

Degradation Studies of Fenazaquin in Soil under Field and Laboratory Conditions

Anil Duhan · Beena Kumari

Received: 22 November 2010 / Accepted: 17 May 2011 / Published online: 12 June 2011
© Springer Science+Business Media, LLC 2011

Abstract Degradation of fenazaquin in sandy loam soil was investigated under field and laboratory conditions. Fenazaquin (Magister 10EC) was applied @ 125 and 250 g a.i./ha in field and in pot under field capacity moisture in laboratory. Samples drawn periodically were analyzed on GC-NPD. The residues of fenazaquin in both the doses and conditions dissipated almost 90% in 90 days. Half-life period were 32.04 and 31.35 days at two doses, respectively at field conditions and 30.10 and 28.94 days at laboratory conditions. Dissipation was approximated to first order kinetics in both conditions having correlation coefficient ranging from -0.9848 to -0.9914 .

Keywords Fenazaquin · Residues · Degradation · Field and laboratory conditions · Half-life

The soil burden resulting from the repeated long-term applications of pesticide chemicals necessary for protection of plant crops is of special concern as soil serves as an ultimate sink for pesticides whether applied directly or received indirectly from spray drift and crop residues of treated plants. Such treatments may suppress soil microflora and hence affect soil properties. A proportion of the pesticide applied eventually becomes incorporated into the soil. Dispersion of pesticides and their transformation products within the soil environment, or from the soil to

other environments, is influenced by the properties of pesticides like splitting of the pesticide molecule by chemicals, photochemical, or biological processes and soil. Whereas transfer processes are characterized by the pesticide molecule remaining intact. Pesticides may have potential to bind to soil, the extent of which depends greatly on the nature of the chemical used. Therefore it has been observed that the physico-chemical nature of the soil is important for the persistence, metabolism and binding of pesticides in soil. They include sorption to soil constituents, movement of the soil in runoff, movement into the air by volatilization, movement downward in the soil by leaching, and movement upward in the soil through capillary forces. The main processes potentially affecting the ultimate fate of pesticides in soil are retention by soil materials (involving adsorption/desorption processes), transformation processes (biological and chemical degradation), and transport (through soil, atmosphere, surface water, or ground water) (Saltzman and Yaron 1986; van der Hoff and van Zoonen 1999).

Fenazaquin 4-[[4(1,1-dimethylethyl) phenyl] ethoxy] quinazoline, is a low dose emerging acaricide used to control mites attack in different vegetables. It has been found very effective insecticide for the control of insects/mites pests of okra crop (Anonymous 2007). Perusal of literature revealed that not much information is available on degradation studies of fenazaquin in soil under field and laboratory conditions. This experiment was thus carried out to generate information on the persistence and dissipation of fenazaquin in sandy loam soil at different doses under two conditions.

Materials and Methods

For field experiment, fenazaquin (Magister 10EC) @ single (125 g a.i. ha⁻¹) and double dose (250 g a.i. ha⁻¹) was

A. Duhan
Department of Chemistry and Physics, CCS Haryana
Agricultural University, Hisar 125004, Haryana, India

B. Kumari (✉)
Department of Entomology, CCS Haryana Agricultural
University, Hisar 125004, Haryana, India
e-mail: beena@hau.ernet.in

Table 1 Physical properties of soil

Soil type	Sand (%)	Silt (%)	Clay (%)	pH	EC (dSm ⁻¹)	O.C.	N (kg ha ⁻¹)
Sandy loam	28	24.3	42.0	7.85	0.21	90	110

applied in the field on the okra crop with Knap Sap sprayer in plots of size 3 × 3 m size along with a control plot where no pesticide was applied in a randomized design (RBD). Physico-chemical characteristics of the soil (Table 1) were determined by standard methods. Soil samples were drawn in triplicate from top 15 cm of soil profile on 0 (1 h after treatments), 3, 7, 15, 30, 60 and 90 days of treatments from 6–8 different places from each plot in polythene bags and brought to the laboratory for further analysis. The field samples were air dried under shade, ground and passed through 2 mm sieve. Representative 15 g soil was taken as an analytical sample after quartering and processed for residue content.

