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Dissipation kinetics of diclosulam 84% WDG in soils of four different agro-climatic regions under laboratory simulated condition

P. GANGULY, S. R. BARIK, AND A. BHATTACHARYYA

Department of Agricultural Chemicals, Bidhan Chandra Krishi Viswavidyalaya, Mohanpur-741252, Nadia, West Bengal, India,

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ABSTRACT

Laboratory simulation studies were performed to investigate dissipation kinetics of diclosulam 84% WDG in soils of four different agro-climatic regions viz.new alluvial soil from Mohanpur, red & lateritic soil from Jhargram, coastal saline soil from Canning and black soil from Pune at the rates of 1.0 and 2.0 ìg ml^{-1}. Soil samples collected on 0 (2h after application), 3, 7, 15, 30, 60 and 90 days after applicationwere processed for residue analysis of diclosulam by HPLC. Diclosulam was dissipated linearly with progress of time irrespective of dose and substrate.The calculated half-life values for black soil (pH 8.14) was found to be in the range between 28.67-29.51 days whereas for saline soil (pH 7.92) it was 30.41-34.21 days, for red and lateritic soil (pH 5.56) it was 35.42-40.14 days and for new alluvial soil (pH 6.85) it was 40.68-43.63 days irrespective of treatment doses.

Keywords: Diclosulam, dissipation, half-life, pH

Diclosulam (N-(2,6-dichlorophenyl)-5-ethoxy-7-fluoro[1,2,4]triazolo-[1,5-c]pyrimidine-2-sulfonamide)belongs to triazolopyrimidine sulfonamide class of herbicide (Fig. 1).

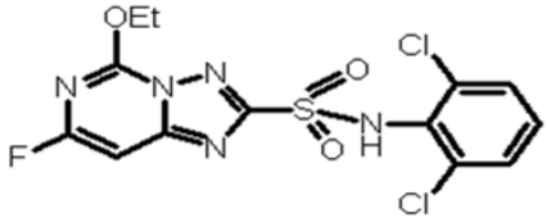

Fig. 1: Structure of diclosulam

The persistence action of diclosulam makes itself a very suitable tool for control of broadleaf weeds in soybeans and peanuts fields. Since 1990, it has been tested in the laboratory as well as on field and was first registered in 1997 in Argentina and Brazil. It is registered in India since 2014 & recommended for broad spectrum weed control in soybean crop @ 22-26 g.a.i. ha^{-1} in between 0-3 days after sowing. Dose of post-emergent application of diclosulam employ can be lowered down than soil-applied treatments. Diclosulam can act as both soil-applied and post-emergent. It can be soil applied in any tillage system since it does not require incorporation. The herbicide is a highly active, low dose compound. Diclosulam is having high mobility in the environment due to its anionic form&low sorption coefficient (Koc). It is generally observed that laboratory data generated for diclosulam are very much in line with findings in field studies such as rapid field dissipation rates, metabolite formation patterns, etc. Hence, the present study has been undertaken to investigate the dissipation kinetics of diclosulam in soils of four different agro-climatic regions of India under laboratory simulated conditions. The results could be utilized for further research in determining the fate of this herbicide in Indian soils in field conditions

MATERIALS AND METHODS

Experimental details

The present study has been designed to find out the dissipation kinetics of diclosulam in soils of four different agro-climatic regions *viz.*new alluvial soil from Mohanpur, red & lateritic soil from Jhargram, coastal saline soil from Canning and black soil from Pune under laboratory simulated condition (Physico-chemical properties of these soils are given in the table 1). Diclosulam analytical standard (99.5%) as well as diclosulam 84% WDG formulation (~11.905 mg), supplied by M/S Dow AgroSciences Ltd., were separately taken in two 100 ml volumetric flasks. The volumeswere made up to the mark with HPLC grade methanol to prepare stock standard of 100 µg ml^{-1} of each formulation & technical grade. Necessary dilutions were made from these standards as and when required.

Table 1: Physico-chemical properties of different soils

Type of Soil	Texture	pH	Bulk density (g.cm^{-3})	Organic carbon (%)
New alluvial	Sandy loam	6.85	1.28	0.76
Red &lateritic	Sandy	5.56	1.58	0.52
Coastal saline	Silty loam	7.92	1.45	1.21
Black soil	Clayey	8.14	1.60	0.67

Email: pritam0410@gmail.com

Soil samples (50 g) of each type were taken in 250 ml conical flasks and 10 ml of water was added to it. Then 1 ml and 2 ml of the 50 µg ml^{-1} stock solution of diclosulam 84% WDG were added to conical flasks separately containing 50 g of different soil samples. The initial concentrations became 1 µg g^{-1} (T_1) and 2 µg g^{-1} (T_2) respectively. The control soils (50 g) received 10 ml of water only. Three replicate flasks for each treatment were taken for analysis on each day of sampling along with untreated control.Samples (three replicates) were processed for analysis of diclosulam residues at intervals of 0 (2h after application), 3, 7, 15, 30, 60 and 90 days after application.

