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Effect of Household Processing on Fenazaquin Residues in Okra Fruits

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Abstract Fenazaquin (4-[[4 (1,1-dimethylethyl) phenyl] ethoxy]quinazoline) is a new acaricide of the quinazoline class. Residue levels of fenazaquin were determined in unprocessed and processed okra fruits to evaluate the effect of different processes (washing, boiling and washing followed by boiling) in reduction of residues of this pesticide in okra. The study was carried out on okra crop (Variety, Varsha Uphar) in research farm of Chaudhary Charan Singh Haryana Agricultural University, Hisar with application of fenazaquin (Magister 10 EC) @ 125 ga.i./ha (Single Dose, T_1) and 250 g a.i./ha (Double Dose, T_2). Samples of okra fruits were collected on 0, 3, 7, 15 days after treatment and at harvest (30 days). Residues were estimated by gas chromatograph equipped with capillary column and nitrogen phosphorus detector. Residues reached below maximum residue limit of 0.01 mg/kg at harvest. The residues dissipated with half-life period of 3.13 days at lower dose and 4.43 days at higher dose. Processing is shown to be very effective in reducing the levels of fenazaquin residues in okra fruits. Maximum reduction (60–61%) was observed by washing + boiling followed by boiling/cooking (38–40%) and then by washing (31–32%).

Keywords Fenazaquin · Residues · Household processing · Okra · Half-life period

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Vegetables are an essential component of human diet. Besides providing a variety and bulk to the diet, they are good source of vitamins that are essential for human health (Gupta et al. 1988). India has favorable agro-climatic conditions for the cultivation of large number of horticultural crops like fruits and vegetables that is why India is the second largest producer of vegetables after China. Total production of vegetables has been estimated as 71.58 million tons from 5.12 million hectares. In near future, there is a need of around 5–6 million tones of vegetables to feed over 1.3 billion Indian population expected by the year 2020 (Paroda 1999; Dhandapani et al. 2003). Indian vegetables export is very low because of increased domestic requirement and other limitations in crops production. The major limiting factor, include the extensive crop devastation due to increased pest menace, which cause an average of 40% loss in different crops (Srinivasan 1993). Okra (*Abelmoschus esculantus* L.) is the major economical important vegetable, largely affected by parasitoids, general predators like coccinellids, syrphids, spiders, staphylinids, predatory mites and anthocorid bugs which are also highly prone to pesticide. Mites not only result in crop losses but also adversely affect the quality as their infestation results in discoloration of leaves (Gupta 2003). Fenazaquin (4-[[4(1,1-dimethylethyl) phenyl]ethoxy] quinazoline) has excellent contact activity against tetranychid and eriophid mites both in laboratory and field tests. This compound is non-phytotoxic and its activity is independent of temperature with in the range 12.6–30°C (Dreikorn et al. 1991; Shanker et al. 2001). It acts as an electron transport inhibitor, acting at complex I of the mitochondrial respiratory chain (Hollingworth et al. 1992). Use of pesticides by farmers is the only way to sort out the problem of insects/pests and many a time they harvest the crop without observing any waiting period. As a result, considerable quantities of these pesticides that are absorbed by vegetables, reach the human body and results in many health hazards. Considering the fact that on the processing of vegetables, the residues level decreases. The present study was conducted to know the effect of different household processing (i.e. washing, boiling/cooking and washing followed by boiling/cooking (washing + boiling)) on residues level in okra fruit has been carried out.

Materials and Methods

The study was carried out on okra crop (Variety, Varsha Uphar) in the research farm of Chaudhary Charan Singh (CCS) Haryana Agricultural University, Hisar with application of Magister 10 EC (Fenazaquin) @ 50 g a.i./acre or 125 g a.i./ha (T_1) and 100 g a.i./acre or 250 g a.i./ha (T_2) on fruit bearing okra plants. The trial was laid in a randomized block design; each treatment was replicated thrice along with control where no pesticide was applied. The treated samples were allowed to remain as such under field conditions and 0 day sample was taken after 1 h of spray. Further sampling was done 3, 7, 15 days after treatment and at harvest (30 days) from each treatment. These samples were used to study its persistence and effect of different commonly used household processing, viz. washing (under tap water for 30 s), boiling/cooking (till softness) and washing followed by boiling or washing + cooking. The samples were extracted, cleaned and estimated as per the method of (Kumari et al. 2001) with slight modifications.

Representative 20 g of the finely chopped sample was extracted with 150 mL acetone and hexane mixture (1:1v/v) and partitioned with ethyl acetate after diluting with 10% NaCl solution made in double distilled water. The organic layer was concentrated on rotary vacuum evaporator to reduce the volume approximately to 10 mL. Glass column (60 cm × 22 mm i.d.) was packed compactly with activated charcoal and activated Florisil (1:5w/w). Pre-wet the column with 10 mL of hexane, loaded the concentrated extract in the column and eluted with 125 mL of hexane :ethyl acetate mixture (1:1v/v). Concentrated the eluate on vacuum evaporator followed by gas manifold evaporator. Final volume was made to 2 mL in *n*-hexane for GC estimation.

The final extracts were analyzed on Shimadzu 2010 gas chromatograph (GLC) equipped with capillary column, HP-I (10 m × 0.53 mm 10, 2.65 μm) film thickness of methyl silicone and nitrogen phosphorous detector (NPD). The operating parameters of the instrument were: injection port 270°C, column 260°C and detector 300°C, carrier gas N_2 1. 8 mL min^{-1}, H_2, 1.5 mL min^{-1} and zero air 130 mL min^{-1}. Under these operating conditions the retention time of fenazaquin was found to be 5.666 min.

