Journal of Crop and Weed 8(1): 190-192 (2012)

Fate and persistence of herbicide quizalofop-p-tefuryl on black gram

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Received: 29.02.2012, Revised: 16.04.2012, Accepted : 06.06.2012

Key words: Herbicide, HPLC, quizalofop-p-tefuryl, persistence, residue

Quizalofop-p-tefuryl 4.41% EC (Pantera) is having selective post-emergence herbicidal effect on annual and perennial grasses in potato, soybean, sugar beet, peanut, oilseed rape, sunflower, vegetable crops, etc. Quizalofop-p-tefuryl (belonging to aryloxyphenoxypropionate) acts as systemic herbicide, absorbed from the leaf surface and translocates throughout the plant. It works as an acetyl CoA carboxylase inhibitor. Black gram is small sized pulse having black seed coat that is used in the preparation of fermented foods mainly in the south Indian recipes and other regional foods. It belongs to the family leguminosae and sub family papilionaceae. It is a good source of phosphoric acid, proteins, carbohydrates and calcium (Anon., 2011b). The production of black gram is mostly confined to the tropical climatic areas of Asian countries where soil type suit the pulse cultivation. India is the largest producer of this pulse followed by Myanmar and Thailand (Anon., 2011a).

Pesticides, increase crop productivity, reduce cost of production, improve quality and thus help in the farmers' income. One of the major disadvantages of pesticide use is the presence of residue in foodstuff though seldom exceed the maximum residue limits (MRLs) set by the food authorities. For this reason proper monitoring of pesticide residues is very important for reducing health hazard to consumers. In this respect, the present study, a field trial of quizalofop-p-tefuryl in black gram was conducted at Experimental Research Farm of Bidhan Chandra Krishi Viswavidyalaya, Nadia, West Bengal in July – October, 2008 to understand the residue and persistence behaviour of herbicide quizalofop-p-tefuryl in black gram (both plant and soil).

Pesticide standard and 4.41% EC quizalofop-p-tefuryl (Pantera) formulation were used for the purpose. All solvents used were of HPLC grade and the chemicals were of analytical grade. High performance liquid chromatograph (Varian ProStar) with variable wavelength detector connected to auto-sampler was used for residue analysis. An agilent ZORBAX eclipse XDB- C 18, 5 µm, 4.6×150 mm was employed as analytical column. Methanol and water (0.1% acetic acid) in a ratio of 8 :2 was used as mobile phase in a HPLC analysis. The wave lenghth (λ_{max}) was maintained at 240 nm during the HPLC analysis (Hao *et al.*, 2006).

The formulation 4.41% EC quizalofop-p-tefuryl (Pantera) was applied plot to plot by knapsack sprayer with flat pan nozzle @ 40g a.i. ha^{-1} (T_1), @ 80g in a Randomized Block Design. Three replications were used for each treatment. Soil samples were collected at an interval of 0,1,5,10,15 , 30 days and at harvest from plot. Five soil cores were randomly taken from each plot from 0-15 cm depth using a soil auger. The cores were bulked together from each plot, air dried, powdered and passed through a 3 mm sieve to achieve uniform mixing. Samples from the controlled plots were collected similarly. Plant samples of 250 g from each plot were taken similarly. A composite sample was collected also from the untreated control. A representative (20 g) of plant and soil sample were prepared by quartering technique in the laboratory and taken for final analysis. All the samples were extracted immediately after collection.

A representative laboratory plant sample (20 g) from each plot was homogenized in a mixture for 2-3 min. Then 5 gm homogenized sample was accurately weighed into a 50 ml centrifuge tube and mixed with 10 ml of methanol and water (0.1% Acetic acid) with the ratio of 8:2. The mixture was vortexed for 2 min and allowed to stand for 1 hr. Afterwards 50 mg of activated charcoal black and 200 mg of anhydrous Na_2SO_4 was added to it. The tube was again vortexed for 2 min and centrifuged for 10 min at 5000 rpm. Then 2 ml supernatant liquid was collected and transferred to another centrifuge tube. The collected fraction was then filtered through a syringe filter and analyzed in HPLC equipped with variable wavelength detector. Soil (5 g) sample was taken in a 50 ml centrifuge tube and similar method as mentioned in plant sample was followed. At harvest black gram (plant, seed and soil) samples were collected and the same extraction method as mentioned above was followed.

Short communication

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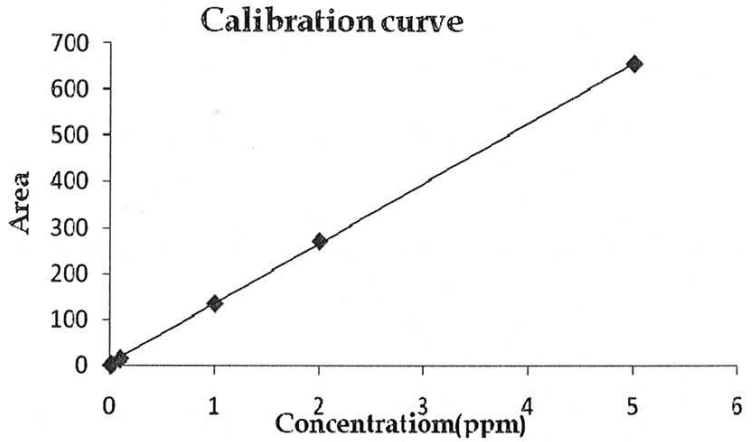

