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To study the degradation and downward movement of oxyfluorfen in sandy, sandy loam and clayey soils

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Abstract
In order to study the persistence and degradation of oxyfluorfen from three different types of soils *viz*., sandy, sandy loam and clayey soil, sandy soil was collected from Agronomy Farm of Sardar Krushinagar Dantiwada Agricultural University, Dantiwada. Sandy loam soil was collected from Bidi Tobacco Research Station Farm of Anand Agricultural University, Anand and clayey soil from Organic Farm of Navsari Agricultural University, Navsari. The persistence of oxyfluorfen in different soils followed first order degradation kinetics and dissipated in one phase. The residues of oxyfluorfen recorded in sandy soil on 0 day was 0.408 µg g^{-1} with half-life (DT_{50}) of 43.00 days. The residues of oxyfluorfen recorded in sandy loam soil on 0 day was 0.496 µg g^{-1} with half-life (DT_{50}) of 60.20 days. The residues of oxyfluorfen recorded in clayey soil on 0 day was 0.498 µg g^{-1} with half-life (DT_{50}) of 75.25 days. The order of persistence of oxyfluorfen in soil denoted by half-life i.e. DT_{50} observed in the experiment were as follows: Clayey soil (75.25 days) > Sandy loam soil (60.20 days) > Sandy soil (43.00 days).

Keywords: Oxyfluorfen, Sandy-Sandy Loam-Clayey Soil, PCV Column, Persistency, QuEChERS, GLC-ECD

Introduction
Oxyfluorfen is formulated for agricultural use as an emulsifiable liquid concentrate and as a granular product, although it is most frequently used in a liquid formulation for food crops and as a granular formulation for ornamental nursery crops for the weed control. Oxyfluorfen is used for the control of annual grasses and broad-leaved weeds in tropical and sub-tropical crops, by pre- or post-emergence application in maize, rice, gram, groundnut, soybean, cotton and onion crops. It is selective contact herbicide and absorbed more readily by the foliage than by the roots, with very little translocation. Its application as liquid or dry formulations on the crop or soil may contaminate soil or leave residues on crop produce.
Oxyfluorfen is moderately persistent in most soil environments, with a representative field half-life of about 30 to 40 days. Oxyfluorfen is not subject to microbial degradation or hydrolysis. The main mechanism of degradation in soils may be photodegradation and evaporation/co-distillation in moist soils. In laboratory studies, its soil half-life was 6 months, indicating very low rates of microbial degradation. Soil binding is highest in soils with high organic matter and clay content. Once oxyfluorfen is adsorbed to soil particles, it is not readily removed. It is practically insoluble in water, and therefore is unlikely to be appreciably mobile in most instances, unless the sorptive capacity of the soil is exceeded.
Oxyfluorfen did not leach below 10.2 cm in any soil except sand. In water, oxyfluorfen is rapidly decomposed by light. Because oxyfluorfen is nearly insoluble in water and has a tendency to adsorb to soil, it will be sorbed to suspended particles or sediments. There is very little movement of oxyfluorfen within treated plants. It is not readily metabolized by plants, but since it is not readily taken up by roots, residues in plants are generally very low.

Material and Methods
Persistence of oxyfluorfen in different soils
A laboratory study was conducted at All India Network Project on Pesticide Residues, Centre for Organic Farming, ICAR, Unit-9, Anand Agricultural University, Anand-388 110, Gujarat (India).

Three types of soils which were denoted as S_1, S_2 and S_3 for sandy, sandy loam and clayey soil, respectively were collected from Sardar Krushinagar Dantiwada Agricultural University, Dantiwada, Anand Agricultural University, Anand and Navsari Agricultural University, Navsari, respectively. These soils were air dried passed through 2 mm sieve and used for the study. A 36 cm long PVC column with an internal diameter (i.d.) of 6 cm was used by marking into sections each of 6 cm. The bottom ring was tied up with muslin cloth with thread to avoid contamination of leachate with soil particles from bottom and 30 cm column from bottom was filled with soil. Before applying the herbicide, soil column was wetted to their apparent water holding capacity by applying 0.01 M $CaCl_2$ to the top of column at an interval of 24 h. After this initial equilibration 30 mg/column of oxyfluorfen was applied to the top of column. Polythene sheet was placed on each column in order to prevent volatization. The entire experiment was conducted at room temperature.

