.998 ± 0.100	0.998 ± 0.101	730.6	584.8	572.3
3	500	1,350	0.108 ± 0.019	0.142 ± 0.016	0.172 ± 0.025	784.6	655.8	658.3
4	550	1,900	0.006 ± 0.001	0.005 ± 0.001	0.007 ± 0.002	787.9	658.6	662.2
5	550	2,450	BDL[d]	0.002 ± 0.000	0.002 ± 0.000	787.9	659.6	663.3

[a] Standard deviation

[b] AG, Analytical grade

[c] Cruiser and Actara, thiamethoxam formulations; BDL, below detectable level

[d] Leachate <0.001 µg mL^{-1}

capacity and submerged conditions, respectively. Similar results have been reported for flufenacet (Gupta et al. 2001) and β-cyfluthrin (Gupta and Gajbhiye 2002). As per first order rate kinetics, the rate of dissipation is independent of rate of application provided all the conditions remain same. Under adequate moisture content (field capacity and submerged condition), the major route of loss is microbial degradation. However, at higher concentration level, the pesticide may affect microbial growth resulting in slower dissipation (Monkiedje et al. 2002).

Under dry condition dissipation was faster at high than low level of treatment (Fig. 3). The overall half-life value was 200.7 days (first phase 103.8 days and second phase 231.5 days) at high and 301.0 days (first phase 115.5 days and second phase 376.3 days) at low level of treatment. Similar observation of faster dissipation at higher level of treatment under dry condition has been reported for β-cyfluthrin (Gupta and Gajbhiye 2002). The reversal of trend under dry condition could be attributed to the effect of concentration on major process involved in dissipation. Under dry condition, the microbial activity is expected to be negligible and in absence of microbial activity, volatilization seems to be the major route of loss. Appreciable volatilization losses have been reported even for low vapor pressure compounds like DDT (1.9×10^{-7} mmHg), dieldrin (1.78×10^{-7} mmHg) and methyl parathion (9.6×10^{-6} mmHg). Volatilization losses as much as 47% for DDT (Nash 1983), 18% for dieldrin (Willis et al. 1972) and ~70% for methyl parathion (Kubiak et al. 1995) have been reported in literature. Thus, the losses under dry conditions, which were quite low as compared to adequate moisture conditions, could be attributed to volatilization of thiamethoxam even though it has very low vapor pressure (6.6×10^{-9} Pa at 25°C). The higher concentration is favorable for volatilization (Farmer et al. 1972) and hence there is faster dissipation at higher level of treatment. Faster volatilization of DDT in soil at higher rate of application has already been reported (Gajbhiye and Agnihotri 1998).

Thiamethoxam showed high potential for leaching under laboratory conditions. It appeared in the first leachate fraction (500 mL), increased up to 2nd fraction and thereafter decreased (Table 3). After the passing of ~2,500 mL water (equivalent to 65 cm rainfall) 66–79% of the added amount was recovered from leachate. None of the soil cores contained detectable residues at the end of the tests. There was not much difference in leaching behavior of thiamethoxam when applied as analytical grade material or formulations. However, slightly higher amount was recovered in leachate of analytical grade than formulation treatment. High potential for leaching has been reported for another neonicotinoid imidacloprid (Gupta et al. 2002; Pradas et al. 1999).

In laboratory experiments, thiamethoxam showed moderate persistence in soil under normal field capacity moisture regime ($T_{1/2}$ 91–94 days). Further increase in moisture level to submerged condition increased the rate of dissipation ($T_{1/2}$ 46–75 days). Under extreme dry condition, the persistence was quite high. Again under laboratory study, thiamethoxam showed high potential for leaching. However, under natural field condition of inadequate moisture and faster dissipation rate, the chances of contamination of ground water by thiamethoxam use is low. While using it under adequate moisture condition like rice cultivation, proper precautionary measures needs to be taken so that it does not leach down to ground water.

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