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Persistence and carryover effect of oxyfluorfen residues in red sandy clay loam soil

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Abstract

A Field experiments were conducted for two years at Agricultural Research Station, Bhavanisagar of Tamil Nadu Agricultural University, Coimbatore, India during *kharif* season of 2010 and 2011 to evaluate the new formulation of oxyfluorfen (23.5% EC) on weed control in onion and their residual effect on succeeding crops. Herbicides are being chemical in nature, excessive and repeated use may pose phytotoxicity to crop plants, carryover effects on succeeding crops and also leads to adverse effects on non-target organisms. This might due to causes of health hazards to humans, animals and the environment. Many herbicides are bound residues which make them not only unavailable to the targets, but also polluting the soil ecosystem in a number of ways. Thus monitoring of herbicides residue in soil, plant and other matrixes are very much important. Therefore, a laboratory and field experiments were undertaken to investigate the persistence of oxyfluorfen in soil and onion crop under red sandy clay loam soil. Based on two years field experimentation, phytotoxicity symptoms of oxyfluorfen was complete recovery of affected on onion got completely recovered plant could be observed subsequently in 30 days after herbicide application (DAHA); and the phytotoxicity was not evident thereafter in onion plant. Field persistence of oxyfluorfen at varying doses applied in soil and residue content in onion plant sample were well below the prescribed maximum residue limits ($0.05 \mu\text{g g}^{-1}$) at the time of harvest. Residue of oxyfluorfen herbicide dissipated faster in plant than in soil. It is highly bound to the soil organic carbon and has low mobility in red sandy clay loam soil which indicates the binding of this herbicide to soil particles is high. Bioassay results showed that the oxyfluorfen herbicide to be secure on the succeeding crops and this might be due to detoxification of herbicide in soil and do not adversely affect the growth and yield attributes of the succeeding crops.

Keywords: Onion, oxyfluorfen, phytotoxicity, GC, persistence, bioassay, succeeding crop safety.

Introduction

Onion is one of the important bulbous vegetable crop of economic importance and widely cultivated all over the world, with particular distribution in the Asian continent and in Europe. Many scientists have pointed out that onion plants are poor competitor of weeds. The poor competitive ability with its initial growth and lack of adequate foliage makes onion weak against weeds was given by Smith *et al.* (2008) [15]. In addition, their cylindrical upright leaves do not shade the soil to block weed growth. Unrestricted weed growth reduced the bulb yield upto 40-80 per cent depending upon the nature of intensity and duration of weed competition and also the crop was more found to be more sensitive to weed competition between two to six weeks after its emergence was given by Prakash *et al.* (2000) [12]. Critically viewing, the manual and mechanical methods of weed control in onion, besides being less effective, costly and time demanding it requires as well as need to be repeated weeding at frequent intervals (Tesfay Amare, 2014) [18]. Under such situations, chemical method of weed control has shown good promise in a variety of crop with the advancement of agriculture and technology. Several workers have found pendimethalin, oxyfluorfen and oxadiazon to be efficient in controlling the weeds in onion. Oxyfluorfen is widely used by the farmers as it is a low dose herbicide and it is easy to apply either in pre or post-emergence. As herbicides occupy 47 per cent of the agrochemicals usage, there could be a chance for the bioaccumulation and biomagnifications of the components or its metabolites in the onion crop, which may cause ailing effects to human being through food chain.

Active ingredient in oxyflourfen (23.5% EC) is a 2-Chloro-1-(3-ehtoxy-4-nitrophenoxy)-4-(trifluoromethyl) benzene belonging to a diphenyl-ether group of herbicide. It is a pre or post-emergence herbicide used for control of annual and perennial broad leaved weeds in a variety of field crops. Oxyflourfen is a contact, herbicide and light is required for its herbicidal activity in plants. Oxyflourfen acts by free radical formation *via* inhibition of protoporphyrinogen oxidase (Protopx), which is the enzyme that converts protoporphyrinogen IX to protoporphyrin IX and also induces necrosis in applied plants.