To study the persistence of oxyfluorfen in sandy, sandy loam and clayey soils, each soil type (10 g) was taken in a 50 mL capacity of polypropylene tube separately in triplicate with a control. Each polypropylene tube was fortified @ 0.5 µg g^{-1}.

Extraction (Qu EChERS method)

Arepresentative 10 g soil sample was taken in 50 mL polypropylene centrifuge tube, and 20 mL acetonitrile was added followed by mixture of 1 g NaCl + 4 g $MgSO_4$ and shaken vigorously by hand for 1 minute before centrifuge. The tubes were centrifuged at 3500 rpm for 3 minutes. A 10 mL aliquot from the supernatant was transferred by auto pipette in to a 15 mL polypropylene centrifuge tube containing 1.5 g $MgSO_4$ and 0.25 g PSA, followed by centrifugation at 2500 rpm for 2 minute. A 4.0 mL aliquot was transferred into a glass test tube and acetonitrile was completely evaporated on a TurboVap (AOAC, 2007) [3]. Final volume was made up to 1.0 mL using 1:1 (petroleum spirit: acetone) and subjected to ECD analysis on a Varian 450 GC-ECD.

The statistical analysis was carried out in the Microsoft Excel Version-2007 programme with the help of computer. The average, Standard Deviation (S.D.), regression equation, R^2, half-life and residues (µg g^{-1}) were calculated in excel software.

Result and Discussion

Validation of oxyfluorfen extraction method from different soils

Prior to dissipation study of oxyfluorfen, different types of soils *viz*., sandy, sandy loam and clayey were subjected to recovery study. Each soil type was spiked at 2 different levels i.e. 0.1 and 0.5 µg g^{-1} and further analysed The recovery experiment was performed with 7 replications along with a control and reagent blank. The results obtained from the recovery study revealed that mean recovery of oxyfluorfen from sandy, sandy loam and clayey soils was 106.15, 112.85 and 119.56 % at 0.1 µg g^{-1} level of fortification, respectively. The corresponding values for 0.5 µg g^{-1} fortification level was 94.79, 106.05 and 96.72 %. Percent RSD_{WR} recorded for sandy, sandy loam and clayey soils at 0.1 µg g^{-1} spiking level was 2.10, 3.50 and 2.36 %. The corresponding values for 0.5 µg g^{-1} spiking level were 4.88, 4.10 and 3.63 %. LOD determined at 0.1 µg g^{-1} for sandy, sandy loam and clayey soils was 0.007, 0.011 and 0.007µg g^{-1}, respectively. The corresponding values for 0.5 µg g^{-1} spiking level were 0.007, 0.013 and 0.011 µg g^{-1}. The LOQ at 0.1 µg g^{-1} fortification level for sandy, sandy loam and clayey soils were 0.021, 0.033 and 0.022 µg g^{-1}, respectively. The corresponding values for 0.5 µg g^{-1} fortification levels were 0.020, 0.039 and 0.034 µg g^{-1}.

The analytical method employed for the extraction of oxyfluorfen from soil was found accurate and precise as mean recovery was in the range of 94.79 to 119.56% and RSD_{WR} was 2.10 to 4.88 %which was within the limits of ≤ 20 % as prescribed by the SANCO (2011) [16]. According to the SANCO (2011) [16] guidelines, any analytical method which records mean recovery in the range of 70-120% and % RSD_{WR} ≤ 20% is the sufficiently accurate and precise. Hence the method employed for the extraction of oxyfluorfen from different soils is accurate and precise.