Extraction and clean up

Soil samples in the respective sampling dates were added with 100 ml mixture of acetone: water (8:2), kept in overnight and were shaken for a period of 30 minutes using a mechanical shaker (25ÚC). It was then filtered and the extract was collected and re-extracted the sample using 100 ml mixture of acetone:water (8:2). Combined filtrate was concentrated to evaporate acetone portion and then transferred to a 500 ml separatory funnel. Then 100 ml of distilled water was added to it. This mixture was partitioned thrice with 200 (100+50+50) ml dichloromethane. Dichloromethane fraction was collected through anhydrous Na_2SO_4. This combined fraction was concentrated to 1-2 ml in rotary vacuum evaporator at 40ÚC.

A chromatographic column was packed up with a mixture of 10 g silica gel and florisil (1: 1). Anhydrous sodium sulphate was placed in the bottom and top of the column using n-hexane. The residue was transferred in to the column. Elution was done with 100 ml hexane followed by 100 ml of hexane:dichloromethane (8:2) mixture and then 100 ml methanol. Methanol fraction was evaporated to dryness in a rotary vacuum evaporator at 40ÚC and the volume was reconstituted in HPLC grade methanol for HPLC analysis.

HPLC – operating parameters

Thermo C18 column (250 mm X 4.6 mm; Reversed Phase) was used for chromatographic separation with methanol: water (1:1) as the mobile phase at a flow rate of 0.5 ml min^{-1}. Under these working conditions diclosulam was detected (at ëmax = 235 nm) at the retention time of 5.36 ± 0.20 min(Fig. 2). The LOD and LOQ of the method were determined as 0.02 and 0.05 µg g^{-1}, respectively(Fig 3).

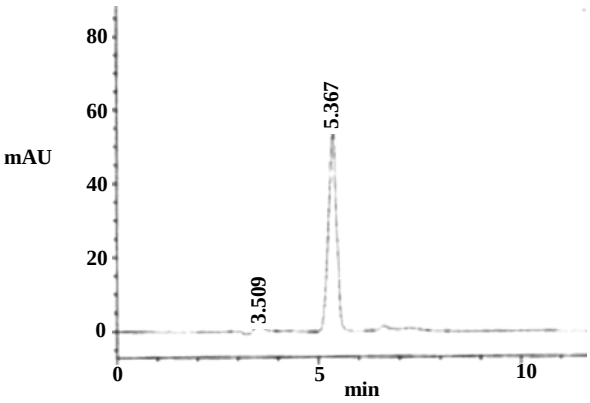

Fig. 2: Chromatogram of diclosulam analytical standard under HPLC operating conditions Recovery study

Recovery studies were carried out in order to establish the reliability of the analytical method and to know the efficiency of extraction and clean up steps employed in the present study, by fortifying the soil samples of different types with 0.05, 0.10 and 1.00 µg g^{-1} of diclosulam 84% WDG.

RESULTS AND DISCUSSION

Results of recovery study

Average recoveries of diclosulamin soils fortified at 0.05, 0.10 and 1.00 µg ml^{-1}were 88.67% for new alluvial soils, 89.67% for red & lateritic soils, 88.33% for coastal saline soils and 91.67% for black soils (Table 2).Hence the analytical method was quite satisfactory and adopted for the present study.

Table 2: Recovery study of diclosulam 84% WDG in different soil samples

Fortification level (µg ml^{-1})	Recovery (%)			
	New alluvial soil	Red & lateritic soil	Coastal saline soil	Black Soil
0.05	86.00	92.00	88.00	94.00
0.10	89.00	90.00	91.00	89.00
1.00	91.00	87.00	86.00	92.00
Average	88.67	89.67	88.33	91.67
Recovery factor	1.13	1.12	1.13	1.09

Persistence in soil

The mean residue values, percent dissipation, regression equation and half-life values of diclosulam in four different types of soils are presented in table 3. From the table it was revealed that diclosulam dissipates linearly with the progress of time,which was supported by Murdock*et.al.*(1999)[3] who found thatdiclosulam followed first order rate kinetics. Residues were below