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Oxyfluorfen degrades in soil mainly by photolysis and to some extent by evaporation in moist soil surfaces (WSSA, 1994) [22]. The dissipation of oxyfluorfen was usually moderate with the average field half-life of 35 days (Wauchope *et al.*, 1992) [21] and typically ranges from 30-40 days. While, reported the persistence of oxyfluorfen residues in the rhizosphere soil of rice upto 60 days after application (Das *et al.*, 2003) [4]. In maize crop, more than 95 per cent of atrazine dissipated from field at the time of crop harvest. The half-life values were found to be 9.38 - 21.54 in soil was reported by Janaki *et al.* (2012) [8]. Oxyfluorfen residues present in the soil and rice grain upto harvest (100 days) and suggested, there is a chance for the bioaccumulation of residues if it is used at higher dose and also continuously (Sondhia, 2009) [16]. Oxyfluorfen is classified as a highly toxic and persistent herbicide, which persists in soil and accumulates in terrestrial plants and certain aquatic environments through runoff (USEPA, 1992) [19]. Recently, oxyfluorfen has been registered in India for pre-emergence herbicide in crops and is used widely for annual crops. Many field experiments revealed the bio-efficacy of this herbicide and its persistence in soil grown with rice crop; however, no information is available on the persistence of this herbicide in soils grown with bulbous vegetable onion under Indian tropical conditions. Therefore, laboratory and field experiments were undertaken to investigate the behavior of oxyfluorfen in soil and its bioaccumulation in onion plant. Gas chromatograph with Electron Capture Detector (GC-ECD) was standardized for the micro quantitative determination of oxyfluorfen from soil and onion plant and the persistence of oxyfluorfen in field soil and plant sample was studied.

Most of the presently available herbicides provide only a narrow spectrum of weed control. Many of them have are actively only on annual species, while a few are only effective against perennial weeds. Plenty of the new formulation of herbicides is recommended for each crop and in a cropping system, sequential application of herbicides for every crop leads to residue accumulation in soil and crop, thus causing adverse effect of succeeding crops. Most of the herbicides are selective and specific to the crop and the residue persists in the soil for few months to a few years depending upon the chemical and concentration used. Knowledge on the persistence and residual effect of herbicides in soil is essential to use them safely, effectively and for non-hazardous chemical weed control schedules. Bioassay remains a major tool for qualitative and quantitative determination of herbicides residue in soil. Jayakumar (1987) [10] reported that detection of the presence of an herbicide can be done by bioassay which measures the biological response of a living plant to the herbicide. Considering the above facts, an attempt has been made to study the carryover effect of herbicides applied to *kharif* onion on succeeding *rabi* sunflower and pearl millet crops was formulated with the following objectives,

- ❖ To examine the phytotoxicity effect of oxyfluorfen (23.5% EC) in onion crop,
- ❖ To assess the residual toxicity of the oxyfluorfen in field soil and on onion crop,
- ❖ To evaluate the residual effect of oxyfluorfen on succeeding crops.

Materials and Methods

Field experiments

Field experiment pertaining to the persistence of oxyfluorfen residue in onion and the field soil was conducted at the farm

of Agricultural Research Station, Bhavanisagar, Tamil Nadu, India. The experimental farm was located in Western Zone of Tamil Nadu is at 11°29'N latitude and 77°08'E longitude with an altitude of 256 m above MSL. Experiments were laid out in Randomized Block Design and the treatments were replicated thrice. The experimental field soils were of red sandy clay loam in texture. The size of each plot was 3 m × 2 m. Small onion (*Allium cepa* var. *aggregatum* L.) variety CO 4 was planted during 1st June 2010 and 2011. The test chemical (oxyfluorfen 23.5% EC) obtained from M/S. Crystal Crop Protection Pvt. Ltd., New Delhi was applied at five different dosages *viz.*, 150, 200, 250, 300 and 400 g a.i. ha⁻¹, as a pre-emergence spray in onion sown field at 3rd day after planting (DAP) with the help of a knapsack sprayer. Soil samples were drawn randomly from 0-10 cm depth using a tube auger from 6-7 spots in each plot. Soil sample was collected approximately 500 g from each plot. The samples were collected at 0 (2 hrs), 10, 30, 45 and 60 day time intervals after the second treatment of herbicide and at the crops harvest time from all the treated and control plots. Samples were mixed thoroughly and spread on a glass plate and divided into four parts (quarters). Soil of two opposite quarters was retained, rejecting the remaining two. The process was further repeated to obtain 100 g of representative sample for the final analysis. A whole plant samples (about 500 g) from each plot was collected at 0 (2 hrs), 10, 15, 30 and 45 days. The plant samples were chopped to small pieces, homogenized and a representative sample of 500 g from each treatment was processed for analysis. The soil of the experimental field was red sandy clay loam with pH 6.90, EC 0.19 dS m⁻¹ and 0.54% organic carbon (Walkley and Black, 1934) [20].

