*Mysore J. Agric. Sci., 60 (2) : 310-322 (2026)*

# Impact of Soil Moisture and Organic Matter on Degradation of Fipronil in Tropical Paddy Soil

D. C. Preethu[1], K. R. Ashoka[2], T. Bhagyalakhsmi[3], L. Vijaykumar[4],
S. B. Yogananada[5] and N. Kirankumar[6]
[1]ICAR- Krishi Vigyan Kendra, Chandurayanahalli, Magadi - 562 120,
[2&3]Department of Soil Science and Agricultural Chemistry, [4]Department of Agricultural Entomology,
[5]Department of Agronomy, [6]Department of Plant Pathology, College of Agriculture, V.C. Farm, Mandya - 571 405
e-Mail : preethusrikanth24@gmail.com

## Authors Contribution

D. C. Preethu :
Investigation, data analysis, interpretation and draft preparation

K. R. Ashoka &
T. Bhagyalakhsmi
Conceptualization, supervision, interpretation, final validation and editing

L. Vijaykumar;
S. B. Yogananada &
N. Kirankumar :
Supervision and draft correction

***Corresponding Author :***
D. C. Preethu

*Received* : November 2025
*Accepted* : December 2025

## Abstract

The persistence of fipronil insecticide and its metabolites in paddy soils is influenced by agronomic practices, posing significant environmental and food safety concerns. A pot culture study was conducted to investigate the dissipation dynamics of fipronil and its metabolites (sulfone, sulfide and desulfinyl) in paddy soil under varying soil moisture regimes [flooded (FL), saturated (ST) and field capacity (FC)], with and without organic matter (OM- pressmud compost) and a co-applied weedicide (WD- Bensulfuron methyl + Pretilachlor). Soil samples were analyzed at intervals from 0 to 70 days after application (DAA) using a validated QuEChERS-LC-MS/MS method. Results demonstrated that soil moisture was the most critical factor governing fipronil persistence. Dissipation of fipronil was fastest under flooded conditions from 1.29 µg $g^{-1}$ on 0DAA to 0.06 µg $g^{-1}$on 70 DAA with degradation constant (K) of 0.032 recorded shortest half-life of 18.34 days. In contrast, fipronil persisted longest under field capacity moisture, with degradation constant (K) of 0.015, longest half-life of 45.60 days and highest residue accumulation from 1.62 µg $g^{-1}$ on 0DAA to 0.07 µg $g^{-1}$on 70 DAA. The formation of major metabolites was redox-dependent: sulfone was predominant under aerobic (FC/ST) conditions, while sulfide accumulated significantly in anaerobic (FL) environments. Organic amendment exhibited a dual role, accelerating degradation in flooded soils but enhancing persistence in FC soils *via* adsorption. The presence of paddy plants significantly enhanced degradation compared to uncropped control soil. Co-application of the weedicide did not substantially alter the dissipation pattern relative to moisture effects. This study concludes that,organic matter amendment in alkali soils served as an effective buffer, promoting gradual dissipation and sustained availability of fipronil to plant tissues under aerobic condition, thereby offering a safe and efficient strategy for rice cultivation in tropical climates.

*Keywords* : Fipronil, Metabolites, Soil moisture, Organic matter, Dissipation kinetics, Paddy soil, LC-MS/MS

India, with the largest area under rice cultivation, ranks as the world's second-largest producer of rice (FAO, 2025). Rice belongs to the family graminae, recognized as 'Millennium Crop' expected to contribute towards food security in the world; It is one of the staple cereal crops and a primary source of food for more than half of the world's population (Deepika and Devaraju, 2023).

Paddy cultivation often faces severe pest infestations, leading to significant production losses. The losses caused by various insect pests of paddy have been estimated to be 240 billion rupees, accounting for approximately 25 per cent of the overall crop losses (Savary *et al.,* 2019). Pesticides are essential products commonly used to manage pests and diseases in agricultural crops. However, the continuous application of various pesticides in fields can result in pesticide residues accumulating in agricultural soil. These residues can be absorbed by crop roots and transported to edible parts, posing a persistent risk to food safety (Anu *et al*., 2024).

Fipronil, a phenyl pyrazole insecticide, exhibits both contact and ingestion activity and is effective against a wide range of insect pests, including rice stem borer and leaf folder, at larval and adult stages (Kamaljeet and Gurpreet. 2016). It is particularly recommended in areas where pests have developed resistance to traditional insecticides such as organophosphates, carbamates and pyrethroids. Moreover, the recommended application rate of fipronil (0.6-200 g a.i. ha$^{-1}$) is lower than that of conventional insecticides, contributing to its popularity from an environmental safety point of view (Kumar and Singh, 2013). Fipronil 0.3 per cent GR is a granular insecticide formulation that facilitates easy application through broadcasting on standing paddy crops. In the paddy fields of Karnataka, it is commonly applied as a soil treatment at a dose of g a.i. ha$^{-1}$, equivalent to kg ha$^{-1}$ of granules, to manage leaf hopper, stem borer, gall midge and whorl maggot in paddy (Mukherjee *et al.,* 2021).

With its widespread use, numerous studies over the past two decades have highlighted the toxicity of fipronil and its metabolites to various non-target organisms. Fipronil is now recognized as one of the most persistent and lipophilic insecticides available. In the environment, it can undergo oxidation, reduction, hydrolysis or photolysis, producing metabolites such as fipronilsulfone, fipronilsulfide, fipronil amide and fipronil desulfinyl. Except for fipronil amide, these metabolites are generally as toxic or more persistent than the parent compound and have been detected in a variety of environmental samples (Mukherjee *et al.,* 2021).

The increasing number of pesticides used in agriculture has recently acquired great importance due to the contamination of the environment. Degradation studies in soil are essential for evaluation of persistence of pesticides and their breakdown products. Data on the rate of pesticide degradation are extremely important as they permit prediction of the potential risk associated with exposure. It is well recognized that there are risks attached to the consumption of pesticide-treated crops because of the presence of residues on them. Therefore, rational recommendation of a pesticide requires that it must not only provide an effective control of pests, but at the same time its residues on the commodity must be toxicologically acceptable.

When a pesticide is applied, it encounters a complex environment where biological (enzymatic), chemical (hydrolysis) and physical (photolysis) processes can alter its chemical structure. These transformation products, known as metabolites or degradation products, often possess properties different from the parent compound, necessitating evaluation of their non - target environmental and human health impacts. Metabolism studies are therefore conducted to gain a comprehensive understanding of the pesticide's fate and behaviour in environmental systems.

The key governing factors including pesticide properties, soil characteristics, interaction of pesticides with soil organic matter, moisture contents and soil microbes are all affecting the dissipation of pesticides (Bonmatin *et al.* 2015). With this background, our investigation was designed to study the effect of soil moisture, organic matter (pressmud) and weedicide (Bensulfuron methyl + Pretilachlor) on persistence and degradation of fipronilin paddy soil.

## MATERIAL AND METHODS

The analytical standards of fipronil (purity of 99.2%) and its three metabolites desulfinyl (purity 98.5%), sulphide (purity 98.0%) and sulfone (purity 99.7%) were all obtained from Bayer Crop Science limited, Maharashtra, India. The formulation of fipronil 0.3% GR (trade name: REGENT®) was procured from

Adama India Pvt. Ltd., Hyderabad for application in paddy soil. Ethyl acetate and acetonitrile (purity 99.9%, J.T. Baker, USA), millipore water (prepared from Ultra pure water purification system, Evoqua), magnesium sulphate heptahydrate (Loba Chemie Pvt. Ltd.), sodium sulphate anhydrous (Merck, India), sodium acetate (Isochem Laboratories, India), acetic acid (Merck, India), methanol (Merck, India), Primary Secondary Amine (PSA; 40 µm, Bondesil, Agilent, USA), C18 (Agilent, USA) and graphitized carbon black (GCB; Loba Chemie Pvt. Ltd.) were procured for use in residue analysis.

