



## Leaching potential of oxyfluorfen in soil

Shishir Tandon\*

G.B. Pant University of Agriculture & Technology, Pantnagar, Uttarakhand 263 145

Email: shishir\_tandon@lycos.com

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### ABSTRACT

Leaching potential of oxyfluorfen herbicide was evaluated in clay loam soil under laboratory conditions with simulated rainfall. Oxyfluorfen was applied at 240 and 480 g/ha on 60 cm long soil columns. Oxyfluorfen leached upto 10 cm soil depth but, maximum concentration (84.5%) of the total herbicide applied was observed on upper top soil. Residues were not detected in leachates. Study indicates low mobility of oxyfluorfen in clay loam soils under saturated moisture conditions and may not pose ground water contamination problem if not used indiscriminately.

Leaching and runoff are the major transportation processes responsible for water contamination (Singh *et al.* 2017). The presence of pesticide residues in water is a major concern worldwide, as water is a source of drinking and irrigation in most of the countries (Tandon 2017). Oxyfluorfen [4-[2-chloro-4-(trifluoromethyl)phenoxy]-2-ethoxy-1-nitrobenzene] is a diphenyl ether herbicide with selective pre-emergence and post-emergence activity, used primarily to control broadleaf weeds and some grasses and used in various agricultural, plantation, medicinal and horticultural crops. Oxyfluorfen has potential to affect aquatic ecological systems and is toxic to aquatic plants, invertebrates and fishes. Oxyfluorfen is classified as a possible human carcinogen (EPA 2002).

Leaching of pesticides is considered as one of the main cause for ground water contamination (Tandon *et al.* 2016, Tandon 2018). Literatures on oxyfluorfen under Indian conditions are fewer (Janki *et al.* 2013, Sondhia 2008). Therefore pesticide-leaching studies, are necessary to determine their potential to contaminate ground water. Keeping in view of above facts leaching behaviour of oxyfluorfen was studied under simulated rainfall in laboratory conditions to estimate the extent of leaching of the herbicide in soil and predicting the extent of pollution it can cause to ground water.

Soil samples of different depth, *i.e.* 0-15 cm, 15-30 cm, 30-45 cm, and 45-60 cm from the N.E.B. Crop Research Centre, G.B. Pant University of

Agriculture and Technology, Pantnagar, Uttarakhand, India was collected, air dried and passed through a 2mm sieve and analysed for its physio-chemical properties.

Chemicals used during the course of this study were all of Analytical and HPLC grade. Triple distilled water was prepared in laboratory by using quartz distillation unit. All the glasswares used in the present study were of Borosil and Corning make. The technical grade oxyfluorfen (95.0% pure) was obtained by courtesy of M/S Willowood Chemicals, Hongkong and commercial grade of oxyfluorfen (Goal 24 EC) was obtained by courtesy of M/S Dow Agro sciences, India for leaching experiments.

Leaching experiment was conducted in department of Chemistry (Division of Agricultural Chemicals), G. B. Pant University of Agriculture and Technology, Pantnagar at room temperature ( $28\pm 2^\circ\text{C}$ ) and arranged in completely randomized design with three replications. The leaching studies were performed in PVC (polyvinyl chloride) columns (10 cm internal diameter and 60 cm long). The column were cut longitudinally into two halves and rejoined by using packing tape. The average volume of the tubes was recorded. The one end of PVC column was covered with muslin cloth and a funnel was attached to the bottom of each column for collection of leachates into 1000 ml flasks. Individual columns were packed with 6500 g clay loam soil. Columns were filled with soil according to the different depth taken. Columns were saturated

overnight by flowing water from above and also keeping them dipped in water contained in a bucket. Excess water was drained out by 1 day drainage cycle and columns were covered with aluminum foil to prevent evaporation. For monitoring vertical movement and leaching loss of oxyfluorfen, 10 ml of oxyfluorfen formulation was applied to surface of column with pipette at recommended dose (240 g/ha) and double recommended (480 g/ha).

The addition of water was done for ten days at the rate of 45 ml for 12 hrs per day so that infiltration rate of soil would not be exceeded. A set of soil columns receiving same amount of water only served as control. Water eluting from the column was collected daily in flask and processed for analysis for herbicide. After ten days, when addition of water was completed, the soil columns were allowed to dry for 24 hours. Columns were cut into two halves and the soil was cut into 5 cm segment each and processed for residue analysis.

A 20 g representative air dried soil sample was extracted with 50 ml of dichloromethane: methanol (1:3 v/v) shaken over a orbital shaker for one hour and filtered. The procedure was repeated twice with 25 ml of solvent mixture. All filtrate were pooled and concentrated to 1 ml under vacuum at  $45 \pm 1^\circ\text{C}$ . The extract was loaded on a pre-washed solid phase C-18 solid phase extraction (SPE) cartridge and eluted by using methanol. Elute obtained was dried under vacuum and the residue was filtered through 0.22 $\mu\text{m}$  Millipore PTFE filter and dissolved into HPLC grade acetonitrile for further analysis.