Crop phytotoxicity assessment through visual observation

Crops response is also rated in the scale 0-10 to record herbicide toxicity on plant stand and growth. Visual assessment of crop response is based on such effects as plant kill, crop growth and population and also injury to plants *etc.*, by a particular herbicide treatment. This was done by using the scale of 0-10, where 0 represents no effect (crop no injury and normal) and 10 correspond to complete effect (complete destruction of crop).

Field persistence study of oxyfluorfen (23.5% EC)

Chemicals, reagents and soil

A reference standard of oxyfluorfen (purity 99.5%) and the test chemical of oxyfluorfen 23.5% EC was supplied by Crystal Crop Phosphates Ltd., (CCPL), Private Agency, New Delhi, India. All the solvents were analytical grade and purchased locally. Anhydrous sodium sulfate (AR grade) was used as a drying agent for different samples. For GC analysis, HPLC-grade acetone and 0.2µm filtered milli-Q water were used.

Instrument and operating conditions

A Thermo Gas chromatography (model GC8610) equipped with Electron Capture Detector (GC-ECD) was used for quantitative analysis and a computer enabled software (IRIS 32) was used for recording chromatograms. The stationary phase consisted of megapore capillary column (50 m x 0.53 mm; ID-BP-1; 0.5µM). Nitrogen was used as a carrier with a flow rate of 10 mL/min. GC analysis was carried out in the following temperature conditions: Injector port – 240 °C; Oven – 210 °C which was hold for 15 minutes; Detector – 260 °C. The approximate retention time of oxyfluorfen standards

and samples with the above said conditions was 4.92 ± 0.2 min. A calibration curve was prepared by plotting concentrations of oxyfluorfen on the x-axis against the average peak area on the y-axis.

Extraction and clean-up

Valid representative laboratory samples of soil from each replication of each treatment were homogenized and 50 g was weighed. A quantity of 100 mL of acetone was added to flasks containing soil and shaken on a horizontal shaker for 2 hrs. The contents of the flasks were allowed to settle, and the supernatant phase was filtered in a flask through a Buchner funnel using a water pump. The extraction was repeated twice with the same solvent (50 mL each time) and filtered in the same flask. The combined filtrated was transferred to a glass column filled with anhydrous sodium sulfate and activated charcoal and eluted with 100 ml of acetone. Eluted layer was concentrated on rotary vacuum evaporator at 60 °C to moistened level and finally re-dissolved in acetone for GC analysis. The plant samples (after cutting into very small pieces) were homogenized and ground into fine pieces and the oxyfluorfen extraction was done for soil samples.

Method Validation and detection limits

Oxyfluorfen technical material (100 mg) was taken in a 100 mL volumetric flask and dissolved in acetone, and the volume was brought up to the mark to obtain a stock solution containing 1000 $\mu\text{g mL}^{-1}$. From this stock solution, working standards of 10, 5, 2.5, 1.0, 0.5, 0.1, 0.05, 0.01 and 0.005 $\mu\text{g mL}^{-1}$ concentration of oxyfluorfen were prepared by serial dilution (fig. 1) Then, 0.5 μL of each working standards were injected into GC and the peak area measured to have a linearity check study (Table 1).

Linearity Check Study

A linearity check study was carried out with the help of analytical standard. In this study a calibration curve was prepared by taking the areas corresponding to different concentrations of analytical standard.

Calculation

The amount of oxyfluorfen in the sample was calculated with the following formula:

$$\text{Residue in ppm } (\mu\text{g g}^{-1}) = \frac{A_1 \times C \times V_1}{A_2 \times W} \times R_f$$

Where,

A_1 = Area of compound from sample, in chromatogram

A_2 = Area of compound from standard, in chromatogram

V_1 = Total volume of sample in ml

C = Concentration of analytical standard (95.0%) in ppm

W = Weight of the sample in g

R_f = Recovery factor

Validation of method was performed in terms of recovery studies before the analysis of unknown samples. Whole onion plant and bulb samples (25 g) or 10 g soil samples were weighed and added into extraction flasks. One milliliter of standard solution of 1.0 to 0.005 $\mu\text{g mL}^{-1}$ oxyfluorfen was added uniformly on the surface of the matrix and mixed before adding extraction solvent. The extraction and cleanup processes were then performed as described in the methodology. Quantification of oxyfluorfen residues was

accomplished by comparing the peak response for samples with peak area of the standards. The Instrumental detection limits (IDL) and estimated method detection limit (EMDL) for oxyfluorfen was done as described in GC (Sondhia, 2010) [17].

