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Optimizing Pre-emergence Herbicide Oxyfluorfen Dose based on Its Sorption and Desorption for Effective Weed Management under Different Soil Types

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ABSTRACT

Background: Adsorption and subsequent release of pre-emergence herbicide into the soil solution decides the herbicide availability to kill the weeds. Herbicides applied on clay or organic rich soil type, absorb more results in lowering its availability in soil solution become sub lethal to kill the weeds, whereas sandy soil adsorb less and desorb more, sometime it increases the herbicide concentration to toxic level to crop. Effective weed control could be achieved by studying the variability of soil and deciding the dose based on the sorption and desorption properties of the soil.

Methods: With this information on the background a laboratory experiment was conducted at the Department of Agronomy, Agricultural College and Research Institute, Tamil Nadu Agricultural University, Madurai, India during 2021 to study the optimum level of oxyflourfen concentration in soil solution required to kill the weeds effectively. The study on sorption and desorption was carried out with pre-emergence herbicide oxyflourfen with five different concentrations in five different soil types.

Result: The highest sorption of 98.53 per cent was observed with clayey soils followed by sandy clay loam soils with high organic matter content (96.97 per cent). The lowest sorption (77.96 per cent) was recorded with sandy loam soils. The highest desorption of 57.44 per cent was recorded with sandy loam soil and the lowest desorption percentage (13.18 per cent) was obtained with sandy clay loam soil with higher organic matter. Based on the analytical results of sorption and desorption, oxyflourfen dose was optimized as 0.53 kg ha^{-1} for sandy clay loam soils with high organic matter, 0.34 kg ha^{-1} for clay soil, 0.29 kg ha^{-1} for sandy clay, 0.21 kg ha^{-1} for sandy clay loam soil and 0.12 kg ha^{-1} for sandy loam soils. In conclusion, the clay soil required more quantity of oxyflourfen compared to other types of soil for effective management of weeds under irrigated condition.

Key words: Desorption, Dose optimization, Herbicide, Oxyfluorfen, Soils, Sorption, Weed.

INTRODUCTION

Oxyfluorfen (2-chloro-4-trifluoromethylphenyl-3-ethoxy-4-nitrophenyl ether) is widely used herbicide to control certain annual broadleaf and grassy weeds (Kidd and James, 1991) in crops like groundnut, cotton, sunflower, onion, citrus, olive crops and asparagus (Hermosin *et al*., 2013). It inhibit protoporphyrinogen oxidase and produced necrosis in treated plants (Vencill, 2002). Light is the important factor for its herbicidal effects (Ensminger and Hess, 1985). It can be used as pre and post emergence herbicide (Thakare *et al*., 2002).

The behaviour of herbicides in soil depends on the physico-chemical characteristics of the herbicide, the active surface of the soil mineral, organic composition (Triantafyllidis, 2012) and the amount of the herbicides applied. Efficiency of any soil applied herbicides in generally pre-emergence herbicides is based on the adsorption and subsequent release in to the soil solution (Wauchope and Koskinen, 1983 and Kalpana *et al*., 2002). Sorption and desorption of herbicide refers to bonding of the chemical to the sites on soil mineral or organic surfaces and release from these in to the soil solution to maintain equilibrium. Intermolecular forces like low and high molecular bonds, ionic and ligand bonds, Vander Waals forces are responsible for the behaviour in soil (Senesi and Testini, 1984). These mechanisms are influenced by soil properties (Stevenson, 1972). Adsorption of herbicides by soils has frequently been found to be correlated with soil type, organic matter and clay contents. Herbicides exhibiting strong adsorption with organic matter and clay content (Sha'Ato, 2012). Adsorption and desorption is an important process for determining the ultimate fate of herbicides in soil because detoxification mechanisms such as degradation, metabolism, microbial uptake and mobilization are operate only on the non-sorbed fractions of the chemical to the sites on soil mineral or organic surfaces. Adsorption-desorption influences the mobility, persistence, degradation and volatility of pesticide in soil (Kalpana *et al*., 2002).

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The interaction between herbicide and soil properties greatly affects its weed control efficacy and the potential for crop injury (Koch and Khosla, 2007). Soil organic matter content appeared to be a predominant factor influencing herbicide retention (Tang *et al*., 2009). Chemical property of oxyfluorfen is associated with a strong organic soil and clay adsorption (Vencill, 2002) that results in limited soil mobility (Alister *et al*., 2009).