Fipronil, fipronil-sulfone, fipronil-sulfide and desulfone stock standard solutions of 1000 $mgL^{-1}$ were prepared in methanol and the working standard solutions required for fortification and quantification were prepared from stock solution by dilution with methanol. All standard solutions were stored at -20°C before use. The stability of the four compounds in methanol was reported to be stable at -20°C for more than 15 months (DG-SANTE, 2017).

*Soil Sampling* : Soil required for the study was collected from the top layer of soil (0 to 30 cm) of paddy field from College of Agriculture, V.C. Farm Mandya where paddy has been grown continuously for more than 10 years and not added fipronil to the soil. Collected soil samples were air dried. Then, the soil was manually ground and passed through a 2 mm screen sieve. Soil samples were analysed for mechanical composition, pH, EC, OC, CEC and nutrients content by following standard procedures (Table 1).

**Pot Culture Experiment**

The pot study was conducted under shade net at V.C. Farm Mandya to assess the effect of moisture and organic matter (OM) on fipronil degradation in paddy soil. Pressmud manure was used as organic matter (organic amendment) was analysed for chemical composition (Table 2).

Pesticide Residue Analysis was carried out at Pesticide Residue Analysis Laboratory, Karnataka

**TABLE 1**

**Physico-chemical characteristics of Mandya soil used in the study**

| Particulars | Paddy soil | Methods | References |
|---|---|---|---|
| pH (1: 2.5 soil water suspension) | 8.38 | Potentiometric method | Jackson (1973) |
| EC | 0.28 | Conductometric method | Jackson (1973) |
| Total organic carbon (%) | 0.6 | Walkley and Black's wet oxidation method | Walkley and Black (1934) |
| Sand (%) | 50.3 | International pipette method | Piper (1966) |
| Silt (%) | 19.5 | | |
| Clay (%) | 34.2 | | |
| Soil texture | Sandy Clay Loam | | |
| CEC (meq/100g) | 11.7 | Ammonium acetate leaching method | Jackson (1973) |
| MWHC (%) | 23.66 | Keen's cup | Piper (1966) |
| Available N (kg $ha^{-1}$) | 401.4 | Alkaline potassium permanganate method | Subbaiah and Asija (1956) |
| Available $P_2O_5$ (kg $ha^{-1}$) | 53.60 | Olsen's method using spectrophotometer | Jackson (1973) |
| Available $K_2O$ (kg $ha^{-1}$) | 75.66 | Ammonium acetate method | Piper 1966) |
| Exchangeable Ca (me/100g) | 8.8 | Complexometric titration | Piper 1966) |
| Exchangeable Mg (me/100g) | 4.6 | | |
| Exchangeable Na (me/100g) | 3.8 | | |

### TABLE 2

### Chemical composition of pressmud compost used in the study

| Particulars | Pressmud | Methods | References |
|---|---|---|---|
| pH (1:10) | 7.74 | Potentiometric method | Jackson, 1973 |
| Total OC (%) | 35.65 | Dry combustion method | Walkley and Black (1934) |
| Total Nitrogen (%) | 1.28 | Microkjeldahl method | Piper, 1966 |
| C:N ratio | 27.85 | | |
| Total Phosphorus (%) | 3.3 | Spectrophotometry | Piper, 1966 |
| Total Potassium (%) | 1.82 | Flame photometry | Piper, 1966 |
| Total Ca (%) | 2.5 | Complexometry using versenate solution | Piper, 1966 |
| Total Mg (%) | 1.0 | | |
| Total S (%) | 1.20 | Turbidity using barium chloride | Black (1965) |
| Total Zn (ppm) | 115 | Atomic absorption spectrophotometer | Jackson, 1973 |
| Total Cu (ppm) | 80 | | |
| Total Fe (ppm) | 400 | | |
| Total Mn (ppm) | 450 | | |

### Experimental treatments details

| | | |
|---|---|---|
| $T_1$ : FL+WOM | Without organic matter | Flooded condition |
| $T_2$ : ST+WOM | | Saturated condition |
| $T_3$ : FC+WOM | | Field capacity condition |
| $T_4$ : FL+OM | With organic matter (Pressmud compost) | Flooded condition |
| $T_5$ : ST+OM | | Saturated condition |
| $T_6$ : FC+OM | | Field capacity condition |
| $T_7$ : FL+WD | Weedicide-Bensulfuron methyl + Pretilachlor | Flooded condition |
| $T_8$ : ST+WD | | Saturated condition |
| $T_9$ : FC+WD | | Field capacity condition |
| $T_{10}$ : CT | Control- without crop | |

FL-Flooded; ST- Saturated; FC- Field Capacity; WOM-without organic matter; OM- Organic matter (pressmud); WD- Weedicide (Bensulfuron methyl 0.6%+ Pretilachlor6% GR); Control-Without crop+FC+WOM

State Department of Agriculture, Banashankari, Bengaluru, Karnataka, India. Experiment was conducted with nine treatments replicated three times. Details of the treatments are given above.

## Experimental Setup

Soil (2 kgs) was weighed into pots for nine treatments in triplicate. Press mud compost was used as organic matter for the $T_4$ to $T_6$ treatments. Pressmud compost. was thoroughly mixed with the soil at 0.5 per cent levels which corresponds to the recommended dose of farm yard manure for paddy fields. Combination of two active ingredients, bensulfuron-methyl (0.6%) and pretilachlor (6%) (a pre-emergence weedicide widely used in rice cultivation to control a broad spectrum of weeds) was used to study the effect of pesticide interaction on fipronil degradation for $T_7$ to $T_9$ treatments at

10mg kg$^{-1}$ which corresponds to the recommended dose of weedicide for paddy fields. $T_1$ to $T_3$ treatments were without organic matter and weedicide. These nine treatment combinations were replicated thrice.

Prior to pesticide application, soil in pots was left for 10 days for stabilization. Ten ml of aqueous fipronil suspension (Commercial grade Fipronil 0.3 GR at 75 g a.i. ha$^{-1}$) was added to each pot, resulting in a final concentration of 12.5 mg a.i. kg$^{-1}$ soil which corresponds to the recommended dose of fipronil for paddy fields. The moisture content of soil was maintained by adding deionised water in saturated, field capacity moisture (measured by Keens cup method) on weight basis and a consistent water level was maintained for other sets by adding deionised water at specified height to maintain the flooded conditions. Twenty days old paddy seedlings of RNR 15048 paddy variety were planted in the pot for $T_1$ to $T_9$ treatments. Control (CT-$T_{10}$) was the pot with soil without the paddy crop. All the treatments received recommended dose of fipronilon 20 days after transplanting.

The residues of fipronil and its metabolites sulfone, sulfide and desulfinyl in soil samples were analysed at 0 (2 h after application), 1, 3, 5, 10, 15, 30, 50, 60 and 70 days after the application (DAA) of fipronil pesticide by collecting the sample in scoop from the pot. Collected samples were transported to the laboratory in an ice packed box for pesticide residue analysis. The residue of fipronil and its metabolites in soil samples were analysed using Liquid Chromatography Mass Spectrometry Mass Spectrometry (LCMSMS) by following QuChERS method (Anastassiades *et al.,* 2003).