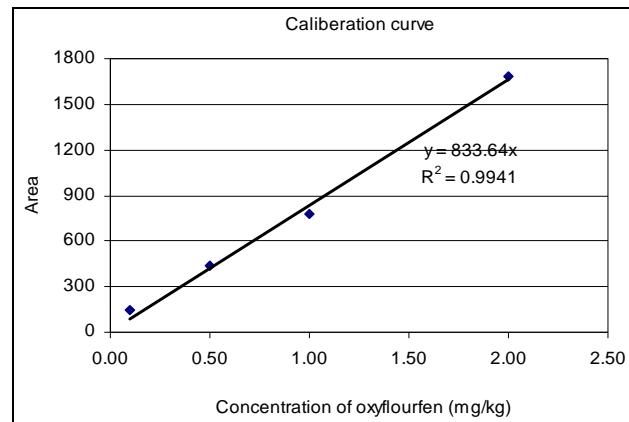


Fig 1: Calibration curve of area corresponding to different concentration of analytical standard of Oxyflourfen

Table 1: Area corresponding to different concentration of analytical standard of Oxyflourfen

Concentration ($\mu\text{g mL}^{-1}$)	Area
0.05	149.81
0.10	440.23
0.50	783.92
1.00	1683.25

Gas chromatography analysis

A sample solution in acetone was filtered through a 0.2 μm membrane using a millipore syringe filter. A 0.5 μL sample was injected into GC by 5 μL GC syringe. The area was recorded and the concentration in the sample was calculated.

Bioassay study of oxyfluorfen residues

The onion crop was harvested on the first week of October during both the years. After harvesting of the onion crop to know the residual effect of oxyfluorfen (23.5% EC) herbicide, without disturbing the layout of each plot was manually prepared for sowing of succeeding crops. Seven rows of each succeeding sunflower and pearl millet were sown in each plot in *rabi* season. The germination percentage, plant height, dry weight of plants and yield of sunflower (CO 4) and pearl millet CO (Cu) 9 crops were recorded for the test crops to evaluate bioassay of herbicide residues.

Results and Discussion

Phytotoxicity effect on onion plant

Phytotoxicity symptom of oxyfluorfen at higher doses of 300 and 400 g ha^{-1} was visualized in onion during both the years of study. Oxyfluorfen at 300 and 400 g a.i. ha^{-1} as pre-emergence application showed phytotoxicity symptoms like bleaching, leaf tip burn, leaf curling and stunting of growth upto 30 DAHA in onion. Application of oxyfluorfen at higher dose of 400 g a.i. ha^{-1} caused moderate crop damage with a rating of 3 at 7 DAHA. Phytotoxicity symptom was visualized even at 21 DAHA, with a rating of 1 and the plants were not completely recovered from phytotoxicity symptoms. Complete recovery of affected plants could be observed only after 30 DAHA and the phytotoxicity was not evident thereafter. Channappagoudar and Biradar (2007) [3] reported

that application of oxyfluorfen at 400 g a.i. ha⁻¹ controlled weeds effectively but resulted in lower yield because of due to its higher phytotoxicity on onion crop and it was observed only for a short period. There was no phytotoxicity symptoms observed in onion crop with application of oxyfluorfen at lower doses *viz.*, 150, 200 and 250 g a.i. ha⁻¹ and oxyfluorfen (goal) at 200 g a.i. ha⁻¹. Daugovish *et al.* (2008)^[5] was noticed that application of pre-emergence application of oxyfluorfen at 300 to 600 g a.i. ha⁻¹ resulted in 30 per cent injury in strawberry. Banga *et al.* (1998)^[2] observed that application of oxyfluorfen at 150 g a.i. ha⁻¹ showed phytotoxicity symptoms in pea as a post emergence herbicide. Pre-emergence application of oxyfluorfen at 200 g ha⁻¹ reduced the density of broad leaved weeds (70 to 90%) in onion compared with non-treated plots was given by Sathya Priya *et al.* (2013)^[14].