Table 1 Recovery of fenazaquin from okra fruits

Substrate	Level of fortification (mg/kg)	% Recovery[a]
Okra	0.10	94.8 ± 7.6
	0.20	100.7 ± 3.8

[a] Average of three replicates; Results and Discussion

The control samples of okra fruits were spiked at 0.1 and 0.2 mg/kg, and processed by following the methodology as described above. The results revealed that per cent mean ± SD recoveries for okra samples at both these levels were 94.8 ± 7.6 and 100.7 ± 3.8, respectively (Table 1).

Result and Discussion

The results have been reported as such without applying any correction factors. The minimum limits of determination for fenazaquin in okra fruits were found to be 0.01 mg/kg.

The average initial deposits of fenazaquin at single and double dose were observed to be 0.770 and 1.070 mg/kg, respectively. Residues on 30th day of application reached below detection limit (BDL) as well as maximum residue limit (MRL) of 0.010 mg/kg (Anonymous 2007) in single dose and equal to MRL in double dose. The residues dissipated with half-life period of 3.13 days at lower dose and 4.43 days at higher dose following first order kinetics. It was interesting to note that the residues of fenazaquin dissipated initially up to 3 days to the extent of 23 and 20% at single and double dosages, respectively. However on 7th day, residues declined suddenly to the extent of 60 and 55% at lower and higher doses, respectively. At the end of 30 days, the residues reached BDL of 0.10 mg/kg showing thereby 100% dissipation in single dose and equal to 0.10 mg/kg with 99% dissipation in double dose. Kumar et al. (2006) studied the fate of fenazaquin and its transfer to brew and found that residue dissipated faster in wet season than in the dry season. Half life period in green shoots was suggested between 1.43–1.70 and 2.10–2.21 days in single and double dose, respectively. Degradation rate followed first order kinetics. Sharma et al. (2006) in a field trial on apple fruits revealed that residue levels of fenazaquin 10EC applied @ 100 and 200 g a.i./ha reached to non detectable level on 30th day at two locations. Half-life period ranged between 1.9–5.3 days at lower dose and 3.6–5.2 days at higher dose (Table 2).

Effect of Processing

Okra fruits were subjected to processing like washing, boiling/cooking and washing followed by boiling in order to investigate the reduction of residues. It has been found that washing followed by boiling was found to be more effective followed by boiling and washing in reducing the residues. In this process, residues reduced by 61.03% in single dose and 60.00% in double dose, respectively on 0 day (Tables 3, 4). Thereafter, reduction of residues was 42.56, 23.84 and 18.33% on 3rd, 7th and 15th days after application in single dose whereas at double dose percent reduction on respective days were 40.00, 20.82 and 16.09. In boiling/cooking process, per cent reduction of residues on 0, 3, 7 and 15 days after treatment were 40.26, 25.67, 17.20 and 10.83 at lower dose and 37.94, 23.39, 16.08 and 9.07 at higher dose. However by washing, residues were reduced in the range of 9.16–32.46% at lower and 7.80–30.93% at higher dose. From the results it has been concluded that all the three processes used in this study were more effective in reducing the residues on 0 day because of the deposition of residues on surface which could be dislodged easily. On the passage of time residues penetrate in to fruit and less reduction was observed on successive days. Kumar et al. (2006) reported considerable loss (42–70%) of

Table 2 Residues ($\mu g g^{-1}$) of fenazaquin in okra fruits

Days after treatment	Single dose (125 g a.i./ha)		Double dose (250 g a.i./ha)	
	Average ± SD	% Dissipation	Average ± SD	% Dissipation
0	0.770 ± 0.05	–	1.070 ± 0.27	–
3	0.592 ± 0.08	23.11	0.855 ± 0.24	20.09
7	0.302 ± 0.04	60.77	0.485 ± 0.04	54.67
15	0.120 ± 0.01	84.41	0.205 ± 0.29	80.84
30	BDL	100	0.010 ± 0.02	99.06
	Correlation coefficient $r = -0.9752$		Correlation coefficient $r = -0.9890$	
	Regression equation $y = 3.1004 - 0.09607x$		Regression equation $y = 3.139 - 0.060794x$	
	$t_{1/2} = 3.13$ days		$t_{1/2} = 4.43$ days	

Average ± SD of three replicates, BDL: 0.010 mg/kg

Table 3 Effect of processing on reduction of fenazaquin residues in okra at single dose

Days after treatment	Single dose (125 g a.i./ha)			
	Average residues ($\mu g g^{-1}$)[a]	Washing	Boiling	Washing + boiling
0	0.770	0.520 (32.46)	0.460 (40.26)	0.300 (61.03)
3	0.592	0.480 (18.91)	0.440 (25.67)	0.320 (42.56)
7	0.302	0.260 (13.90)	0.250 (17.20)	0.230 (23.84)
15	0.120	0.109 (9.16)	0.107 (10.83)	0.098 (18.33)

[a] Average ± SD of three replicates; Figs in parenthesis is percent reduction of residues

Table 4 Effect of processing on reduction of fenazaquin residues in okra at double dose

Days after treatment	Double dose (250 g a.i./ha)			
	Average residues ($\mu g g^{-1}$)[a]	Washing	Boiling	Washing + boiling
0	1.070	0.739 (30.93)	0.664 (37.94)	0.642 (60.00)
3	0.855	0.710 (16.95)	0.655 (23.39)	0.513 (40.00)
7	0.485	0.425 (12.37)	0.407 (16.08)	0.384 (20.82)
15	0.205	0.189 (7.80)	0.186 (9.07)	0.172 (16.09)

[a] Average ± SD of three replicates; Figs. in parenthesis is per cent reduction of residues

fenazaquin residues during processing of green shoots to made tea. Holland et al. (1994) reported appreciably reduction in pesticide residues in different commodities by using different processing methods. Hence the present results are in consistent with the earlier reports.

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