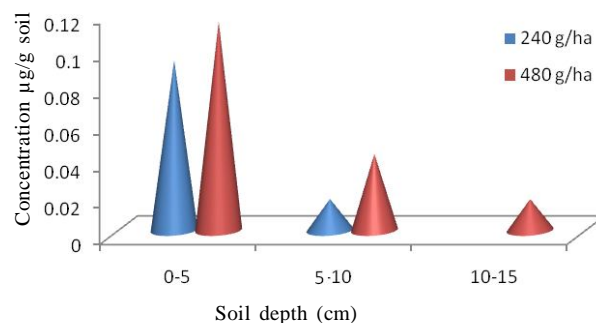
Leachate samples (25 ml) were filtered and liquid-liquid partitioned with methylene chloride (50 ml x 3), organic layer was collected, pooled and was dried over anhydrous sodium sulphate and volume was reduced to dryness in a rotary vacuum evaporator ( $40 \pm 1^\circ\text{C}$ ). The residue was dissolved in 5 ml mobile phase and filtered through 0.22 $\mu\text{m}$  Millipore PTFE filter.

The Beckman model 322 Gradient HPLC system with fixed UV wavelength detector was used for detection. The operating parameters were Supelco's ODS-II column 2.5 $\mu\text{m}$  (250mm X 4.6mm), mobile phase Acetonitrile: Methanol: Water: (6:3:1v/v) with isocratic mode at a flow rate of 1ml/min, chart speed 1 cm/min,  $\lambda$  0.02 and UV detection at 229nm. The retention time was 4.1 min.

Recovery studies were carried out by fortifying the untreated sample of soil and water at 0.5 and 1.0

mg/kg with oxyfluorfen and processed by following the methodology as described above. Average recoveries for oxyfluorfen in soil and water were 87.5-90.0% and 93.4-95.6%, respectively. The compound in the samples was identified and quantified by comparison of the retention time and peak heights of the sample chromatograms with those of standard runs under identical operating conditions. Limit of detection (LOD) was 0.003 mg/kg and limit of quantification (LOQ) was 0.01 mg/kg.

Soil used was clay loam (clay 36%: silt 40%: sand 24%) with 1.32% organic carbon, pH 7.54,  $\text{CaCO}_3$  0.482 and EC 0.177 ms. The amount of residues of oxyfluorfen in different fractions of leachates which were collected from 1 to 10 days showed that, no residues of oxyfluorfen was detected in the fractions of leachates from day 1<sup>st</sup> to 10<sup>th</sup> day at recommended and double rate of application. The distribution of herbicide residues in soil cores at different depths after passing 450 milliliters of water are presented in **Figure 1**.



**Figure 1. Leaching of oxyfluorfen at 240 g/ha and 480 g/ha in soil**

Data revealed that the surface applied oxyfluorfen got distributed only in the upper part of the column. At both application rates the residue concentration was maximum in the upper soil layers of column, mainly at the depth from 0 to 5 cm, it start decreasing from 5 to 10 cm and beyond this soil depth. Oxyfluorfen did not moved beyond 15 cm soil column indicating low mobility of oxyfluorfen in soil column. At 240 g/ha dose total herbicide retained was 89.0% while, in case of 480 g/ha dose the retention was 89.9% in soil. At 240 g/ha rate about 84.5% of the herbicide remained in 0-5 cm soil depth while, 15.5% leached to the 5-10 cm depth, whereas, in case of 480 g/ha 66.02% remained at 0-5 cm depth and 30.0% leached to lower depths.

The results are contrary with the findings of Sondhia (2008), where high movement of oxyfluorfen in clay soil was reported and residues of oxyfluorfen reached beyond 85 cm soil depth under natural rainfall condition (830 mm rainfall), and herbicide residues were also detected in leachates. According to Wauchope *et al.* (1992) oxyfluorfen is very well adsorbed to soils and herbicide-soil binding were highest in soils with high organic matter and clay content and oxyfluorfen would not leach below 4 inches in soils except sandy soils. Once oxyfluorfen is adsorbed to soil particles, it is not readily removed. Oxyfluorfen 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 (WSSA 1994).

Oxyfluorfen has high affinity to organic carbon and therefore less susceptible to leaching. In our condition the soil was rich in organic matter with high percentage of clay and silt, this might be the reason for slow movement of oxyfluorfen under saturated moisture conditions in mollisol soil. Yen *et al.* (2003) also reported similar findings of low movement of oxyfluorfen in soil. In very low organic carbon content and coarse texture soils, oxyfluorfen has potential to contaminate ground water less than 3 m deep (Ying and Williams 2000)

Results revealed that oxyfluorfen was not mobile in clay loam soil column, it did not leached beyond 15 cm depth (at double dose) that would cause significant ground water pollution. Results of the controlled laboratory studies cannot be reliably extrapolated to field conditions and field studies will be a more realistic approach for finding the real leaching potential.

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