### Pesticide Residue Analysis

Ten grams soil samples were taken in triplicates in teflon centrifuge tubes. Deionized water was added to soils at saturation and field capacity moisture on weight basis. Acetonitrile (10 ml), 6 g anhydrous sodium sulphate and 1.5 g sodium acetate were added and vortexed followed by shaking on a rotospin for 15 min at 50 rpm. The samples were centrifuged at 5000 rpm for 5 minutes and 2 ml of the supernatant was transferred for clean-up into 5ml eppendorf tubes containing dispersive Solid Phase Extraction (d-SPE) sorbents *viz*., 150mg magnesium sulphate, 100mg Primary Secondary Amine (PSA). Eppendorf tubes were then vortexed and centrifuged for 5 min at 10000 rpm. After centrifugation, the upper solvent layer (1.5 ml) is collected. Finally, the supernatants of clear extracts were filtered by a syringe filter using 25 mm, 0.2 µm nylon filter paper and transferred into the vials for analysis in LCMSMS.

The recovery of fipronil from the sandy clay loam soil was studied at fortification level of 0.005, 0.01 and 0.1 µg g$^{-1}$. Soil (10g) in triplicate were taken in a 50 ml teflon centrifuge tube and required amount of mother stock solution of fipronil was added. Fipronil untreated soil sample was served as control. Same procedure was followed for the analysis of fipronil in LCMSMS.

### Analytical Procedure by LCMSMS

All samples were analysed using a Shimadzu LCMS-8045 equipped with a triple quadrupole MS/MS. The separation was performed using Shim pack GISS C18 column (100 mm×2.1 mm×3 µm particle size) which contains octadecyl stationary phase and high purity porous spherical silica support. The mobile phase was isocratic and consisted of methanol/water (50/50 by volume), with a flow rate of 0.4 ml/min. The MS/MS system gas was nitrogen at a constant flow of 3.0 l/min and the source temperature was 375°C. The nebulizer was 35.0 psi and the injection volume was set at 1 µl. Spectral acquisition was performed using negative electro-spray ionization mode for all four compounds, employed Multiple Reaction Monitoring (MRM).

### Instrumentation-Method Validation

Calibration linearity ($R^2$) and analytical sensitivity was checked by plotting calibration curve (0.001-0.500 µg g$^{-1}$) of mixture of standards having fipronil and its metabolites. The limit of detection (LOD) and limit of quantification (LOQ) was calculated by dividing three and ten times respectively and the average standard deviation of the peak area across all calibration levels by the slope of the calibration curve (DG-SANTE, 2017). The trueness and precision of the method was tested through a recovery experiment, conducted by spiking the control matrix with the respective compounds at different concentration levels of 0.005, 0.010 and 0.100 µg $g^{-1}$ and processed using the aforementioned method. Precision was assessed by calculating the relative standard deviation (RSD) in terms of intra-day repeatability check

### Statistical Analysis

Individual data sets on fipronil residues and other soil parameters were statistically analyzed using the IBM SPSS Statistics 27.0.1 software package. The dissipation curves of fipronil were analyzed using nonlinear regression in Microsoft Excel 2010. The degradation followed first-order kinetics, described by the equation $C = C_0e^{-kt}$, where C is the chemical concentration (mg $kg^{-1}$) at time t (days), $C_0$ is the initial concentration (mg $kg^{-1}$) and k is the first-order rate constant ($d^{-1}$), independent of C and $C_0$. The correlation coefficient ($R^2$) was used to evaluate the congruence between the data and the first-order kinetic model. The dissipation of these compounds was commonly expressed as $DT_{50}$ ($t_{1/2}$), the time required for 50% of the pesticide to degrade was calculated using Hoskins' formula ($DT_{50} = (\ln 2)/k$).

## RESULTS AND DISCUSSION

*Method Validation* : The analytical method was successfully validated for quantifying fipronil and its metabolites in paddy soil matrices. The method demonstrated excellent linearity ($R^2 > 0.99$) across a range of 0.005-0.100 µg $g^{-1}$. Average recoveries ranged from 78.01 to 95.47 per cent with RSDs ≤ 12.92 per cent and matrix effects were negligible (±10%), confirming high accuracy, precision and reliability. The LOD and LOQ were established at 0.001 and 0.003 µg $g^{-1}$, respectively meeting all quality control standards (DG-SANTE 2017).

### Persistence of Fipronil in Paddy Soil

Residue dynamics of fipronil along with three major metabolites *viz*., sulfone, sulfide and desulfinyl metabolites under different soil moistures, organic matter and weedicide is presented in Table 3, 4, 5 and 6.

TABLE 3

**Persistence of fipronil (µg $g^{-1}$) in paddy soil under different soil moisture, organic matter and weedicide under pot culture**

| DAA | Residue level (µg $g^{-1}$) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 0 | 1 | 3 | 5 | 10 | 15 | 30 | 50 | 60 | 70 |
| $T_1$ | $1.04^{a}$ | $1.03^{a}$ | $0.72^{a}$ | $0.71^{a}$ | $0.53^{a}$ | $0.33^{a}$ | $0.07^{a}$ | $0.02^{a}$ | $0.01^{a}$ | $0.00^{a}$ |
| $T_2$ | $1.07^{a}$ | $1.06^{ab}$ | $0.82^{a}$ | $0.72^{a}$ | $0.59^{a}$ | $0.35^{a}$ | $0.16^{b}$ | $0.11^{b}$ | $0.07^{b}$ | $0.03^{b}$ |
| $T_3$ | $1.62^{d}$ | $1.55^{d}$ | $1.38^{c}$ | $1.24^{e}$ | $0.90^{b}$ | $0.50^{b}$ | $0.48^{e}$ | $0.23^{de}$ | $0.15^{de}$ | $0.07^{d}$ |
| $T_4$ | $1.29^{bc}$ | $1.27^{bc}$ | $1.23^{bc}$ | $1.04^{cd}$ | $0.82^{b}$ | $0.37^{a}$ | $0.30^{c}$ | $0.21^{d}$ | $0.11^{c}$ | $0.06^{c}$ |
| $T_5$ | $1.34^{c}$ | $1.33^{c}$ | $1.25^{bc}$ | $1.18^{de}$ | $0.85^{b}$ | $0.39^{a}$ | $0.36^{d}$ | $0.22^{de}$ | $0.13^{d}$ | $0.06^{cd}$ |
| $T_6$ | $1.88^{e}$ | $1.84^{e}$ | $1.77^{d}$ | $1.57^{fg}$ | $1.21^{c}$ | $0.71^{c}$ | $0.51^{e}$ | $0.25^{e}$ | $0.18^{f}$ | $0.15^{f}$ |
| $T_7$ | $1.13^{ab}$ | $1.13^{abc}$ | $0.82^{a}$ | $0.82^{ab}$ | $0.60^{a}$ | $0.35^{a}$ | $0.25^{c}$ | $0.15^{c}$ | $0.08^{b}$ | $0.04^{b}$ |
| $T_8$ | $1.26^{bc}$ | $1.25^{abc}$ | $1.14^{b}$ | $1.00^{bc}$ | $0.65^{a}$ | $0.36^{a}$ | $0.30^{c}$ | $0.17^{c}$ | $0.09^{b}$ | $0.05^{c}$ |
| $T_9$ | $1.73^{de}$ | $1.71^{de}$ | $1.70^{d}$ | $1.45^{f}$ | $1.20^{c}$ | $0.69^{c}$ | $0.49^{e}$ | $0.25^{de}$ | $0.15^{e}$ | $0.11^{e}$ |
| $T_{10}$ | $1.90^{e}$ | $1.89^{e}$ | $1.87^{d}$ | $1.66^{g}$ | $1.34^{d}$ | $0.74^{c}$ | $0.66^{f}$ | $0.34^{f}$ | $0.22^{g}$ | $0.17^{g}$ |
| $R^2$ | 0.92 | 0.86 | 0.93 | 0.93 | 0.97 | 0.95 | 0.97 | 0.95 | 0.97 | 0.99 |