For laboratory experiment, sandy loam soil was collected from the untreated plots of research farm of CCS Haryana Agricultural University- Hisar, Haryana, India. Soil after drying was sieved through 2 mm sieve before use to remove the debris. The quantity of the acaricide required per pot (4 kg of soil) was calculated based on the soil weight of plough depth (i.e. 2 million kg soil per ha.). Soil was treated with two doses equivalent to field doses, single dose (T₁: 125 g a.i.ha⁻¹) and double dose (T₂: 250 g a.i.ha⁻¹) and packed in separate pots in triplicate for each dose and control for each treatment. The pots were kept at field capacity moisture level of 1/3 bar tension. The moisture content in pot was checked periodically (3–5 days interval) and the required amount of water was added to maintain the desired moisture content throughout the experimental period. Samples of soil from each treatment were drawn and analyzed periodically after 0 (1 h after treatment), 1, 3, 7, 15, 30, 60 and 90 days after treatment. From each pot, a thoroughly mixed soil sample was taken with steel auger and quartered to a required size (15 g) and analyzed for degradation studies.

Extraction and clean-up was carried out as per method of Kumari et al. (2008). Ground, sieved and dry representative (15 g) of soil mixed with charcoal and florisil (0.3 g each) and 10 g of anhydrous sodium sulphate. The mixture was packed compactly in a glass column (60 cm × 22 mm) in between two layers of anhydrous sodium sulphate. Residues were eluted with 100 mL of hexane:acetone (9:1 v/v) at flow rate of 2–3 mL/min. The eluate was concentrated on flash evaporator and made the final volume to 10 mL for GC analysis.

Fenazaquin residues were quantified using a gas chromatograph (GC) [Shimadzu Model 2010] equipped with a nitrogen phosphorus detector (NPD) and fitted with HP-1,

Table 2 Recovery of fenazaquin from soil

Substrate	Level of fortification (mg kg ⁻¹)	Recovery (%) (mean ± SD) ^a
Soil	0.25	90.14 ± 2.64
	0.50	92.60 ± 4.01
	1.0	94.13 ± 2.90

^a Each value is mean ± SD of three replicate determinations

capillary column (30 m × 0.32 mm × 0.25 μm film thickness) of 5% diphenyl and 95% dimethyl polysiloxane. The operating parameters were as follows: column, temperature 100°C (1 min) @ 10°C min⁻¹ to 200°C (0 min) @ 20°C min⁻¹ to 260°C (3 min); injection port 270°C; detector 275°C and carrier gas (N₂) flow 18 mL min⁻¹. The flow rate of the gas was 2 mL min⁻¹ through the column with split ratio 1: 10. The retention times observed for fenazaquin was 14.38 min. The percent recoveries of fenazaquin in soil were found to be consistent and more than 80% (Table 2). Therefore, the results are presented as such without applying any correction factor. Limit of detection was 0.001 and limit of determination/quantitation 0.005 mg kg⁻¹.

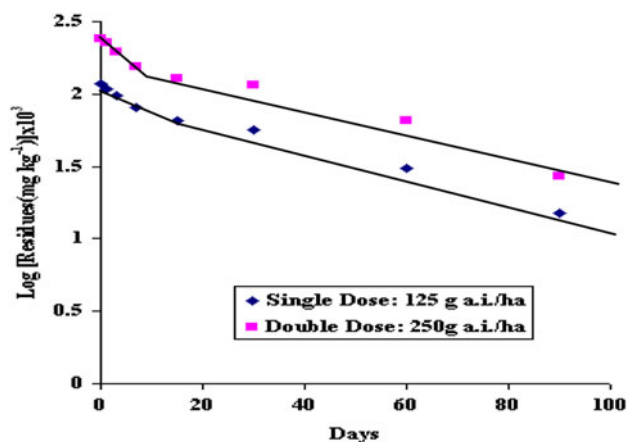
Results and Discussion

Residue data and percent dissipation of fenazaquin in soil under field conditions at two treatments are given in Table 3. The average initial deposits estimated in soil were found to be 0.119 and 0.242 mg kg⁻¹, respectively, following application of fenazaquin (magister 10EC) @ 125 and 250 g a.i./ha. Three days after application, these residue levels dissipated to an extent of about 17 and 19%, at single and double the application rate, respectively (Fig. 1). In 90 days, residues dissipated to the extent of 87 and 89% at the respective doses. Half-life (t_{1/2}) of fenazaquin calculated as per Hoskins (1961) was observed to be 32.04 and 31.35 days, respectively, when applied @ 125 and 250 g a.i./ha. Statistically analyzed data showed that with increase in duration, significant reduction (CD = 0.006; *p* = 0.05) in residues level was observed. Double dose showed significantly more residues (0.161 mg kg⁻¹) at the end of study period as compared to single dose (0.080 mg kg⁻¹) (CD = 0.003; *p* = 0.05). Maximum residues (0.180 mg kg⁻¹) were at the initiation of the