Persistence of oxyfluorfen (23.5% EC) under field condition

IDL, recovery of oxyfluorfen and EMDL

Under the given conditions of GC, oxyfluorfen resolved at 4.65 min as a single sharp peak. The IDL for oxyfluorfen by GC was 0.01 µg mL⁻¹ and the calibration curve was linear ($r^2 = 0.9979$) from 0.05 to 10 µg mL⁻¹. The EMDL by this method using equation was found to be 0.001 and 0.005 µg g⁻¹ of soil and plant, respectively and 0.0001 µg mL⁻¹ of water.

Residue of oxyfluorfen in onion field soil

Degradation is defined as a substantive change in the molecular makeup of the given herbicide, with a component of the parent molecule removed by some process to form a metabolite or metabolites. Dissipation is considered to be the sum of all possible outcomes of the parent herbicide. An herbicide molecule can dissipate by the process of chemical or microbial degradation which indicates a chemical change in the parent. The inverse process of dissipation is herbicide persistence. Persistence is often considered to be a negative association, such as herbicide persistence that damages rotational crops or herbicide persistence that causes contamination of rivers and groundwater aquifers (Mueller and Senseman, 2015)^[11]. On the other hand, herbicide persistence can allow for residual weed control to provide maximum agricultural productivity in managed ecosystems. Application of oxyfluorfen was sprayed to tuber vegetable crop field at all five rates of application (150, 200, 250, 300 and 400 g a.i. ha⁻¹). Herbicide residues were monitored upto harvest after its last application. At 0 (2 hrs) days after herbicide application (DAHA), initial deposits of oxyfluorfen were observed as 0.1079, 0.1295, 0.1403, 0.1608 and 0.2275 µg g⁻¹ of soil at 150, 200, 250, 300 and 400 g a.i. ha⁻¹, respectively (Table 2). At 30 and 45 DAHA, the highest concentration of oxyfluorfen active substance was determined in soil from plots where it was applied at 400 g a.i. ha⁻¹ (0.0281 and 0.0092 µg g⁻¹) and it was followed by 300 g a.i. ha⁻¹ (0.0229 and 0.0064 µg g⁻¹) and 250 g a.i. ha⁻¹ (0.0193 and 0.0048 µg g⁻¹). Increase in doses of oxyfluorfen herbicide progressively increased the residue content in soil. An average field half life of oxyflourfen was 35 days (WSSA, 1994)^[22]. In The present study, showed that lower half life of oxyfluorfen was due to the enhanced photolysis of oxyfluorfen by the high temperature (28 °C to 40 °C) and sunshine hours (6.1 hrs) prevailed during the early crop growth period. As the soil photolysis is the significant route of oxyfluorfen degradation as it proceeds via cleavage of the either bridge (Janaki *et al.*, 2012)^[8] the formation of photo

degradates is unlikely; hence the metabolites of oxyfluorfen in the soil was not studied. The low solubility of oxyfluorfen in water (0.116 mg L⁻¹ at 20 °C) might have got retained it in soil surface and could have augmented the faster photochemical decomposition from soil as reported for metamifop. These results were in accordance with the findings of Janaki and Chinnusamy (2012)^[7]. Soil organic matter and clay content might have also influenced the dissipation of oxyfluorfen from soil. At 60 DAHA, no oxyfluorfen residues could be detected in soil samples with higher doses of oxyfluorfen at 300 and 400 g a.i. ha⁻¹. Residues of **oxyfluorfen** applied in soil were well below the prescribed maximum residue limit (0.05 µg g⁻¹) at the time of harvest.