Table 3: Dissipation of diclosulam in different soils at different days interval

Days	Mean± S.D. (% Dissipation) (µg g^{-1})							Regression Equation	Half- life ($T_{1/2}$) (days)
	0	3	7	15	30	60	90		
					New alluvial soil (pH 6.85)				
T_1	0.99±0.01 (-)	0.91±0.02 (8.42)	0.82±0.02 (17.51)	0.79±0.02 (20.20)	0.66±0.01 (33.00)	0.35±0.02 (64.31)	0.21±0.02 (79.12)	Y=2.9954- 0.0074X	40.68
T_2	1.97±0.01 (-)	1.90±0.02 (3.72)	1.74±0.01 (11.51)	1.61±0.02 (18.44)	1.37±0.02 (30.63)	0.78±0.03 (60.41)	0.47±0.03 (75.97)	Y=3.3041-0.0069X	43.63
					Red &lateritic soil (pH 5.56)				
T_1	0.98±0.01 (-)	0.91±0.01 (6.80)	0.86±0.01 (12.79)	0.74±0.02 (25.25)	0.59±0.02 (40.07)	0.34±0.02 (65.99)	0.16±0.02 (83.84)	Y=3.0005-0.0085X	35.42
T_2	1.99±0.01 (-)	1.95±0.01 (1.84)	1.80±0.03 (9.55)	1.65±0.03 (17.25)	1.21±0.02 (39.20)	0.64±0.03 (67.67)	0.45±0.03 (77.22)	Y=3.3080-0.0075X	40.14
					Coastal saline soil (pH 7.92)				
T_1	0.95± 0.02 (-)	0.88± 0.02 (7.02)	0.79± 0.02 (16.49)	0.68± 0.02 (28.07)	0.49± 0.02 (48.77)	0.24± 0.01 (75.09)	0.12± 0.01 (87.02)	Y=2.9761-0.0099X	30.41
T_2	1.91± 0.01 (-)	1.88± 0.01 (1.75)	1.63± 0.02 (14.66)	1.35± 0.02 (29.32)	0.88± 0.03 (53.93)	0.58± 0.03 (69.81)	0.30± 0.03 (84.12)	Y=3.2711-0.0088X	34.21
					Black soil (pH 8.14)				
T_1	0.92± 0.01 (-)	0.88± 0.01 (4.71)	0.78± 0.02 (15.22)	0.59± 0.02 (35.51)	0.39± 0.03 (57.97)	0.18± 0.02 (80.07)	0.11± 0.01 (87.68)	Y=2.9462-0.0105X	28.67
T_2	1.95± 0.02 (-)	1.90± 0.03 (2.39)	1.60± 0.02 (17.78)	1.30± 0.02 (33.16)	0.81± 0.02 (58.63)	0.47± 0.02 (76.07)	0.24± 0.02 (87.86)	Y=3.2765-0.0102X	29.51

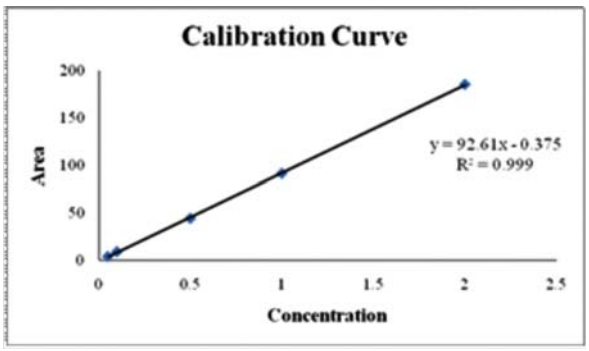

Fig 3: Calibration curve of areas corresponding to different concentrations of analytical standard of diclosulam

detectable limits (BDL) of 0.05 µg g^{-1} in all untreated control samples (T_3) throughout the study.

In new alluvial soil, the initial residues and half-life value of diclosulam were ranged between 0.99-1.97 µg g^{-1} and 40.68-43.63 days, irrespective of T_1 (1.0 µg g^{-1} of soil) and T_2 (2.0 µg g^{-1} of soil). More than 60% of initial residues were dissipated within 60 days after application in both the cases. The initial residues and half-life value of diclosulam were ranged between 0.98-1.99 µg g^{-1} and 35.42-40.14 days, irrespective of T_1 (1.0 µg g^{-1} of soil) and T_2 (2.0 µg g^{-1} of soil) in red & lateritic soil.In both the cases,65% of initial residues were dissipated within 60 days after application.For coastal saline soil, the initial residues and half-life value of diclosulam were ranged between 0.95-1.91 µg g^{-1} and 30.41-34.21 days, irrespective of T_1 (1.0 µg g^{-1} of soil) and T_2 (2.0 µg g^{-1} of soil). More than69% of initial residues were dissipated within 60 days after application in both the cases.In case of black soil (pH 8.14), the initial residues and half-life value of diclosulam were ranged between 0.92-1.95 µg g^{-1} and 28.67-29.51 days, irrespective of T_1 (1.0 µg g^{-1} of soil) and T_2 (2.0 µg g^{-1} of soil). Around 75% of initial residues were dissipated within 60 days in both the cases, irrespective of the treatment.

The present study shows that diclosulam degrades somewhat rapidly in black soil than in coastal saline, red and lateritic and new alluvial soil. The half-life values of different soils were in the range between 28-43 days. Similar observation is found by Sheppard *et al.* (1997) who reported that the half-life of diclosulam is approximately 33 to 65 days and is degraded by common soil microorganisms. Dissipation t of diclosulam ranged from 16 to 54 days in the U.S. and South American soils incubated at dark as reported by Yoder *et al*. (2000). According to Krämer *et al.*(2007), analysis of soil samples from bare-ground applications of Diclosulam gave half-life ranged from 13-43 days. Organic matter content and soil temperature were found to be the two factors that most influenced the soil degradation rates. Soil pH has minimal effect upon this.

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