Table 3 Persistence of fenazaquin residues* in soil under field conditions

Days after treatment	Average residues (mg kg ⁻¹) ± SD				
	Single dose (125 g a.i. ha ⁻¹)	Dissipation (%)	Double dose (250 g a.i. ha ⁻¹)	Dissipation (%)	Mean
0(1 h)	0.119 ± 0.006	–	0.242 ± 0.004	–	0.180
1	0.109 ± 0.003	8.40	0.225 ± 0.005	6.25	0.167
3	0.098 ± 0.001	17.64	0.194 ± 0.003	19.16	0.146
7	0.080 ± 0.003	32.77	0.156 ± 0.004	35.00	0.118
15	0.066 ± 0.004	44.53	0.129 ± 0.004	46.25	0.098
30	0.057 ± 0.002	52.10	0.116 ± 0.002	51.56	0.086
60	0.031 ± 0.001	73.94	0.065 ± 0.003	72.94	0.047
90	0.015 ± 0.004	87.39	0.027 ± 0.005	88.84	0.021
Mean	0.080		0.161		
	Correlation coefficient $r = -0.9914$		Correlation coefficient $r = -0.9861$		
	Regression equation $y = 2.0197 - 0.0094x$		Regression equation $y = 2.3262 - 0.0096x$		
	$t_{1/2} = 32.04$ days		$t_{1/2} = 31.35$ days		

* Average of three replicates; CD ($p = 0.05$) days = 0.006; dose = 0.003; days x dose = 0.008

**Fig. 1** Dissipation kinetics of fenazaquin in soil under field conditions

experiment (0 day) which significantly ($CD = 0.006$; $p = 0.05$) declined to 0.167, 0.146, 0.118, 0.098, 0.086, 0.047 and 0.021 mg kg⁻¹ after 1, 3, 7, 15, 30, 60 and 90 days after treatment. At each duration, significant reduction in residues was observed as compared to other durations. Interaction between dose and duration was found to be non-significant ($CD = 0.008$; $p = 0.05$) which suggested a significantly less residues at each duration of lower dose (125 g a.i. ha⁻¹) as compared to higher dose (250 g a.i. ha⁻¹). The dissipation followed a first order kinetics (Fig. 1).

The residue data under laboratory conditions from two treatments given in Table 4 (Fig. 2), revealed that at single dose, average initial deposits on 0 (1 h after treatment) were 0.059 mg kg⁻¹ which dissipated to 0.035 mg kg⁻¹ in 7 days. The residues further declined to 0.029 mg kg⁻¹ in

15 days, 0.020 mg kg⁻¹ in 30 days and 0.014 mg kg⁻¹ in 60 days with 49.15, 62.71 and 76.27% dissipation, respectively during this period. The residues dissipated by 88.13% leaving 0.007 mg kg⁻¹ residues after 90 days of treatment.

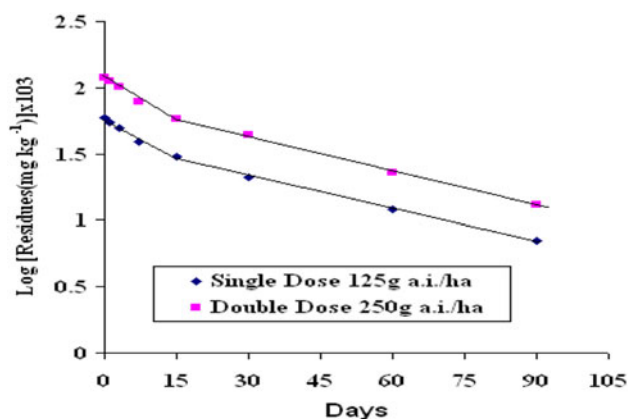
At double dose, the initial residues were found to be 0.120 mg kg⁻¹ on 0 day (1 h after treatment). After 1 day of treatment, the residues declined to 0.112 mg kg⁻¹ indicating per cent dissipation of 6.66. The residues further dissipated to 63.33, 80.83 and 89.16% in 30, 60 and 90 days after treatment with 0.044, 0.023 and 0.013 mg kg⁻¹ residues, respectively in the soil. Statistically analyzed data showed that with increase in duration, significant reduction in residue levels was observed. Double dose showed significantly more residues (0.069 mg kg⁻¹) at the end of study period as compared to single dose (0.034 mg kg⁻¹) ($CD = 0.003$; $p = 0.05$). Maximum residues (0.091 mg kg⁻¹) were at the initiation of the experiment (0 day) which significantly ($CD = 0.006$; $p = 0.05$) declined to 0.083, 0.076, 0.059, 0.044, 0.032, 0.019 and 0.010 mg kg⁻¹ after 1, 3, 7, 15, 30, 60 and 90 days after treatment. At each duration, significant reduction in residues was observed as compared to other durations. Interaction between doses and duration was found to be non-significant ($CD = 0.009$; $p = 0.05$) which suggested significantly less residues at each duration of lower dose (125 g a.i. ha⁻¹) as compared to higher dose (250 g a.i. ha⁻¹).