Residue of oxyfluorfen in onion plant samples

Oxyfluorfen was applied as pre-emergence herbicide, as there was no emergence of onion crop on day 0 (2 hrs), so the plant samples were could not be not collected for residue analysis. Similarly on day 5, onion plants were just emerged and there was no sufficient plant size to collect them for analysis. Hence the residue of oxyfluorfen in onion plant was determined from 10 days after its application onwards. At 10 and 30 DAHA, the higher concentration of oxyfluorfen active substance was determined in onion plant sample collected from plots, where oxyfluorfen was applied at 400 g a.i. ha⁻¹ (0.0218 µg g⁻¹) and it was followed by 300 g a.i. ha⁻¹ (0.0183 µg g⁻¹) and 250 g a.i. ha⁻¹ (0.0143 µg g⁻¹). At 15 DAHA, increase in oxyfluorfen residue concentration was observed (Table 3), thereafter it was decreased linearly. The increase of oxyfluorfen residues in plant samples on 15 DAHA when compared to 10 DAHA this was due to the increased absorption and translocation of oxyfluorfen from soil in concomitant with the establishment of onion plants from just germinated bulbs. At 30 DAHA onwards, the oxyfluorfen residues decreased and dissipated from plant. At 45 DAHA, more than 90% of the oxyfluorfen dissipated from the plant. The pattern of atrazine degradation was similar in sandy clay loam soil under maize crop (Janaki *et al.*, 2012)^[8]. Faster dissipation at initial period could be attributed to the dilution of residue concentration in onion plant by the higher growth rate and biomass. Residues of oxyfluorfen at varying doses applied in soil and residue content in plant samples were well below the prescribed maximum residue limits (0.05 µg g⁻¹) at the time of harvest. At harvest the level of pendimethalin, fluchloralin and oxadiazone residue applied as pre-emergence at 1.0-0.5 kg ha⁻¹ in onion bulbs ranged from 0.003 to 0.021, 0.004 to 0.036 and 0.080 to 0.104 µg g⁻¹, respectively. Marginal increase in the residue was observed with increased FYM application (Raj *et al.*, 1999)^[13].

Bioassay study of oxyfluorfen herbicide in succeeding crops

Chemical weed control method are most ideal, practical, effective, up-to-date, time saving and economical means of reducing early weed competition and crop production losses (Ashiq *et al.*, 2007)^[11]. But the exclusive dependence on herbicides has resulted in pollution of the environment and some weed species becoming resistant and inter and intra-specific shifts, integrating the chemical with cultural is an excellent option for the weed control (Hassan and Marwat, 2001)^[6]. Bioassay study results revealed that germination of succeeding sunflower and pearl millet recorded at 10 days after sowing (DAS) was not significantly affected by residual effect of oxyfluorfen herbicide applied to irrigated onion. Though, the plant stand of sunflower ranged from 85 to 90%

and pearl millet from 88 to 92% under all the treatments at 10 DAS. Further, plant height and dry weight of plants recorded at 30, 60 and 90 DAS were also unaffected due to residual effect of different doses of oxyfluorfen applied to onion. Seed yield of sunflower and grain yield of pearl millet showed no distinct variation due to different dose of oxyfluorfen applied plots. This result is in line with the outcome findings of Jayakumar (2010) [9] where the pre-emergence application of oxyfluorfen in potato at higher doses of 300 and 400 g a.i. ha⁻¹ did not leave any residue in the soil and there was no toxic effect beyond 60 days. It might be the experiment evidently showed that the application of oxyfluorfen with different doses could be very effective against most of the broad leaved and grassy weeds in onion crop. Finally, these results showed that the residual toxicity of oxyfluorfen cannot be ruled out on did not have any effect on the succeeding crops such as sunflower and pearl millet in rotation (Table 4).

Conclusion

Phytotoxicity symptoms in onion plant was visualized even at 21 DAHA, with a rating of 1 (slight stunting, injury or discoloration) and the plants were did not completely recovered from phytotoxicity symptoms. Complete recovery of affected plants could be observed only after 30 DAHA and the phytotoxicity was not evident thereafter in onion crop. Persistence of oxyfluorfen under field study clearly showed that the residues of oxyfluorfen herbicide dissipated faster in onion plant than in soil. The results indicated, with a pH of the field soil of at 6.9 and an organic matter content of 0.54%, the oxyfluorfen residues dissipated with a half-life of 11.2 in

soil and 6.1 days in soil and plant, respectively. Oxyfluorfen herbicide is highly bound to the soil organic carbon and has low mobility in red sandy clay loam soil which indicates the binding of this herbicide to soil particles is higher. Residues of oxyfluorfen at varying doses applied in soil and residue content in plant samples were well below the prescribed maximum residue limits (0.05 µg g⁻¹) at the time of harvest. Bioassay study results showed that the new formulation of oxyfluorfen at 150, 200, 250, 300 and 400 g ha⁻¹ and oxyfluorfen (goal) at 200 g a.i. ha⁻¹ applied in onion was found to be safe on the succeeding crops and this might be due to detoxification of herbicides in soil and do not adversely affect the growth and yield of the succeeding crops in terms of plant height, dry matter production and yield of the succeeding sunflower and pearl millet crops. Hence, it is concluded that pre-emergence application of oxyfluorfen at 200 g a.i. ha⁻¹ can be safely applied for weed control in onion without any residual toxicity. However, the impact of continuous and inappropriate application of oxyfluorfen in red sandy clay loam soil needs to be investigated to assess its risk potential to non-target organisms.