Values superscripted with different letter differ significantly according to Duncan's multiple range test ($p \leq 0.05$)

$T_1$ : FL + WOM; $T_2$ : ST + WOM; $T_3$ : FC + WOM; $T_4$ : FL + OM ; $T_5$ : ST + OM; $T_6$ : FC + OM; $T_7$ : FL + WD;
$T_8$ : ST + WD; $T_9$ : FC + WD; $T_{10}$ : Control

DAA- Days After application of fipronil; FL-Flooded; ST- Saturated; FC- Field Capacity; WOM-without organic matter; OM-Organic matter (pressmud); WD- Weedicide (Bensulfuron methyl 0.6%+ Pretilachlor6% GR); Control-Without crop+FC+WOM

### TABLE 4
### Residue level of fipronil sulfone (µg $g^{-1}$) in paddy soil under different soil moisture, organic matter and weedicide under pot culture

| DAA | Residue level (µg $g^{-1}$) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 0 | 1 | 3 | 5 | 10 | 15 | 30 | 50 | 60 | 70 |
| $T_1$ | $0.01^{a}$ | $0.02^{a}$ | $0.02^{a}$ | $0.02^{a}$ | $0.03^{a}$ | $0.04^{a}$ | $0.05^{a}$ | $0.07^{a}$ | $0.07^{a}$ | $0.08^{a}$ |
| $T_2$ | $0.02^{ab}$ | $0.02^{bcd}$ | $0.02^{b}$ | $0.02^{ab}$ | $0.03^{b}$ | $0.05^{b}$ | $0.07^{abc}$ | $0.08^{ab}$ | $0.10^{b}$ | $0.11^{bc}$ |
| $T_3$ | $0.02^{bc}$ | $0.02^{cd}$ | $0.02^{c}$ | $0.04^{d}$ | $0.05^{c}$ | $0.09^{d}$ | $0.15^{d}$ | $0.15^{d}$ | $0.16^{cd}$ | $0.16^{de}$ |
| $T_4$ | $0.02^{ab}$ | $0.02^{abc}$ | $0.02^{b}$ | $0.02^{ab}$ | $0.03^{ab}$ | $0.05^{ab}$ | $0.07^{abc}$ | $0.08^{ab}$ | $0.09^{b}$ | $0.10^{ab}$ |
| $T_5$ | $0.02^{bc}$ | $0.02^{bcd}$ | $0.02^{b}$ | $0.03^{c}$ | $0.05^{c}$ | $0.06^{c}$ | $0.08^{c}$ | $0.12^{c}$ | $0.14^{c}$ | $0.15^{d}$ |
| $T_6$ | $0.03^{d}$ | $0.03^{e}$ | $0.03^{d}$ | $0.05^{e}$ | $0.09^{e}$ | $0.13^{f}$ | $0.20^{e}$ | $0.23^{e}$ | $0.25^{e}$ | $0.28^{f}$ |
| $T_7$ | $0.02^{ab}$ | $0.02^{ab}$ | $0.02^{b}$ | $0.02^{ab}$ | $0.03^{ab}$ | $0.04^{ab}$ | $0.06^{ab}$ | $0.07^{a}$ | $0.07^{a}$ | $0.08^{a}$ |
| $T_8$ | $0.02^{bc}$ | $0.02^{bcd}$ | $0.02^{b}$ | $0.03^{b}$ | $0.05^{c}$ | $0.06^{c}$ | $0.08^{bc}$ | $0.09^{b}$ | $0.11^{b}$ | $0.13^{c}$ |
| $T_9$ | $0.02^{c}$ | $0.02^{d}$ | $0.03^{c}$ | $0.05^{e}$ | $0.07^{d}$ | $0.11^{e}$ | $0.15^{d}$ | $0.16^{d}$ | $0.16^{d}$ | $0.18^{e}$ |
| $T_{10}$ | $0.02^{bc}$ | $0.02^{bcd}$ | $0.02^{b}$ | $0.02^{ab}$ | $0.05^{c}$ | $0.06^{c}$ | $0.07^{abc}$ | $0.09^{b}$ | $0.10^{b}$ | $0.11^{bc}$ |
| $R^2$ | 0.78 | 0.77 | 0.87 | 0.95 | 0.96 | 0.96 | 0.95 | 0.97 | 0.96 | 0.95 |

Values superscripted with different letter differ significantly according to Duncan's multiple range test ($p \leq 0.05$)

$T_1$ : FL + WOM; $T_2$ : ST + WOM; $T_3$ : FC + WOM; $T_4$ : FL + OM ; $T_5$ : ST + OM; $T_6$ : FC + OM; $T_7$ : FL + WD; $T_8$ : ST + WD; $T_9$ : FC + WD; $T_{10}$ : Control

DAA- Days After application of fipronil; FL-Flooded; ST- Saturated; FC- Field Capacity; WOM-without organic matter; OM-Organic matter (pressmud); WD- Weedicide (Bensulfuron methyl 0.6%+ Pretilachlor6% GR); Control-Without crop+FC+WOM

The result found that the residue level of fipronil (Table 3) was significantly highest in field capacity (FC) condition (0.07, 0.15 and 0.11µg $g^{-1}$ at 70 DAA) while flooded conditions had significantly lower residues (0.00, 0.06, 0.04 µg $g^{-1}$ at 70 DAA) under organic unamend, organic amended and weedicide applied soil respectively. Flooded soil (FL) ($T_1$, $T_4$ and $T_7$) yielded statistically lowest residue of fipronil (0.00-0.06 µg $g^{-1}$). Throughout the study, the FC moisture soils have registered slower dissipation, retaining fipronil residues above the MRL among the three soil moisture regimes. (MRL -0.01 µg $g^{-1}$). In contrast, flooded soils showed rapid dissipation, with residues often below detectable levels by harvest. Flooded soils showed faster dissipation and shorter half-life (18.34 days), while FC soils, particularly with organic matter, exhibited slower degradation and extended half-life (45.60 days). Control soils without plants recorded the maximum persistence (51.34 days), highlighting the influence of rhizosphere activity in enhancing degradation.

Numerous studies have shown moisture/ redox strongly controls dissipation and half-life of insecticide, Tang *et al.* (2020) reported much shorter $t_{1/2}$, under submerged conditions than under field capacity or dry conditions for thiamethoxam, illustrating the same pattern as observed in our study. Mahapatra *et al.* (2010) reported large decreases in fipronil half-life under submerged/ flooded conditions compared with field-capacity or low-moisture soils.