Generally blanket recommendation is followed for weed control in various crops. Soils are vary greatly in types with diverse organic matter content. Herbicides applied on clay or organic rich soil type, absorb more results in decreased availability in soil solution (Johnson and Sims, 1993), become sub lethal to kill the weeds. Continuous exposure to mild and sub lethal dose leads to herbicide resistant weeds. On the other hand herbicides applied in the sandy natured soil get released more to soil solution excessively and become toxic to crops plants. Effective weed control could be achieved by studying the variability of soil and deciding the dose based on the sorption and desorption property of the soil (Bauer and Schefcik, 1994). Rather blanket method, “one soil-one crop-one dose” is the right approach to reduce the toxicity as well as prevent the herbicide resistant weeds (Mohammadzamani and Rashidi, 2009). Based on the background information the present study was carried out to optimize the pre-emergence herbicide oxyfluorfen dose based on it sorption and desorption under different soil types for effective weed control.

MATERIALS AND METHODS

Laboratory experiment was conducted in the Department of Agronomy and Centre of Innovation Laboratory, Agricultural College and Research Institute, Tamil Nadu Agricultural University, Madurai, Tamil Nadu, India to study the sorption and desorption capacity of sandy clay, sandy clay loam with high organic matter, clayey soil, sandy loam, sandy clay loam collected from Kovilpatti, Kodaikanal, Aaduthurai, Chettinad, Madurai regions for the experiment. Based on the results, the herbicide dose was optimized and same has been described below. Soil physico chemical properties were furnished in Table 1.

Sorption and desorption studies

A. Sorption study procedure according to OECD, 2000

Five gram of processed soil samples were taken in centrifuge tubes to which different concentration (1, 2, 3, 4, 5 ppm) of oxyfluorfen prepared in 0.01 M $CaCl_2$ was added. The soil solution with 1:5 ratio was used for this study. This soil solution suspension in centrifuge tube was shaken for 4 hours in an orbitory shaker at ambient temperature to attain equilibrium. After equilibration the soil solution suspension was centrifuged at 2000 rpm for 15 minutes and the supernatant was filtered through Paul Nylon Membrane Filters (<2mm). The final filtered solution was analysed for the sorption.

B. Desorption procedure

Soil remaining after sorption studies was added with 0.5 N $CaCl_2$. Shaking procedure was done as that of sorption studies. After shaking it was centrifuged with 2000 rpm for 15 minutes. Supernatant was filtered through Paul Nylon Membrane Filter. It was repeated till desorption for 3 cycles.

Adsorption isotherm

The adsorption isotherm was obtained by plotting data of oxyfluorfen adsorbed against equilibrating concentrations. The isotherm data were interpreted in terms of Freundlich and Langmuir equation.

Freundlich equation:

$$\frac{X}{m} = KC^{1/n}$$

Langmiur equation:

$$\frac{C}{x/m} = \frac{1}{kb} + \frac{C}{b}$$

Whereas,

X/m = Amount of herbicide (X) adsorbed per gram of soil (g).

C = Herbicide concentration in equilibrium solution.

K = Constant.

A straight line was plotted as log (X/m) Vs. log C. The intercept in equal to log K and slope to 1/n. K constant provided extent of adsorption and used for determining the relative importance of the various soil properties on adsorption. Theoretically, Freundlich equation represents a situation in which the quantity of herbicides adsorbed increases indefinitely with increasing concentration. Langmuir equation can be used to find out sorption maxima mg g^{-1}, bonding energy (K) and maximum buffering capacity (1/intercept).

Oxyfluorfen residue analysis

Filtrate was dried in rotary evaporator and rinsed with acetone of 100 ml and it evaporated to make final volume of 10 ml. Content was transferred to a glass column filled with 10 g of $NaSO_4$ and pinch of charcoal (Eluted with 5 ml of acetone). Again dried in water bath until removal of complete moisture. This was rinsed with 2 ml of acetone and collected in the vial for GCMS analysis.

Prerequisite for determination of oxyfluorfen in GC

QP 2020 model GCMS from Shimadzu was used with Mass spectrophotometer as a detector. Rx 5 Sil MS column was used for oxyfluorfen separation. GC temperature conditions: injection temperature was 250°C. The oven temperature was programmed as follows; initial temperature was 120°C then increased to 170°C at 8°C/min and held for 4.5 minutes, increased to 280°C at 9°C/min and held for 5.5 minutes. Detector temperature was 225°C. Carrier gas flow into the column was 1.1 ml/min. Retention time for the oxyfluorfen peak was 19.807 (Fig 1). Calibration curve with standard of five levels were prepared with five concentration with R^2 value of 0.96 and Routine Limit of Quantification (RLOQ) were 0.001 ppm.