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Table 2: Residue of oxyfluorfen (23.5% EC) in onion field soil - mean values of two seasons

Treatments	Residue of oxyfluorfen in onion soil (µg g ⁻¹)				
	0 DAHA	10 DAHA	30 DAHA	45 DAHA	60 DAHA
Control	0.0000	0.0000	0.0000	0.0000	0.0000
T ₁ - PE oxyfluorfen (Goal) at 200 g a.i. ha ⁻¹	0.0629	0.0154	0.0088	0.0012	0.0000
T ₂ - PE oxyfluorfen at 150 g a.i. ha ⁻¹	0.1079	0.0625	0.0137	0.0033	0.0000
T ₃ - PE oxyfluorfen at 200 g a.i. ha ⁻¹	0.1295	0.0716	0.0165	0.0044	0.0000
T ₄ - PE oxyfluorfen at 250 g a.i. ha ⁻¹	0.1403	0.1286	0.0193	0.0048	0.0000
T ₅ - PE oxyfluorfen at 300 g a.i. ha ⁻¹	0.1608	0.1946	0.0229	0.0064	0.0000
T ₆ - PE oxyfluorfen at 400 g a.i. ha ⁻¹	0.2275	0.1984	0.0281	0.0092	0.0000

Table 3: Residue of oxyfluorfen (23.5% EC) in onion plant samples - mean values of two seasons

Treatments	Residue of oxyfluorfen in onion plant samples (µg g ⁻¹)			
	10 DAHA	15 DAHA	30 DAHA	45 DAHA
Control	0.0000	0.0000	0.0000	0.0000
T ₁ - PE oxyfluorfen (Goal) at 200 g a.i. ha ⁻¹	0.0010	0.0000	0.0000	0.0000
T ₂ - PE oxyfluorfen at 150 g a.i. ha ⁻¹	0.0035	0.0000	0.0000	0.0000
T ₃ - PE oxyfluorfen at 200 g a.i. ha ⁻¹	0.0111	0.0124	0.0041	0.0000
T ₄ - PE oxyfluorfen at 250 g a.i. ha ⁻¹	0.0143	0.0156	0.0051	0.0000
T ₅ - PE oxyfluorfen at 300 g a.i. ha ⁻¹	0.0183	0.0197	0.0079	0.0000
T ₆ - PE oxyfluorfen at 400 g a.i. ha ⁻¹	0.0218	0.0222	0.0115	0.0000

Data not statistically analyzed; PE - Pre-emergence; DAHA - Days After Herbicide Application

Table 4: Residual effect of oxyfluorfen on the germination %, dry matter production and yield of succeeding crops - mean values of two seasons

Treatments	Sunflower			Pearl millet		
	Germ. (%) at 10 DAS	DMP (kg/ha) at 60 DAS	Seed yield (kg/ha)	Germ. (%) at 10 DAS	DMP (kg/ha) at 60 DAS	Grain yield (kg/ha)
Control	68.5 (86.6)	1056	851	71.4 (90.3)	2630	688
T ₁ - PE oxyfluorfen (Goal) at 200 g a.i. ha ⁻¹	67.9 (85.9)	1078	784	71.9 (90.4)	2545	627
T ₂ - PE oxyfluorfen at 150 g a.i. ha ⁻¹	68.4 (86.5)	1045	871	73.1 (91.5)	2719	661
T ₃ - PE oxyfluorfen at 200 g a.i. ha ⁻¹	69.8 (88.1)	1158	897	73.2 (91.6)	2736	701
T ₄ - PE oxyfluorfen at 250 g a.i. ha ⁻¹	69.7 (88.0)	1134	881	73.6 (92.0)	2867	709
T ₅ - PE oxyfluorfen at 300 g a.i. ha ⁻¹	68.1 (86.1)	1057	805	71.5 (89.9)	2710	658
T ₆ - PE oxyfluorfen at 400 g a.i. ha ⁻¹	68.3 (86.6)	1061	845	71.7 (90.1)	2641	685

Figures in parenthesis are arc sin transformed values; Germ. % - Germination percentage; PE - Pre-emergence; DAS - Days after sow