Persistence of oxyfluorfen in different types of soils

Persistence study of herbicides is important to determine potential water and soil contamination and verify the actual herbicide concentration in the soil to assure long term weed control. Taking this point under consideration, an investigation was carried out to determine the persistence of oxyfluorfen in three different soils *viz*., sandy, sandy loam and clayey. Prior to persistence study, physico-chemical properties of each soil were determined which are mentioned in Table 3.1 of Materials and Methods chapter. The detailed methodology of the experiment is mentioned in Chapter-3. The persistence of oxyfluorfen was determined at 0.5 µg g^{-1} and samples were collected on 0, 1, 3, 5, 10, 20, 30, 45 and 60 days after application. The data obtained from the study were subjected to regression analysis to determine the persistence and half-life (DT_{50}) which is mentioned in Table.1

Sandy soil

The residues of oxyfluorfen recorded in sandy soil on 0 day was 0.408 µg g^{-1}. This was dissipated at faster rate in this soil. The residues of oxyfluorfen recorded on 1, 3, 5, 10, 20, 30, 45 and 60 days after treatment were 0.407, 0.404, 0.347, 0.346, 0.304, 0.271, 0.198 and 0.149 µg g^{-1}, respectively. The dissipation pattern is depicted in Figure.1. The persistence of oxyfluorfen in sandy soil followed first order kinetics. In order to work out the rate of degradation, BDL level was calculated as half of 0.021 µg g^{-1} (BDL for sandy soil) and thus the degradation showed first order kinetics with half-life of 43.00 days.

Sandy loam soil

The residues of oxyfluorfen recorded in sandy loam soil on 0 day was 0.496 µg g^{-1}. The residues of oxyfluorfen recorded on 1, 3, 5, 10, 20, 30, 45 and 60 days after treatment were 0.442, 0.439, 0.437, 0.428, 0.422, 0.417, 0.361 and 0.225 µg g^{-1}, respectively (Table.1) The dissipation pattern is depicted in Figure.4. The limit of detection (LOD) and limit of quantification (LOQ) of oxyfluorfen determined for sandy loam soil was 0.011 and 0.033 µg g^{-1}, respectively. In order to work out the rate of degradation, BDL level was calculated as half of 0.033 µg g^{-1} (BDL for sandy loam soil) and thus the degradation showed first order kinetics with half-life of 60.20 days.

Clayey soil

The residues of oxyfluorfen recorded in clayey soil on 0 day was 0.498 µg g^{-1} for standard dose of oxyfluorfen. The residues of oxyfluorfen recorded on 1, 3, 5, 10, 20, 30, 45 and 60 days were 0.495, 0.488, 0.482, 0.479, 0.446, 0.440, 0.239

and 0.195 µg g^{-1}, respectively. The dissipation pattern is depicted in Figure.3. The limit of detection (LOD) and limit of quantification (LOQ) of oxyfluorfen determined for clayey soil was 0.010 and 0.030 µg g^{-1}, respectively. In order to work out the rate of degradation, BDL level was calculated as half of 0.030 µg g^{-1} (BDL for clayey soil) and thus the degradation showed first order kinetics with half-life of 75.25 days.

Table 1: Persistence of oxyfluorfen in different soils

	Mean Residues (µg g^{-1})					
Days	**Sandy**		**Sandy loam**		**Clayey**	
	T_0 (Control)	**T_1 (0.5 µg g^{-1})**	**T_0 (Control)**	**T_1 (0.5 µg g^{-1})**	**T_0 (Control)**	**T_1 (0.5 µg g^{-1})**
0	ND	0.408	ND	0.496	ND	0.498
1	ND	0.407 (0.25)	ND	0.442 (10.89)	ND	0.495 (0.60)
3	ND	0.404 (0.98)	ND	0.439 (11.49)	ND	0.488 (2.01)
5	ND	0.347 (14.95)	ND	0.437 (11.90)	ND	0.482 (3.21)
10	ND	0.346 (15.20)	ND	0.428 (13.71)	ND	0.479 (3.82)
20	ND	0.304 (25.49)	ND	0.422 (14.92)	ND	0.446 (10.44)
30	ND	0.271 (33.58)	ND	0.417 (15.93)	ND	0.440 (11.65)
45	ND	0.198 (51.47)	ND	0.361 (27.22)	ND	0.239 (52.01)
60	ND	0.149 (63.48)	ND	0.225 (54.64)	ND	0.195 (60.84)
DT_{50} (Days)		43.00		60.20		75.25
R^2		0.984		0.993		0.958
Regression equation		y = -0.0071x +1.6132 (0-60 day)		y = -0.005x + 1.654 (0-60 day)		y = -0.004x + 1.704 (0-60 day)