It was observed that dissipation after 90 days was slightly faster under laboratory conditions i.e. 88.13 and 89.16% as compared to field conditions where per cent dissipation was 87.39 and 88.84% in single and double doses, respectively. Dissipation followed a biphasic first order kinetics for field as well as laboratory experiments.

Table 4 Persistence of fenazaquin residues* in soil under laboratory conditions

Days after treatment	Average residues (mg kg ⁻¹) ± SD				
	Single dose (125 g a.i. ha ⁻¹)	Dissipation (%)	Double dose (250 g a.i. ha ⁻¹)	Dissipation (%)	Mean
0(1 h)	0.059 ± 0.005	–	0.120 ± 0.004	–	0.091
1	0.056 ± 0.003	6.77	0.112 ± 0.009	6.66	0.083
3	0.047 ± 0.003	15.25	0.101 ± 0.004	15.83	0.076
7	0.035 ± 0.001	33.89	0.079 ± 0.008	34.16	0.059
15	0.029 ± 0.004	49.15	0.058 ± 0.001	51.66	0.044
30	0.020 ± 0.003	62.71	0.044 ± 0.003	63.33	0.032
60	0.014 ± 0.002	76.27	0.023 ± 0.003	80.83	0.019
90	0.007 ± 0.004	88.13	0.013 ± 0.003	89.16	0.010
Mean	0.034		0.069		
	Correlation coefficient $r = -0.9848$		Correlation coefficient $r = -0.9865$		
	Regression equation $y = 1.6992 - 0.010x$		Regression equation $y = 2.0081 - 0.0104x$		
	$t_{1/2} = 30.10$ days		$t_{1/2} = 28.94$ days		

* Average of three replicates; CD ($p = 0.05$) days = 0.006; dose = 0.003; days x dose = 0.009

**Fig. 2** Dissipation kinetics of fenazaquin in soil under laboratory conditions

There is only one report (Sharma et al. 2006) on persistence behaviour of fenazaquin in which contrary to present results, complete dissipation of residues of fenazaquin applied @ 100 and 200 g a.i./ha from tree basin soil under apple tree was observed in 30 days. Kumar et al. (2004) reported faster dissipation of fenazaquin in tea in wet season than in dry season which corroborate the present results as under laboratory conditions slightly faster dissipation was observed, may be due to high moisture

level than field conditions. These studies, therefore, suggests that the use of fenazaquin at the recommended and double the recommended dosages can be considered quite safe from the point of view of soil health hazards as the residues left in soil at the tested doses in 90 days studies were not very high.

Acknowledgment The authors wish to express their gratitude to the Head, Department of Entomology for providing research facilities.

References

- Anonymous (2007) [www.moa.gov.cylmoa/de/de.nlf/Ail/Ac8E23Ad70BE3F012257480076501D/\\$file/fenazaquin](http://www.moa.gov.cylmoa/de/de.nlf/Ail/Ac8E23Ad70BE3F012257480076501D/$file/fenazaquin)
- Hoskins WM (1961) Mathematical treatment of loss of pesticide residues. *Pl Prot Bull FAO* 9:163–168
- Kumar V, Dhananjay KT, Srigiripuram DR, Adarsh S (2004) Investigation in tea on fate of fenazaquin residue and its transfer in brew. *Food Chem Toxicol* 42(3):423–428
- Kumari B, Madan VK, Kathpal TS (2008) Status of insecticide contamination of soil and water in Haryana, India. *Environ Monit Assess* 136:239–244
- Saltzman S, Yaron B (1986) *Pesticides in soils*. Van Nostrand Reinhold, New York
- Sharma D, Dubey JK, Patyal SK (2006) Persistence of fenazaquin in apple fruits and soil. *Pesticide Res J* 18(1):79–81
- van der Hoff GR, van Zoonen P (1999) Trace analysis of pesticides by gas chromatography. *J Chromatogr A* 843:301–322