Control (no-plant) soils often show maximum persistence because rhizosphere processes accelerate degradation *via* root - microbe interactions (Yang *et al.,* 2020). Adsorption to soil constituents and organic matter reducing bioavailability

## TABLE 5

## Residue level of fiproniol sulfide (µg $g^{-1}$) in paddy soil under different soil moisture, organic matter and weedicide under pot culture

| DAA | Residue level (µg $g^{-1}$) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 0 | 1 | 3 | 5 | 10 | 15 | 30 | 50 | 60 | 70 |
| $T_1$ | $0.02^{bc}$ | $0.01^{bc}$ | $0.02^{c}$ | $0.03^{c}$ | $0.03^{d}$ | $0.06^{d}$ | $0.10^{e}$ | $0.11^{c}$ | $0.11^{cd}$ | $0.11^{ef}$ |
| $T_2$ | $0.01^{abc}$ | $0.01^{bc}$ | $0.01^{b}$ | $0.02^{a}$ | $0.02^{b}$ | $0.03^{ab}$ | $0.05^{abc}$ | $0.06^{a}$ | $0.07^{b}$ | $0.08^{bc}$ |
| $T_3$ | $0.01^{a}$ | $0.01^{a}$ | $0.01^{a}$ | $0.01^{a}$ | $0.02^{a}$ | $0.03^{a}$ | $0.04^{a}$ | $0.05^{a}$ | $0.05^{a}$ | $0.06^{a}$ |
| $T_4$ | $0.02^{d}$ | $0.02^{d}$ | $0.02^{d}$ | $0.03^{d}$ | $0.06^{f}$ | $0.09^{f}$ | $0.14^{f}$ | $0.16^{d}$ | $0.17^{e}$ | $0.19^{h}$ |
| $T_5$ | $0.01^{abc}$ | $0.01^{bc}$ | $0.01^{b}$ | $0.02^{b}$ | $0.04^{d}$ | $0.04^{c}$ | $0.06^{c}$ | $0.08^{b}$ | $0.10^{c}$ | $0.10^{de}$ |
| $T_6$ | $0.01^{abc}$ | $0.01^{ab}$ | $0.01^{b}$ | $0.02^{a}$ | $0.02^{ab}$ | $0.03^{a}$ | $0.05^{abc}$ | $0.05^{a}$ | $0.07^{ab}$ | $0.07^{ab}$ |
| $T_7$ | $0.01^{c}$ | $0.01^{c}$ | $0.02^{c}$ | $0.03^{d}$ | $0.05^{e}$ | $0.08^{e}$ | $0.10^{e}$ | $0.11^{c}$ | $0.11^{cd}$ | $0.12^{fg}$ |
| $T_8$ | $0.01^{abc}$ | $0.01^{bc}$ | $0.01^{b}$ | $0.02^{a}$ | $0.03^{d}$ | $0.04^{bc}$ | $0.05^{bc}$ | $0.06^{a}$ | $0.08^{b}$ | $0.09^{cd}$ |
| $T_9$ | $0.01^{ab}$ | $0.01^{ab}$ | $0.01^{b}$ | $0.01^{a}$ | $0.02^{ab}$ | $0.03^{a}$ | $0.04^{ab}$ | $0.06^{a}$ | $0.05^{a}$ | $0.06^{a}$ |
| $T_{10}$ | $0.02^{e}$ | $0.02^{e}$ | $0.02^{e}$ | $0.07^{e}$ | $0.03^{c}$ | $0.07^{e}$ | $0.09^{d}$ | $0.11^{c}$ | $0.12^{d}$ | $0.13^{gf}$ |
| $R^2$ | 0.83 | 0.91 | 0.91 | 0.98 | 0.96 | 0.96 | 0.96 | 0.96 | 0.95 | 0.93 |

Values superscripted with different letter differ significantly according to Duncan's multiple range test ($p \leq 0.05$)

T : FL + WOM; T2 : ST + WOM; T3 : FC + WOM; T4 : FL + OM; T5 : ST + OM; T6 : FC + OM; T7 : FL + WD; T8 : ST + WD; T9 : FC + WD; T10 : Control

DAA- Days After application of fipronil; FL-Flooded; ST- Saturated; FC- Field Capacity; WOM-without organic matter; OM- Organic matter (pressmud); WD- Weedicide (Bensulfuron methyl 0.6%+ Pretilachlor6% GR); Control-Without crop+FC+WOM

(hence slower degradation) is a recurring theme in the literature (Rasool *et al.,* 2022).

Studies have shown organic matters can both increase sorption (stabilizing residues) and stimulate microbial degradation, depending on context. Silva *et al.* (2016), Pose-Juan *et al.* (2018) and Yu *et al.* (2020) have reported that added OM changes sorption and dissipation variably-filter cake, straw, composts and vinasse altered other pesticide half-lives differently across studies. Sarker *et al.* (2023) and You *et al.* (2020) further demonstrated how different amendments (biochar, vinasse) change pore-water concentration and microbial transformation. These results support the dual behaviour as observed in our study.

### Residue Level of Fipronil Metabolite in Paddy Soil

Among the metabolites, formation of sulfone metabolite was significantly predominant followed by sulfide metabolite and desulfinyl metabolite. Formation of sulfone metabolite (Table 4) was significantly more in FC and saturated soils (0.16, 0.28, 0.18 and 0.11, 0.15, 0.13 µg $g^{-1}$ at 70 DAA under organic unamend, organic amend and weedicide applied soil respectively) compared to flooded soil (0.08, 0.10, 0.08 µg $g^{-1}$ at 70 DAA). Sulfone metabolite formation peaks even at the harvest stage (70 DAA) retaining 0.16-0.28 µg $g^{-1}$ in FC soil. Flooded and organic unamend soil ($T_1$= 0.08 µg $g^{-1}$) shows significantly higher sulfide metabolite accumulation, indicating the reduced oxidation of fipronil in anaerobic conditions.

Sulfide metabolite (Table 5) accumulate significantly more in flooded soil (0.11, 0.19 and 0.12 µg $g^{-1}$ at 70 DAA under organic unamend, organic amended and weedicide soil respectively), suggesting anaerobic degradation pathways dominate in flooded

**TABLE 6**

**Residue level of fipronil desulfinyl (µg $g^{-1}$) in paddy soil under different soil moisture, organic matter and weedicide under pot culture**