Herbicide dose optimization formula

% adsorbed herbicide in soil = X.
Remaining herbicide in equilibrium solution (Y) = 100 - X.
Actually % desorbed herbicide from % adsorbed = a.

Available % herbicide desorbed from 100% herbicide added (b) =

$$\frac{X \times a}{100}$$

Total available herbicide in soil solution after adsorption and desorption (C) = Y+ B

This percentage was the mean of 1, 2, 3, 4, 5 ppm equilibrium solution. So mean of these concentrations were 3 ppm.

So the % of available herbicide in soil solution was converted to 3 ppm = D

$$D = \frac{C}{100} \times 3$$

Available herbicide concentration after adsorption (F) = $\frac{D}{3}$

Herbicide concentration required to kill weeds = P

$$\left.\begin{array}{l}\text{Ratio between herbicide application rate and zero day} \\ \text{herbicide concentration after herbicide spray (S)}\end{array}\right\}$$

$$= \frac{R}{Q} \text{ kg ha}^{-1}$$

Optimized concentration of herbicide spray (D) = $\frac{P}{S}$

RESULTS AND DISCUSSION

Sorption of oxyfluorfen

Observation for sorption of oxyfluorfen on different soil types results are furnished in Table 2, 3, 4, 5, 6 and 7.

Sorption of pre-emergence herbicide oxyfluorfen varied from 98.53 to 77.96 per cent in different soils. Oxyfluorfen sorption was higher (98.53 per cent) in clayey soil with a mean adsorption of 2.9504 mg kg^{-1} (Table 6). It was followed by sandy clay loam soil with high organic matter (96.974 per cent) with the mean adsorption of 2.883 mg kg^{-1} (Table 5). Lowest sorption was recorded in sandy loam soil with 2.275 mg kg^{-1} (77.955 per cent) (Table 6), which contain more of kaolinite clay type.

Such a differential sorption was, in fact, more prominent at higher concentration of added oxyfluorfen. Results further highlighted that with an increase in initial oxyfluorfen concentration, sorption gets declined. The sorption increased to 2.9504 and 2.883 mg kg^{-1} due to increased in clay (Table 6) and organic matter content (Table 5) respectively. Sorption percentage isotherm were also confirmed the results (Table 8).

Sorption isotherms were well described by Freundlich and Langmure equation. This result was validated with the results of Kadlag, 2011. Based on Langmuir equation, maximum sorption (158.73 mg g^{-1}) was recorded with clayey soil, it was followed by sandy clay loam soil with high organic matter (91.74 mg g^{-1}) (Table 7). Lowest sorption was recorded with sandy loam soil. So sorption of oxyfluorfen was recorded in the following rising order: Clayey soil > sandy clay loam (rich in organic matter) > sandy clay > sandy clay loam > sandy loam (Table 7). Same trend was fallowed in Freundlich equation (Table 8). Clay and organic matter content were the most important factor for the adsorption of pesticides (Narayanan *et al*., 2014).

Bonding energy (5.85 K) was the highest with sandy clay soil, this might be the reason for lowest desorption values. Freundlich equation is given in table 8. Freundlich constant K gave intensity of absorption. K values were ranged from 1.15 to 0.09 L kg^{-1}. Based on K value, higher K value (1.15 L kg^{-1}) was recorded with clayey soil, it was followed by sandy clay loam soil (rich in organic matter) 0.56 L kg^{-1}, the lowest k value (0.09 L kg^{-1}) was recorded with sandy loam soil (Table 8). The sorption of herbicide associated with exchangeable cations and hydrogen bonding between C=O, functional group, carboxyl oxygen was assumed to contribute to bond formation between clay and herbicide (Weiping *et al*., 2000).

Desorption of oxyfluorfen

Results for the desorption studies of oxyfluorfen with different soil types are furnished in Table 2, 3, 4, 5, 6.