*Figures in parentheses indicate percentage of total loss.
ND = Not detected, BDL = Below Determination Limit
BDL = 0.021 µg g^{-1}, 0.033 µg g^{-1} and 0.022 µg g^{-1} for sandy, sandy loam and clayey soil, respectively.

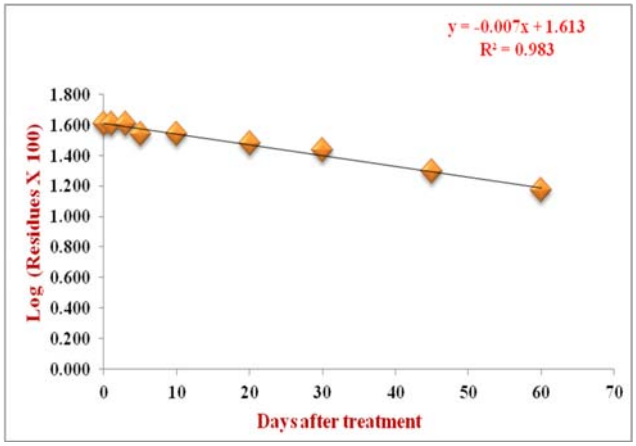

Fig 1: Persistence of oxyfluorfen in sandy soil

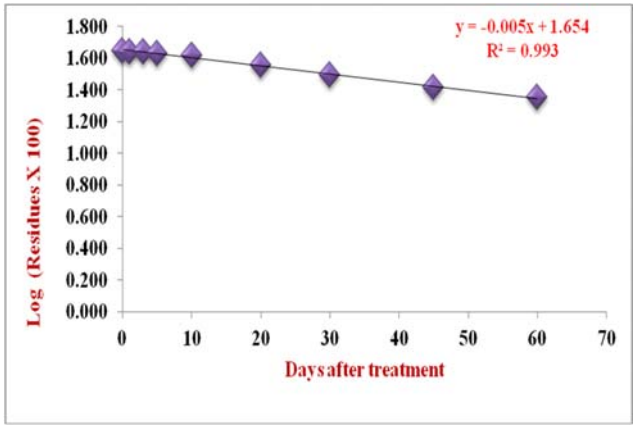

Fig 2: Persistence of oxyfluorfen in sandy loam soil

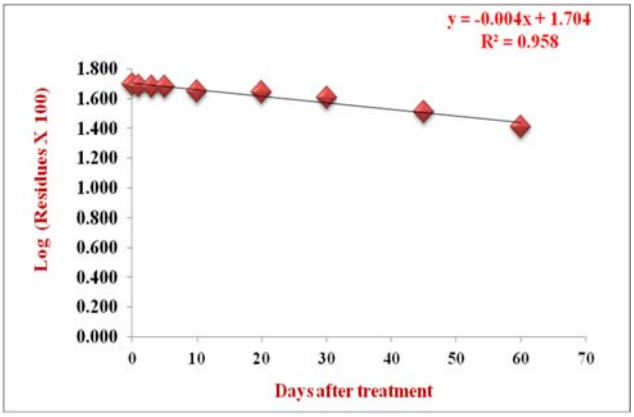

Fig 3: Persistence of oxyfluorfen in clayey soil

Conclusion

1. The persistence of oxyfluorfen was the highest in clayey soil followed by sandy loam and sandy soils.
2. The downward movement of oxyfluorfen in column study was up to 18 cm in sandy soil. In cash of sandy loam and clayey soil residues was below determination limit. This confirms the low risk of sub-soil contamination in sandy loam and clayey soils compared to the sandy soil.
3. High adsorption of oxyfluorfen on soil makes it hazardous from groundwater contamination point of view.

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