References

1. Ashiq NM, Noor A. Comparative efficacy of different herbicides against broadleaf weeds in Wheat. Pak. J. Weed Sci. Res. 2007; 13(3-4):149-158.
2. Banga RS, Yadav A, Malik RS, Malik RK. Evaluation of different herbicides for weed control in Pea (*Pisum sativum* L.). Indian J. Weed Sci. 1998; 30(3-4):145-148.
3. Channappagoudar BB, Biradar NR. Physiological studies on weed control efficiency in direct sown onion. Kar. J. Agric. Sci. 2007; 20(2):375-376.
4. Das AC, Debnath A, Mukherjee D. Effect of the herbicides oxadiazon and oxyfluorfen on phosphates solubilizing microorganisms and their persistence in rice fields. Chemosphere. 2003; 53:217-221.
5. Daugovish O, Fennimore SA, Mochizuki M. Integration of oxyfluorfen in strawberry (*Fragaria X ananassa*) weed management programs. Weed Technol. 2008; 22:685-690.
6. Hassan G, Marwat KB. Integrated weed management in agricultural crops. Proceeding National Workshop on Technologies for Sustainable Agriculture, Sep. 24-26, NIAB, Faisalabad, Pakistan, 2001, 27-34.
7. Janaki P, Chinnusamy C. Determination of metamifop residues in soil under direct-seeded rice. Toxicol. Environ. Chem. 2012; 94(6):1043-1052.
8. Janaki P, Meena P, Chinnusamy C, Murali Arthanari P, Nalini K. Field persistence of repeated use of Atrazine in sandy clay loam soil under maize. Madras Agric. J. 2012; 99(7-9):533-537.
9. Jayakumar B. Efficiency evaluation of oxyfluorfen on weed control in tea and its residual effect on succeeding crop, M.Sc. Thesis, Tamil Nadu Agricultural University, Coimbatore, Tamil Nadu, India. 2010.
10. Jayakumar R. Bioassy for detection certain herbicide residue in soils. Madras Agric. J. 1987; 74:267-272.
11. Mueller TC, Senseman SA. Methods related to herbicide dissipation or degradation under field or laboratory condition. Weed Sci. Special Issue: 2015, 133-139.
12. Prakash V, Pandey AK, Singh RD, Mani VP. Integrated weed management in winter onion (*Allium cepa* L.) under mid-hills conditions of North-Western Himalayas. Indian J. Agron. 2000; 45:816-821.
13. Raj MF, Patel BK, Shah PG, Barevadia TN. Pendimethalin, fluchloralin an oxadiazon residue in onion. Pestic. Res. J. 1999; 11(1):68-70.
14. Sathy Priya Ramalingam, Chinnusamy Chinnagounder, Manickasundaram Perumal, Murali Arthanari Palanisamy. Evaluation of new formulation of oxyfluorfen (23.5% EC) for weed control efficacy and bulb yield in onion. American Journal of Plant Science. 2013; 4: 890-895
15. Smith R, Fennimore SA, Orloff S, Poole GJ. IPM-Pest management Guidelines: onion and Garlic University of California, Agriculture and Natural Resources Publication. 2008, 3453.
16. Sondhia S. Persistence of oxyfluorfen in soil and detection of its residues in rice crop, Toxicol. Environ. Chem. 2009; 91:425-433.
17. Sondhia S. Persistence and bioaccumulation of oxyfluorfen residues in onion, Environ. Monitor. Assessm. 2010; 162(1-4):163-168.
18. Tesfay Amare. Effect of weed management methods on weed and wheat (*Triticum aestivum* L.) yield. Afr. J. Agric. Res. 2014; 9(24):1914-1920.
19. USEPA - US Environmental Protection Agency. Pesticide environmental fate one linear summaries: Oxyfluorfen. Environmental fate and effects division, Washington, DC, 1992, 10-111.
20. Walkley A, Black IA. An examination of the Degkjareff method for determining the soil organic matter and proposed modification of the chromic acid titration method. Soil Sci. 1934; 37:29-38.
21. Waughope RD, Buttler TM, Hornsby AG, Augustijn-Beckers PWM, Burt JP. SCS/ARS/CES pesticide properties database for environmental decision making. Rev. Environ. Contamin. Toxicol. 1992; 123:1-157.
22. Weed Science Society of America (WSSA). Herbicide handbook, 7th ed. Champaign, IL. 1994; 10-59.