Highest desorption of 57.44 percentage was recorded from sandy loam soil with desorbed herbicide concentration of 1.243 mg kg^{-1} (Table 4). This concluded that sandy loam soil doesn’t hold the oxyfluorfen herbicide for a longer period. It was followed by sandy clay loam soil, which recorded 27.54 per cent desorption with the release of 0.691 mg kg^{-1} (Table 2). Sandy clay soil representing soil desorbed 22.44 per cent, which is lesser than clayey soil type (22.41 per cent) (Table 3). Lowest desorption percentage (13.18 per cent) was obtained from the sandy clay loam soil texture with higher organic matter (Table 5). This might be due to higher bonding energy (Majee and Reddy, 2023). Same result was obtained by (Rai *et al*., 2000). Especially soil contains 2:1 type montmorillonite clay (Sandy clay soil, clayey soil) absorbs more than kaolinite clay. Oxyfluorfen showed lower desorption percentage from the adsorbed (Cooper *et al*., 1994).

Desorption of soil tracks the following order of sandy loam > sandy clay loam > sandy clay > clayey soil > sandy clay loam with higher organic matter, it is in line with Sireesha *et al*. (2013).

Herbicide dose optimization for oxyfluorfen

After maximum sorption, oxyfluorfen available in soil solution is maximum with sandy loam soil which recorded 22.05 per cent of availability, followed by sandy clay loam soils (16.49 per cent) location. Herbicide available in sandy clay soil solution (8.03 per cent) was higher than sandy clay loam soil with high organic matter (3.03 per cent). The lowest availability of herbicide in soil solution after maximum herbicide sorption was recorded in clayey soil with 1.47 per cent. Total availability of herbicide acquired from the

Table 1: Physico-chemical properties of Collected soil samples.

Parameters	ARS, Kovilpatti	DARS, Chettinad	TRRI, Aduthurai	AC and RI, Madurai	HRS Kodaikanal
I. Physical properties					
Clay (%)	44	24	52	34	29
Slit (%)	9	9	7	8	13
Sand (%)	47	69	40	53	50
Textural class	Sandy clay	Sandy loam	Clayey	Sandy clay loam	Sandy clay loam
Bulk density (gcc^{-1})	1.11	1.25	1.10	1.18	1.05
% pores space (%)	61.00	50.00	63.00	47.17	50.00
Particle density	1.54	2.86	1.52	2.00	1.82
II. Chemical properties					
pH	7.79	6.35	7.20	7.50	4.45
EC (dS m^{-1})	0.23	0.20	0.35	0.46	0.52
Organic carbon (%)	0.28	0.55	0.52	0.925	4.20

ARS- Agricultural Research Station, Kovilpatti, Tamil Nadu, India.
DARS- Dry land Agriculture Research Station, Chettinadu, Tamil Nadu, India.
TRRI- Tamil Nadu Rice Research Institute, Aaduthurai, Tamil Nadu, India.
AC and RI- Agricultural College and Research Institute, Madurai, Tamil Nadu, India.
HRS- Horticulture Research Station, Kodaikanal, Tamil Nadu, India.

Table 2: Sorption and desorption of oxfluorfen in sandy clay loam soil.

Oxyfluorfen added (mg liter^{-1})	Equilibrium concentration of oxyfluorfen (C) (mg litre^{-1})	Amount of oxyfluorfen adsorbed (mg kg^{-1})	% adsorbed	Amount of oxyfluorfen desorbed	% oxyfluorfen desorbed
1	0.140	0.860	86.000	0.241	28.023
2	0.232	1.768	88.400	0.453	25.622
3	0.540	2.460	82.000	0.622	25.285
4	0.790	3.210	80.250	0.911	28.380
5	0.955	4.045	80.900	1.230	30.408
Mean	0.531	2.469	83.510	0.691	27.544

Table 3: Sorption desorption of oxyfluorfen in sandy clay soil.

Oxyfluorfen added (mg kg^{-1})	Equilibrium concentration of oxyfluorfen (C) (mg kg^{-1})	Amount of oxyfluorfen adsorbed (mg kg^{-1})	% adsorbed	Amount of oxyfluorfen desorbed	% oxyfluorfen desorbed
1	0.053	0.947	94.700	0.201	21.225
2	0.169	1.831	91.550	0.350	19.115
3	0.263	2.737	91.233	0.592	21.630
4	0.310	3.690	92.250	0.871	23.604
5	0.494	4.506	90.120	1.200	26.631
Mean	0.258	2.742	91.971	0.643	22.441

Table 4: Sorption and desorption of oxyfluorfen in sandy loam soil.