| DAA | Residue level (µg $g^{-1}$) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 0 | 1 | 3 | 5 | 10 | 15 | 30 | 50 | 60 | 70 |
| $T_1$ | 0.002 $^{a}$ | 0.002 $^{a}$ | 0.002 $^{a}$ | 0.002 $^{a}$ | 0.003 $^{a}$ | 0.006 $^{a}$ | 0.008 $^{a}$ | 0.009 $^{a}$ | 0.004 $^{a}$ | 0.011 $^{a}$ |
| $T_2$ | 0.002 $^{abc}$ | 0.002 $^{bc}$ | 0.002 $^{ab}$ | 0.003 $^{bc}$ | 0.004 $^{b}$ | 0.005 $^{ab}$ | 0.006 $^{abc}$ | 0.008 $^{ab}$ | 0.008 $^{b}$ | 0.009 $^{bc}$ |
| $T_3$ | 0.002 $^{bc}$ | 0.002 $^{bc}$ | 0.003 $^{c}$ | 0.004 $^{e}$ | 0.006 $^{c}$ | 0.013 $^{d}$ | 0.017 $^{e}$ | 0.017 $^{d}$ | 0.017 $^{e}$ | 0.020 $^{de}$ |
| $T_4$ | 0.002 $^{abc}$ | 0.002 $^{ab}$ | 0.002 $^{ab}$ | 0.002 $^{ab}$ | 0.003 $^{ab}$ | 0.006 $^{a}$ | 0.009 $^{abc}$ | 0.010 $^{ab}$ | 0.012 $^{bc}$ | 0.014 $^{ab}$ |
| $T_5$ | 0.002 $^{bc}$ | 0.002 $^{bc}$ | 0.002 $^{b}$ | 0.004 $^{d}$ | 0.005 $^{c}$ | 0.005 $^{c}$ | 0.009 $^{c}$ | 0.013 $^{c}$ | 0.016 $^{d}$ | 0.016 $^{d}$ |
| $T_6$ | 0.003 $^{d}$ | 0.003 $^{d}$ | 0.003 $^{d}$ | 0.005 $^{g}$ | 0.010 $^{e}$ | 0.014 $^{f}$ | 0.022 $^{f}$ | 0.026 $^{e}$ | 0.028 $^{g}$ | 0.031 $^{f}$ |
| $T_7$ | 0.002 $^{ab}$ | 0.002 $^{ab}$ | 0.002 $^{ab}$ | 0.002 $^{a}$ | 0.003 $^{ab}$ | 0.005 $^{a}$ | 0.007 $^{ab}$ | 0.007 $^{a}$ | 0.002 $^{a}$ | 0.009 $^{a}$ |
| $T_8$ | 0.002 $^{abc}$ | 0.002 $^{bc}$ | 0.002 $^{b}$ | 0.003 $^{c}$ | 0.005 $^{c}$ | 0.007 $^{bc}$ | 0.008 $^{bc}$ | 0.009 $^{b}$ | 0.011 $^{c}$ | 0.013 $^{c}$ |
| $T_9$ | 0.002 $^{c}$ | 0.002 $^{c}$ | 0.003 $^{c}$ | 0.005 $^{f}$ | 0.008 $^{d}$ | 0.010 $^{e}$ | 0.017 $^{e}$ | 0.017 $^{d}$ | 0.018 $^{e}$ | 0.018 $^{e}$ |
| $T_{10}$ | 0.003 $^{e}$ | 0.004 $^{e}$ | 0.004 $^{e}$ | 0.005 $^{e}$ | 0.010 $^{e}$ | 0.012 $^{e}$ | 0.014 $^{d}$ | 0.017 $^{d}$ | 0.020 $^{f}$ | 0.021 $^{f}$ |
| $R^2$ | 0.87 | 0.91 | 0.88 | 0.98 | 0.98 | 0.96 | 0.96 | 0.96 | 0.97 | 0.96 |

Values superscripted with different letter differ significantly according to Duncan's multiple range test ($p \leq 0.05$)

T : FL + WOM; T2 : ST + WOM; T3 : FC + WOM; T4 : FL + OM; T5 : ST + OM; T6 : FC + OM; T7 : FL + WD;
T8 : ST + WD; T9 : FC + WD; T10 : Control

DAA- Days After application of fipronil; FL-Flooded; ST- Saturated; FC- Field Capacity; WOM-without organic matter;
OM- Organic matter (pressmud); WD- Weedicide (Bensulfuron methyl 0.6%+ Pretilachlor6% GR); Control-Without crop+FC+WOM

environments. FC and saturated soil have significantly lower sulfide metabolite levels, indicating limited anaerobic activity. Residue level is highest at 70 DAA in flooded soil, under organic amended soil (0.19 µg $g^{-1}$).

Desulfinyl metabolite formation (Table 6) was most pronounced under field capacity soil moistures (0.018-0.031 µg $g^{-1}$) at 70 DAA and in control conditions, pointing to enhanced photodegradation or oxidative transformation ($T_{10}$ = 0.021 µg $g^{-1}$). Flooded anaerobic environments, particularly with organic matter or weedicide (0.01 µg $g^{-1}$ or less), suppressed desulfinyl metabolite accumulation likely due to limited light availability and favorable conditions for alternative reductive pathways. Statistical analysis confirms significant differences between treatments.

Metabolite profiling showed sulfone as the predominant metabolite, especially under FC and saturated soils (0.16-0.28 µg $g^{-1}$ at 70 DAA), indicating that aerobic conditions favored oxidative transformation. These findings align with Gairhe *et al.* (2021), who reported sulfone as the major metabolite of fipronil under oxygen-rich conditions. In contrast, flooded anaerobic soils favoured sulfide accumulation (0.11-0.19 µg $g^{-1}$), confirming reductive degradation pathways under submerged environments. Desulfinyl metabolite was detected primarily under FC and control treatments (0.018-0.031 µg $g^{-1}$), suggesting a role of photolytic or oxidative processes, which were suppressed in flooded soils. The absence or very low levels of desulfinyl under anaerobic conditions supports earlier findings that photoproducts are minimized in shaded or reduced environments (Toolkiattiwong *et al.,* 2023).

Bobe *et al.* (1998) identified sulfone as a major oxidative product of fipronil under oxygenated conditions Toolkiattiwong *et al.* (2023) reported predominance of fipronil in paddy soils and discussed transformation under different rice systems. Anaerobic/reductive environments favour sulfide formation-this redox-driven metabolite partitioning is consistent with Mohapatra *et al.* (2010) and the results on redoxpotential are summarized in Gairhe *et al.* (2021); Bobe *et al.* (1998); Toolkiatti wong *et al.*(2023) and Mohapatra *et al.* (2010). Photolytic or oxidative products (*e.g.*, desulfinyl) require exposure to oxygen/light; reduced/anaerobic or shaded conditions suppress such pathways (Tool kiatti wong *et al*., 2023).

Organic matter exerted dual effects: under flooded conditions it accelerated dissipation, possibly by increasing dissolved organic carbon and microbial activity, while in FC soils it enhanced persistence through adsorption, thereby reducing bioavailability. Such contrasting behavior has been noted in studies with chlorpyrifos and metolachlor, where organic matter either promoted degradation or stabilized residues depending on soil moisture and binding strength (George *et al.,* 2014). The rhizosphere and microbial community composition as key drivers of faster or altered degradation and metabolite patterns (Sarkar *et al*., 2023).

**Dissipation Kinetics of Fipronil in Paddy Soil**

Fipronil degradation data obtained from paddy soil under different soil moisture, organic matter and weedicide was fitted to the first order kinetics equation (Fig. 1, Table 7). The amount of fipronil

