Short communication

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Persistence of oxyfluorfen in acid soil and tea leaves

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India is the highest producer of tea in the world. Tea is grown in about 5,78,458 hectares in India. In south India, tea is grown at an altitudes ranging from 500 to 2200 meters above mean sea level in undulating terrains lying between 10 and 16 per cent slopes, even up to 33 per cent gradient (Sharma 1977). Tea is grown in a wide range of soil types that are chiefly acidic in reaction in south India (Barua 1989) and at an altitudes ranging from 500 to 2200 meters above mean sea level in undulating terrains. Weeds pose a serious problem in young tea clearing, pruned fields and vacant patches in mature tea fields. Hence, chemical weed control methods are widely followed by the farmers. Since, tea is grown in sloppy terrains, possibility of the herbicides to runoff and contaminates the water bodies and environment is more. Paraquat, 2,4-D and glyphosate are extensively used herbicides in tea fields and oxyfluorfen, glufosinate ammonium, simazine and diuron are used in a limited scale (Barooah, 2011) in India. Oxyfluorfen is used for broad spectrum pre- and post-emergence control of annual broadleaved and grassy weeds (Scrano *et al*. 1999) in a variety of crops. Wauchope *et al*. (1992) also reported an average field half life of 35 days for oxyfluorfen.

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) and parent oxyfluorfen is of toxicological concern for human health risk assessment. Although there are several reports on the efficacy of oxyfluorfen against various weeds, reports on the methods of chemical analysis and environmental fate in tea leaves and its degradation in acid soil is lacking. Therefore, field experiment was undertaken to investigate the behavior of oxyfluorfen in acid soil and the possibility of bioaccumulation in tea leaves and its hazard to environment.

Field experiment pertaining to the persistence of oxyfluorfen in tea and field soil was conducted at the farmer's field near TANTEA Research Farm, Coonoor, Nilgiris, Tamil Nadu. The experimental farm was located in Western Zone of Tamil Nadu at 11°15'00"N latitude and 76°40'00"E longitude with an altitude of 1501 m above mean sea level (MSL). Experiment was laid out in randomized block design and the treatments were replicated thrice. The size of each plot was 5 × 3 m. The spacing between the tea bushes and the rows was 100 cm. The clone variety '*CR 6017*' was used in the field experiment. Oxyfluorfen was applied at four different doses, *i.e*. 200, 250, 300 and 400 g/ha, as a pre-emergence spray in tea plantation field with the help of a knapsack sprayer. Control plot was maintained in triplicate by only spraying water, without herbicide. The treatments were imposed during September after pruning during March.

Soil samples were drawn randomly from 0–15 cm depth using a tube auger from 6–7 spots in each plot. Approximately 500 g of soil was collected from each plot. The soil and tea leaf samples were collected at 0 (2 h), 5, 10, 25, 50, 75 and 100 day time intervals from all the treated and control plots. Soil 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. Newly emerged young tea leaf samples collected (about 200 g) from different treatments were chopped to small pieces, homogenized and a representative sample of 50 g from each treatment was processed for analysis. The soil of the experimental field was sandy clay loam with pH 4.30, EC 0.24 dS/m and 4.10% organic carbon.

A gas chromatography (model GC8610 of Thermo Fischer India Ltd, India) equipped with electron capture detector (GC-ECD) was used for quantitative analysis of oxyfluorfen and its extraction and determination in soil and tea leaves was done as per the protocol described by Janaki *et al*. (2013). Validation of method was also executed in terms of recovery studies before analyzing unknown samples. Oxyfluorfen degradation was described using the first

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order kinetics equation. Under the given conditions of GC-ECD, oxyfluorfen resolved at 4.90 min as a single sharp peak. While the instrument detection limit for oxyfluorfen by GC was 0.01 µg/mL, the method detection limit was found to be 0.01 and 0.05 µg/g of soil and tea leaves respectively. The average recovery of oxyfluorfen across different matrices and levels of oxyfluorfen fortification was acceptable which ranged from 75 - 93 per cent with the RSD values of 1.1 to 3.3.