Oxyfluorfen added (mg kg^{-1})	Equilibrium concentration of oxyfluorfen (C) (mg kg^{-1})	Amount of oxyfluorfen adsorbed (mg kg^{-1})	% adsorbed	Amount of oxyfluorfen desorbed	% oxyfluorfen desorbed
1	0.181	0.819	81.900	0.651	79.487
2	0.320	1.680	84.000	0.873	51.964
3	0.690	2.310	77.000	1.025	44.372
4	0.893	3.107	77.675	1.654	53.235
5	1.540	3.460	69.200	2.011	58.121
Mean	0.725	2.275	77.955	1.243	57.436

Table 5: Sorption and desorption of oxyfluorfen in sandy clay loam soil with high organic matter content.

Oxyfluorfen added (mg kg^{-1})	Equilibrium concentration of oxyfluorfen (mg kg^{-1})	Amount of oxyfluorfen adsorbed (mg kg^{-1})	% adsorbed	Amount of oxyfluorfen desorbed	% oxyfluorfen desorbed
1	0.001	0.999	99.900	0.121	12.112
2	0.045	1.955	97.750	0.203	10.384
3	0.089	2.911	97.033	0.532	18.276
4	0.155	3.845	96.125	0.760	19.766
5	0.297	4.703	94.060	0.251	5.337
Mean	0.117	2.883	96.974	0.373	13.175

Table 6: Sorption and desorption of oxyfluorfen in clayey soil.

Oxyfluorfen added (mg kg^{-1})	Equilibrium concentration of oxyfluorfen (C) (mg kg^{-1})	Amount of oxyfluorfen adsorbed (mg kg^{-1})	% adsorbed	Amount of oxyfluorfen desorbed	% oxyfluorfen desorbed
1	0.01	0.990	99.000	0.213	21.515
2	0.025	1.975	98.750	0.522	26.430
3	0.043	2.957	98.567	0.740	25.025
4	0.051	3.949	98.725	0.942	23.854
5	0.119	4.881	97.620	1.034	21.184
Mean	0.050	2.9504	98.5324	0.6902	23.6016

Table 7: Langumuir constants: sorption maxima (mg g^{-1}), bonding energy (K), maximum buffering capacity, regression equation and R^2 calculated for a different oxyfluorfen concentration.

Soil texture	Langmuir constant		Maximum buffering capacity	Regression equation	R^2
	Sorption maxima (mg g^{-1})	Bonding energy (K)			
Sandy clay loam (Rich in organic matter)	91.74	0.77	70.42	y = 0.0142x + 0.0109	0.963
Clayey soil	158.73	2.17	344.83	y = 0.0029x + 0.0063	0.681
Sandy clay	17.27	5.85	101.01	y = 0.0099x + 0.0579	0.616
Sandy clay loam	8.29	4.62	38.31	y = 0.0261x + 0.1207	0.689
Sandy loam	8.00	2.29	18.35	y = 0.0545x + 0.125	0.766

Table 8: Freundlich sorption coefficients (*K*, *1/n*), partition coefficients *K*oc, Regression coefficient and R^2 calculated for a different oxyfluorfen concentration.

Soil texture	Freundlich constant		Regression equation	R^2
	K	1/n		
Sandy clay loam (Rich in organic matter)	0.56	3.77	y = 0.2654x + 1.7497	0.936
Clayey soil	1.15	1.48	y = 0.6735x + 3.1713	0.975
Sandy clay	0.70	1.39	y = 0.722x + 2.0184	0.975
Sandy clay loam	0.33	1.40	y = 0.7154x + 1.3954	0.948
Sandy loam	0.09	1.52	y = 0.6599x + 1.0891	0.932

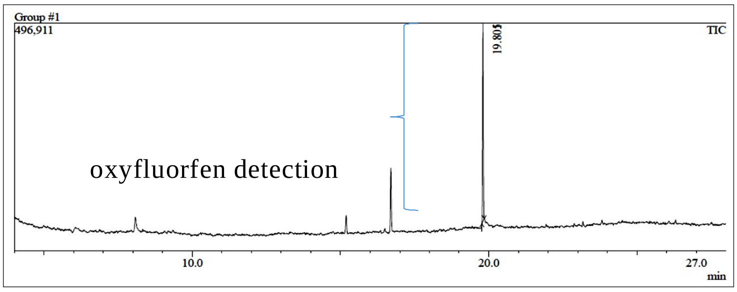

Fig 1: Herbicide residue detection.