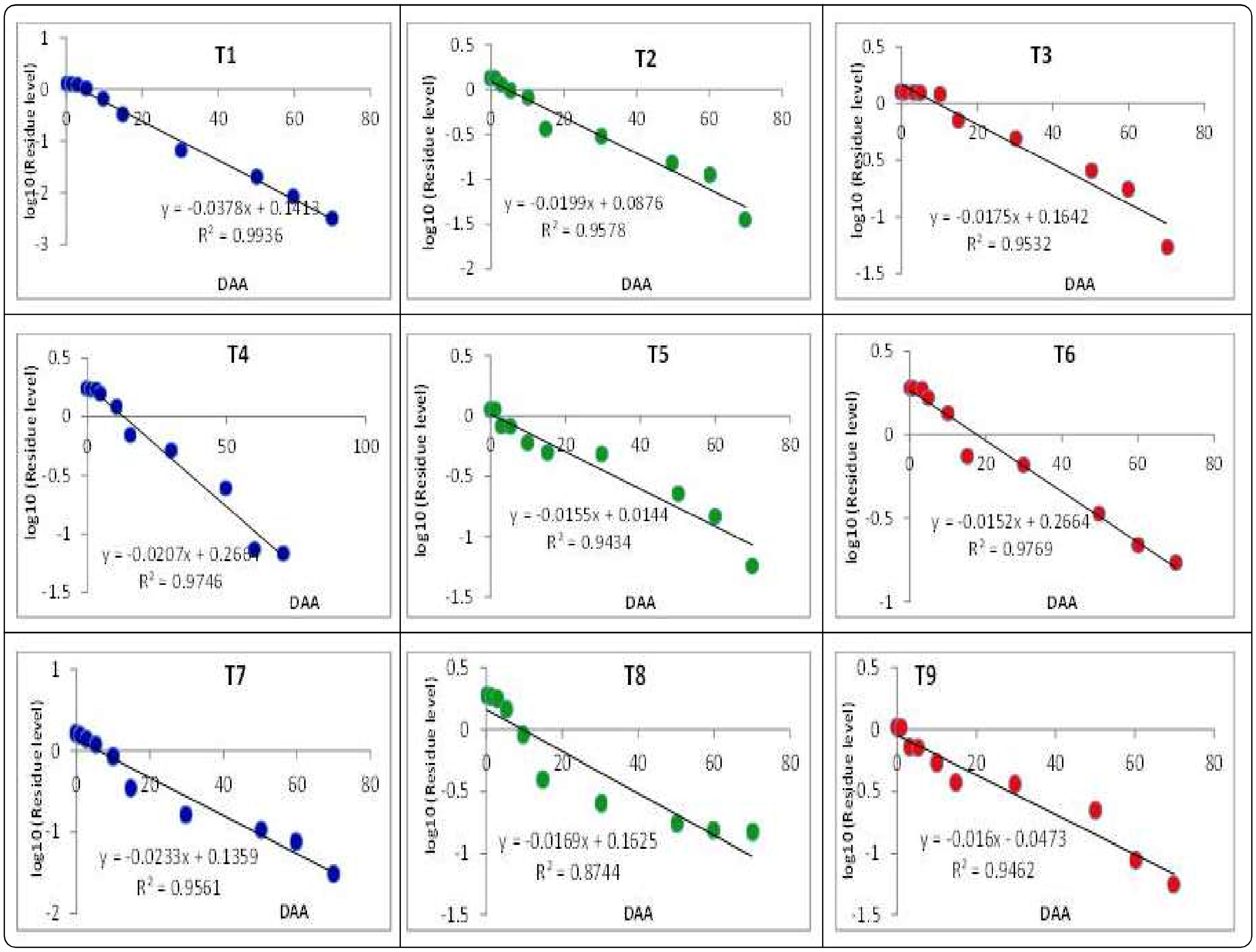

Fig. 1 : First order dissipation of fipronil in paddy soil under different soil moistures with organic matter and weedicide in pot culture

TABLE 7

**Dissipation kinetics of fipronil in paddy soil under different soil moistures with organic matter and weedicide application in pot culture experiment**

| DAA | Regression equation (Y) | Regression coefficient ($R^2$) | Dissipation constant (K) | Half life ($t_{1/2}$) |
|---|---|---|---|---|
| $T_1$ | y = -0.0378x + 0.1413 | 0.99 | 0.038 | 18.34 |
| $T_2$ | y = -0.0199x + 0.0876 | 0.96 | 0.020 | 34.83 |
| $T_3$ | y = -0.0175x + 0.1642 | 0.95 | 0.018 | 39.61 |
| $T_4$ | y = -0.0207x + 0.2664 | 0.97 | 0.021 | 33.49 |
| $T_5$ | y = -0.0155x + 0.0144 | 0.94 | 0.016 | 44.72 |
| $T_6$ | y = -0.0152x + 0.2664 | 0.98 | 0.015 | 45.60 |
| $T_7$ | y = -0.0233x + 0.1359 | 0.96 | 0.023 | 29.75 |
| $T_8$ | y = -0.0169x + 0.1625 | 0.87 | 0.017 | 41.01 |
| $T_9$ | y = -0.016x - 0.0473 | 0.95 | 0.016 | 43.32 |
| $T_{10}$ | y = -0.0135x - 0.0629 | 0.95 | 0.014 | 51.34 |

$T_1$: FL + WOM; $T_2$ : ST + WOM; $T_3$ : FC + WOM; $T_4$ : FL + OM; $T_5$ : ST + OM; $T_6$ : FC + OM;
$T_7$ : FL + WD; $T_8$ : ST + WD; $T_9$: FC + WD; $T_{10}$ : Control

DAA- Days After application of fipronil; FL-Flooded; ST- Saturated; FC- Field Capacity; WOM-without organic matter; OM- Organic matter (pressmud); WD- Weedicide (Bensulfuron methy l 0.6% + Pretilachlor 6% GR); Control-Without crop+FC+WOM

remaining in the soil under pot culture experiment was plotted on a logarithmic scale against the incubation time. The degradation of fipronil in soil followed a first-order reaction, as indicated by the linear plots based on the equation: $C = C_0e^{-kt}$ where C is the concentration of the insecticide remaining in the soil after time t, $C_0$ is the initial concentration and k is the first-order kinetic constant.

The half-life ($t_{1/2}$) values were calculated using the formula: $t_{1/2} = \ln2/k$. The slope of the curve, correlation coefficient, rate constant and half-life of the fipronil as obtained from the regression equation are summarized in Table 7. The results showed that the lowest half-life of 18.34 days was observed in flooded soil with organic unamended soil indicated the faster dissipation of fipronil with highest dissipation rate constant of 0.038 (k). Highest half-life (45.60 days) in FC moisture soil with organic matter indicated the longer persistence of fipronil with lowest dissipation rate constant (k) of 0.015. Treatment without plant showed longer half life of 51.34 days with dissipation rate constant of 0.014. A regression coefficient ($R^2$) value more than 0.87 confirmed a strong linear relationship between time and fipronil degradation, with the highest $R^2$ value recorded at 0.98 in FC soil with organic matter.

Kinetic analysis confirmed that fipronil dissipation followed first-order reactions across all treatments, with half-lives ranging from 18.34 days in flooded soils (rapid loss) to 45.60 days in FC soils with organic matter (prolonged persistence). These values are within the range reported by Rasool *et al.* (2022), who emphasized that soil moisture and organic matter largely determine dissipation dynamics. The shorter half-life under flooded soils parallels findings of Ge *et al.* (2017) on thiame thoxam, where submergence enhanced degradation. Conversely, prolonged persistence under FC soils mirrors the results of Tang *et al.* (2020), where relatively drier soils slowed degradation.

This study demonstrates that soil moisture is the primary factor controlling the fate of fipronil in paddy soils. Flooded (anaerobic) conditions rapidly degraded fipronil (half-life: 18.34 days), while field capacity (aerobic) conditions led to prolonged persistence (half-life up to 45.60 days) and residues exceeding the MRL. Metabolic pathways were redox-dependent: fipronil sulfone dominated in aerobic soils, whereas fipronil sulfide accumulated in anaerobic soils. Organic amendment exhibited a dual role, accelerating degradation in flooded soils but enhancing persistence in moist soils *via* sorption. Furthermore, the presence of plants significantly shortened the pesticide's half-life, underscoring the critical role of the rhizosphere in its degradation. Therefore, water management and organic matterare the decisive practice for mitigating fipronil persistence.

## REFERENCES

ANASTASSIADES, M., LEHOTAY, S. J., STAJNBAHER, D. AND SCHENCK, F. J., 2003, Fast and easy multiresidue method employing acetonitrile extraction/partitioning and 'dispersive solid-phase extraction' for the determination of pesticide residues in produce. *J. AOAC Int.*, **86** (2) : 412 - 431.