On day 0, initial deposit of oxyfluorfen was determined 0.078, 0.136, 0.247 and 0.372 mg/kg of soil at 200, 250, 300 and 400 g/ha respectively (Table 1). At all the rates of application, more than 50% oxyfluorfen was dissipated from soil within 10 days. On day 25, while the residue was not detected in the lowest dose applied plot, more than 90% residue was degraded from the 250 g/ha applied plot. This could be the result of high soil binding which is the likely route of its dissipation from soil (Wauchope *et al*. 1992) as influenced by the high organic carbon content. This could also be ascribed to the washing out of residues from soil through runoff from soil (Janaki *et al*. 2012) by the high rainfall (>50 mm) received during that period. At rates of 300 and 400 g/ha, oxyfluorfen dissipated slowly from soil, and the dissipation rate was found to be 87.9 and 87.8% respectively on day 25. More than 95% of the applied oxyfluorfen dissipated from soil at the higher rates of 300 and 400 g/ha application on day 50 and its residue was BDL on 75th and 90th days. The rate of disappearance of oxyfluorfen in soil followed first-order kinetics with the R^2 value ranged of 0.91-0.98. The half life of oxyfluorfen in soil ranged between 5.7 to 6.4 days (Table 1). Such a shorter half life of oxyfluorfen found in the present study might be due to the enhanced photolysis of oxyfluorfen by high temperature and sun shine hours prevailed during the time of herbicide application. The low solubility of oxyfluorfen in water (0.116 mg/L at 20^{0}C) might have retained it in soil surface and could have augmented the faster photo chemical decomposition from soil as reported for metamifop (Janaki *et al*. 2012).

As there was no growth of new leaves on 0 and 5 day, tea leaf samples were not collected and analyzed for oxyfluorfen residue. On day 10, the oxyfluorfen concentration detected was as 0.045, 0.066, 0.091 and 0.16 mg/kg of leaves at 200, 250, 300 and 400 g/ha respectively (Table 2). This could be ascribed to the increased absorption and translocation of oxyfluorfen from soil in concomitant with the new growth of leaves from the tea plant. On day 25, the oxyfluorfen residue decreased and on day 50, it becomes BDL at lower doses (200 and 250 g/ha) applied plot. Whereas at higher doses (300 and 400 g/ha) applied plot, approximately 70 per cent of the resi-

Table 1. Persistence of oxyfluorfen in tea field soil

Days after herbicide application	Persistence (mg/kg) ± SD[a]			
	200 g/ha	250 g/ha	300 g/ha	400 g/ha
0	0.078 ± 0.004	0.136± 0.005	0.247± 0.015	0.372± 0.007
5	0.049± 0.007	0.062± 0.019	0.108± 0.001	0.184± 0.005
10	0.023± 0.007	0.46± 0.002	0.075± 0.004	0.125± 0.003
25	BDL	0.010± 0.002	0.029± 0.006	0.045± 0.004
50	BDL	BDL	0.011± 0.006	0.019± 0.004
75	BDL	BDL	BDL	0.007± 0.003
90	BDL	BDL	BDL	BDL
Half life	5.66	6.36	5.80	6.35

[a]Average of three replicates, SD = Standard deviation, BDL = Below detection level

Table 2. Persistence of oxyfluorfen in tea leaves

Days after herbicide application	Persistence (mg / kg) ± SD[a]			
	200 g / ha	250 g / ha	300 g / ha	400 g / ha
0	BDL	BDL	BDL	BDL
5	BDL	BDL	BDL	BDL
10	0.045± 0.008 (0)	0.066± 0.002 (0)	0.091± 0.003 (0)	0.166± 0.013(0)
25	0.028± 0.002 (39.1)	0.039± 0.002 (41.6)	0.051± 0.009 (43.8)	0.081± 0.006 (51.4)
50	BDL	BDL	0.019± 0.001 (78.7)	0.027± 0.002(83.8)
75	BDL	BDL	BDL	BDL
Half life	19.55	18.22	19.35	18.00

[a]Average of three replicates, SD = Standard deviation, BDL = Below detection level, figures in parentheses indicate % dissipation

due only dissipated on 50 day. Detectable residues (>0.01 mg/kg) was not found on day 75 of application at any of the dose which is well below the proposed oxyfluorfen tolerance of 0.2, 0.01 and 0.005 mg/kg respectively by India, Japan and European Union for tea (Barooah 2011). The half life of oxyfluorfen in plant was ranged between 18.0 to 19.6 days at the four rates of application (Table 2). Based on the dissipation rate of oxyfluorfen from the tea leaves, the safe waiting period suggested for plucking the tea leaves from the plant after oxyfluorfen spraying is 75 days.

SUMMARY

The present study was undertaken to determine the persistence and residue of oxyfluorfen in tea and sandy clay loam acid soil with a pH of 4.3 and organic carbon content of 4.1%. Results showed that, the application of oxyfluorfen at 200 g /ha persisted in soil up to 10 days while up to 25 days at 400 g/ha and 50 per cent degraded from the soil before 30 days. Within 3 hrs of application, 6.9 to 35.8% of the oxyfluorfen dissipated from the soil. More than 80% of the applied oxyfluorfen degraded from soil before 10 days of its application under 200 g/ha, while it took 25 days at 400 g/ha. Oxyfluorfen residue was not detected in tea leaves upto 50 days. It is concluded that the waiting period of 75 days should be implemented for plucking the tea leaves after the application of oxyfluorfen or else there will be a chances for the herbicide to enter the food chain due to bioma-gnification.

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