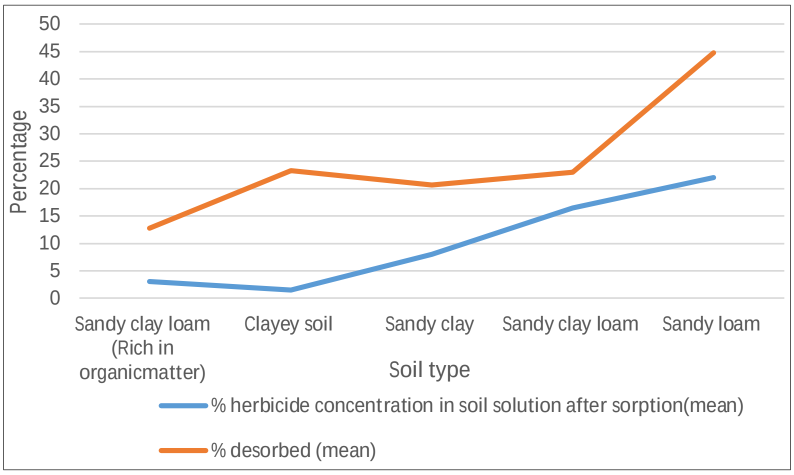

Fig 2: Percentage of herbicide available after sorption and desorption.

Table 9: Critical concentration of herbicide in soil solution to kill the weeds.

Herbicide	Critical concentration to kill weeds	Dose of herbicide sprayed (kg/ha)	Amount of herbicide observed in zero day (ppm)
Oxyfluorfen	10 ppm	0.20	24
Oxyfluorfen	14 ppm	0.75	34

Table 10: Herbicide dose optimization for oxyfluorfen.

Soil type	Total availability of herbicide in soil solution (mg kg^{-1})	Optimized herbicide concentration (kg/ha)	% variation from normal recommendation (0.25 kg/ha)
Sandy clay loam (Rich in organicmatter)	15.80	0.53	+163.67
Clayey soil	24.72	0.34	+68.51
Sandy clay	28.67	0.29	+45.34
Sandy clay loam	39.49	0.21	+5.52
Sandy loam	66.89	0.12	-37.64

available herbicide in soil solution and desorption was highest in sandy loam textured (66.89%) soils followed by sandy clay loam texture with 39.49%. The least total availability of 15.80% was recorded in sandy clay loam soil with high organic matter content (Fig 2).

Based on the current sorption, desorption and critical herbicide concentration required to weed control study (Table 9), optimum concentration of oxyfluorfen was worked out for each soil based on sorption, desorption and. Highest rate of 0.53 kg ha^{-1} is required for the sandy loam soils texture with high organic matter, which was 163.67 per cent more than the current recommendation (0.2 kg ha^{-1}) (Table 10). The recommendation calculated for the clayey soil is 0.34 kg ha^{-1} (Table 10). This was 68.51 per cent increased percentage over normal recommendation. For the sandy clay and sandy clay loam soil, the required rate of herbicide is 0.29 and 0.21 kg ha^{-1} with 45.34 percent and 5.52 per cent increase from normal recommendation, respectively (Table 10). The lowest dose of 0.12 kg ha^{-1} oxyfluorfen is recommended for sandy loam soil, which is 37.64 per cent higher than the normal recommended dose (Table 10). This results are on par with Carrara *et al*. (2004).

CONCLUSION

Sorption and desorption varied with soil type. The highest sorption of 98.53 per cent was observed with clayey soils followed by sandy clay loam soils with high organic matter content (96.97 per cent). The lowest sorption (77.96 per cent) was recorded with sandy loam soils. The highest desorption of 57.44 percent was recorded with sandy loam soil and the lowest desorption percentage (13.18 per cent) was obtained with sandy clay loam soil with higher organic matter. Based on the analytical results of sorption and desorption, oxyflourfen dose may be recommended as 0.53 kg ha^{-1} for sandy clay loam soils with high organic matter, 0.34 kg ha^{-1} for clay soil, 0.29 kg ha^{-1} for sandy clay, 0.21 kg ha^{-1} for sandy clay loam soil and 0.12 kg ha^{-1} for sandy loam soils.

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Author's contribution

Conceptualization and methodology development: S. Selvakumar and K. Shoban Chakravarthy; formal analysis, original draft preparation, visualization: K. Shoban Chakravarthy, P. Baskar, C. Sangeetha.

Compliance with ethical standards

Conflict of interest

The authors declare that they have no conflicts of interest.

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