ANU, C. N., ASOKAN, R., JEMLA NAIK D., MANAMOHAN, M. AND ANITA PETER, 2024, Cloning and *In-vitro* restriction analysis of the sex peptide receptor gene in fall armyworm, *Spodoptera frugiperda* (J. E. Smith) (Lepidoptera: Noctuidae). *Mysore J. Agric. Sci.,* **58** (4) : 16 - 23.

BLACK, C. A., 1965, Method of soil analysis part II. Agronomy Monograph No. 9. American Society of Agronomy, Madison, Wisconsin, pp. 148.

BOBE, A., MEALLIER, P., COOPER, J. F. AND COSTE, C. M., 1998, Kinetics and mechanisms of abiotic degradation of fipronil (hydrolysis and photolysis). *J. Agric. Food Chem.*, **46** (7) : 2834 - 2839.

BONMATIN, J. M., GIORIO, C., GIROLAMI, V., GOULSON, D., KREUTZWEISER, D. P., KRUPKE, C., LIESS, M., LONG, E., MARZARO, M., MITCHELL, E. A. D., NOOME, D. A., SIMON-DELSO, N. AND TAAPPARO, A., 2015, Environmental fate and exposure: neonicotinoids and fipronil. *Environ. Sci. Pollut. Control Ser.*, **22** (1) : 35- 67.

DEEPIKA, C. AND DEVARAJU, P. J., 2023, Morphological Characterization of Selected Land Races of Rice (*Oryzasativa L.*). *Mysore J. Agric. Sci.*, **57** (2) : 72 - 87.

DG-SANTE, 2017, Guidance document on analytical quality control and method validation procedures for pesticide residues and analysis in food and feed. *Document No. SANTE/11813/2017*, European Commission, 21-22, November 2017, rev.0.

FAO, 2025, Rice market monitor. Available at: http://www.fao.org/3/I9243EN/i9243en.pdf.

GAIRHE, S., SAPKOTA, B. AND KARKI, R., 2021, Persistence and dissipation kinetics of fipronil and its metabolites in tropical rice soil ecosystem. *Environ. Sci. Pollut. Res.*, **28** (15) : 19242 - 19251.

GE, J., CUI, K., YAN, H., LI, Y., CHAI, Y., LIU, X., CHENG, J. AND YU, X., 2017, Uptake and translocation of imidacloprid, thiamethoxam and difenoconazole in rice plants. *Environ. Pollut.,* **226** : 479 - 485. https://doi.org/10.1016/j.envpol.2017.04.045

GEORGE, T., NASEEMA, B. S., MATHEW, T. B., PAUL, A., XAVIER, G. AND RAVI, P. K., 2014, Dissipation and safety of spiromesifen on tomato. *Pestic. Res. J.*, **26** (2) : 160 - 163.

JACKSON, M. L., 1973, Soil chemical analysis. Prentice Hall of India Pvt. Ltd., New Delhi, pp. : 134 - 204.

KAMALJEET, S. S. AND GURPREET, S. M., 2016, Efficacy assessment of fipronil 0.6% GR against rice stem borers and leaf folder. *J. Exp. Zool. India*, **19** (2) : 857 - 861.

KUMAR, R. AND SINGH, B., 2013. Persistence and metabolism of fipronil in rice (*Oryza sativa* linnaeus) field. *Bull. Environ. Contam.Toxicol.*, **90** (4) : 482 - 488.

MAHAPATRA, S., DEEPA, M., JAGDISH, G. K., RASHMI, N., KUMAR, S. AND PRAKASH, G. S., 2010, Fate of fipronil and its metabolites in/on grape, leaves, berries and soil under semi-arid tropical climate conditions. *Bull. Environ. Contam. Toxicol.*, **84** : 587 - 591.

Mukherjee, A., Bhattacharya, A., Sarkar, S. and Ghosh, R. K., 2021. Dissipation behaviour and risk assessment of fipronil and its metabolites in a paddy ecosystem. *Environ. Monit. Assess.*, **193** : 348.

Piper, C. S., 1966, Soil and plant analysis. *Inter-science Publishers, Inc.*, New York, pp. : 368.

Pose-Juan, E., Marín-Benito, J. M., Sánchez-Martín, M. J. and Rodríguez-Cruz, M. S., 2018, Dissipation of herbicides after repeated application in soils amended with green compost and sewage sludge. *J. Environ. Manage.*, **223** : 1068 - 1077.

Rasool, S., Rasool, T. and Gani, K. M., 2022, A review of interactions of pesticides within various interfaces of intrinsic and organic residue amended soil environment. *Chem. Eng. J. Adv.*, 11 : 100301.

Sarker, A., Kim, T. K., Kim, S. I. and Jeong, W. T., 2023, Influence of soil types and organic amendment during persistence, mobility and distribution of phorate and terbufos in soils. *Korean J. Pestic. Sci.*, **27** (3) : 242 - 258.

Savary, S., Willocquet, L., Pethybridge, S. J., Esker, P., Mcroberts, N. and Nelson, A., 2019, The global burden of pathogens and pests on major food crops. *Nat. Ecol. Evol.*, **3** : 430 - 439.

Silva, R. O., Scorza, R. P., Bonfa, M. R. L., Campanari, M. F. Z. and Mendes, I. C., 2016, Degradation and sorption of fipronil and atrazine in Latossols with organic residues from sugarcane crop. *Soil Sci.*, **46** (7) : 1172 - 1177.

Subbiah, B. V. and Asija, G. L., 1956, A rapid procedure for determination of available nitrogen in soils. *Curr. Sci.*, **25** (4) : 259 - 260.

Tang, J., Li, J., Wu, F., Zhang, J. and Feng, Y., 2020, Effects of soil properties and flooding on the degradation and sorption of fipronil in sediments. *Ecotoxicol. Environ. Saf.,* **201** : 110823.

Toolkiattiwong, P., Arunrat, N. and Sereenoonchai, S., 2023, Environmental, human and ecotoxicological impacts of different rice cultivation systems in Northern Thailand. *Int. J. Environ. Res. Public Health*, **20** (3) : 2738.

Walkley, A. J. and Black, C. A., 1934, An examination of the method for determining soil organic matter and a proposed modification of the chromic acid titration. *Soil Sci.*, **37** (2) : 28 - 29.

Wu, X., Yu, Y., Xu, J., Dong, F., Liu, X., Du, P., Wei, D. and Zheng, Y., 2017, Residue analysis and persistence evaluation of fipronil and its metabolites in cotton using high performance liquid chromatography-tandem mass spectrometry. *PLoS One*, **12** (3) : e0173690.

Yang, Y., Li, X., Liu, J., Zhou, Z., Zhang, T. and Wang, J., 2020, Microbial community structure and metabolic function in the rhizosphere of Suaeda salsa and their effect on pesticide degradation. *Environ. Pollut.*, 257 : 113586.

You, X. W., Jiang, H. T., Zhao, M., Suo, F. Y., Zhang, C. S., Zheng, H., Sun, K., Zhang, G. Y., Li, F. M. and Li, Y. Q., 2020, Biochar reduced Chinese chive (*Allium tuberosum*) uptake and dissipation of thiamethoxam in an agricultural soil. *J. Hazard. Mater.*, 390 : 121749.

Yu, H., Liu, Y., Wang, X. and Zhang, J., 2020, Equilibrium, kinetic and thermodynamic studies on atrazine adsorption in soils under different moisture regimes. *Environ. Sci. Pollut. Res.*, **27** : 22147 - 22158.