Fig. 1: Calibration curve of areas corresponding to different concentration of analytical standard of quizalofop-p-tefuryl

Table 1: Recovery study from different samples with standard solution prepared from quizalofop-p-tefuryl formulation (Mean of three replicates)

Substrate	No of Replication	Amount fortified (mg kg^{-1})	Amount recovered (mg kg^{-1})	Recovery (%)	Average recovery (%)	Average recovery for each substrate (%)
Black gram plant	1	0.05	0.04	80		
	2	0.05	0.04	84	80	
	3	0.05	0.04	76		
	1	0.20	0.17	85		
	2	0.20	0.19	95	85	85.33
	3	0.20	0.15	75		
	1	1.00	0.91	91		
	2	1.00	0.95	95	91	
	3	1.00	0.87	87		
Field soil	1	0.05	0.04	70		
	2	0.05	0.05	90	80	
	3	0.05	0.04	80		
	1	0.20	0.18	90		
	2	0.20	0.17	85	90	89.00
	3	0.20	0.19	95		
	1	1.00	0.97	97		
	2	1.00	0.95	95	97	
	3	1.00	0.99	99		
Black gram seed	1	0.05	0.04	80		
	2	0.05	0.04	78	82	
	3	0.05	0.04	82		
	1	0.20	0.17	85		
	2	0.20	0.18	90	80	86.33
	3	0.20	0.16	80		
	1	1.00	0.94	94		
	2	1.00	0.96	96	94	
	3	1.00	0.92	92		

Standard calibration curve of quizalofop-p-tefuryl was constructed by plotting concentration against peak area (Fig.1). Good linearity with R^2 value of 0.99 was achieved, limit of detection (LOD) and limit of quantification (LOQ) considered when signal to noise ratio of 3:1 and 10:1 respectively. LOD and LOQ were determined as 0.01 mg kg^{-1} and 0.05 mg kg^{-1}, respectively. In order to establish the reliability of analytical method and to know the efficiency of extraction and cleanup steps employed for the present investigation, black gram (plant, seed and soil) samples were spiked separately in replicate with 0.05, 0.2 and 1 mg kg^{-1} analytical standard of quizalofop-p-tefuryl and the average recoveries (85-90 %) were found satisfactory (Table 1). The mean residue, standard deviation, regression equation and half life values of quizalofop-p-tefuryl (Pantera) formulation at different days intervals in black gram samples (soil and plant) are presented in table - 2 and 3. From the results it is revealed that after the initial concentrations of quizalofop-p-tefuryl dissipated with increment of time and it followed first order kinetics irrespective of dose.

Table 2: Persistence of quizalofop-p-tefuryl - 4.41% EC in soil

Days	Residue in mg kg^{-1} (± SD)	
	T_1 (40 g a.i. ha^{-1})	T_2 (80 g a.i. ha^{-1})
0	0.95±0.07	2.30±0.08
1	0.76±0.02	1.71±0.05
5	0.61±0.06	1.26±0.01
10	0.16±0.01	0.39±0.08
15	BDL	BDL
30	BDL	BDL
Regression equation and half life	**$Y = 3.30037 - 0.0736 X$ $T_{1/2}$= 4.10 days**	**$Y = 3.3622 - 0.0728 X$ $T_{1/2}$= 4.14days**

Table 3: Persistence of quizalofop-p-tefuryl - 4.41% EC in plant

DAYS	Residue in mg kg^{-1} (± SD)	
	T_1 (40 g a.i. ha^{-1})	T_2 (80 g a.i. ha^{-1})
0	0.41±0.01	1.41±.06
1	0.09±0.02	0.48±.01
5	BDL	BDL
10	BDL	BDL
15	BDL	BDL
30	BDL	BDL
Regression equation and half life	**$Y = 2.6128 - 0.6427 X$ $T_{1/2}$= 0.47 days**	**$Y = 3.1492 - 0.4680 X$ $T_{1/2}$= 0.64days**

Note: BDL below detection limit, *SD*- standard deviation

No interfering peak was detected in the untreated control sample. Initial concentrations of quizalofop-p-tefuryl residues in soil and plant samples were found to ranges 0.95-2.3 and 0.41-1.41 mg kg^{-1}, respectively. It was found from the results that the half life value was 4.1-4.14 days and 0.47-0.64 days for soil and plant, respectively. The dissipation patterns of quizalofop-p-tefuryl of the present study are in well agreement with the earlier studies conducted in bean under Chinese climatic condition (Hao *et al.*, 2007). After 5th day of application, near about 50% of the residue was degraded in soil irrespective of treatments and no residue was found after 5 days of application in plant. No residues were detected in the control samples throughout the study. In our study it was observed that residues were below the detectable limit in all the harvest samples (soil, plant and seed) irrespective of treatments. No residues were detected in the control samples also. The MRL value of this compound set by European Food Safety Authority was 0.05 mg kg^{-1} (Anon., 2008). Hence, the tested doses can be considered safe from point of view of hazards.

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