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PERSISTENCE OF QUINALPHOS IN COTTON SEED, LINT AND SOIL UNDER SUB TROPICAL CONDITIONS OF PUNJAB, INDIA

P.K. ARORA[1], KOUSIK MANDAL[2], SARABJIT KAUR[2], R.S. BATTU[2] AND BALWINDER SINGH[2]

[1]PAU Regional Research Station, Abohar – 152 116; Email: parshotamarora@pau.edu

ABSTRACT

Single application of quinalphos at flowering stage was done on cotton @ 500 g a.i. ha^{-1} and 1000 g a.h. ha^{-1} and the residues were estimated in the cotton seed, lint and soil separately using gas liquid chromatography equipped with nitrogen–phosphorus detector (NPD). The interval between insecticidal application and 1st pick was 60 days. The residues of quinalphos at both the dosages on cotton seed, lint and soil were found to be below determination limit of 0.01 mg kg^{-1}.

Key words: Cotton seed, Lint, Quinalphos, Residues, Soil

Cotton (*Gossypium hirsutum* L) is an important commercial fibre crop of India and plays a vital role in agricultural and industrial economy. It is widely cultivated throughout the sub–tropical parts of north India, occupies an area of 607 thousand hectares in Punjab with an annual production of 2678 thousands tones bales per annum. The crop is adversely effected in production from year to year, mainly because of the losses caused by insect pests, of which, Pink bollworm [*Pectinophora gossypiella* (Saunders)], Spotted bollworm [*Earias insulana* (Boisdual) and *E. vittella* (Fabricius)] and American bollworm [*Helicoverpa armigera* (Hubner)] are the most serious. Ekalux/GAIC Quinalphos/Quinguard 25 EC (quinalphos) @ 2 kg ha^{-1} is recommended for the control of bollworm complex on cotton (Anonymous, 2008). However, considerable concern is being expressed by various agencies over the magnitude of pest control chemicals left in food stuffs following their use while raising the crop. Therefore, it is important to ensure that the levels of harvest time residues of quinalphos in lint and cotton seed do not pose any hazard to consumers and are admissible in National and International trade. The field trials were conducted to study the persistence of quinalphos in the cotton seed, lint an soil under sub–tropical conditions of Punjab, India.

MATERIALS AND METHODS

Cotton (var. LH 900) was raised during *kharif* 2008 at P.A.U. Regional Research Station, Abohar, Punjab, according to recommended agronomic practices.

Standard compound quinalphos was obtained from Dr. Ehrenstorfer–Schafers. Bgm.–Schlosser–Str. 6 A.D. 86 199. Augsbur, Germany. Quinalphos 25 EC formulation used for application was obtained from local market. Analysis of acetone extract of the formulation showed only quinalphos and none of its metabolic product was found to interfere with respect to its active ingredient.

The following reagents and chemicals *viz.* Sodium chloride (E. Merck (India) Limited, Mumbai, Sodium sulfate–anhydrous (S.D. Fine Chemicals) and Charcoal decolorizing powder activated (Qualigence Fine Chemicals), Solvents–acetone, dichloromethane, hexane, acetonitrile, methanol (Qualigence Fine Chemicals were used). All common solvents were redistilled in all–glass apparatus before use. The suitability of the solvents and other chemicals was ensured by running reagent blanks before actual analysis. Gas liquid chromatography (GLC) (Perkin Elmer) equipped with nitrogen phosphorus detector (NPD) and a capillary column Elite–5, 30 m long, 0.25 mm i.d. and 0.25 µm film thickness was used for estimation of quinalphos residue.

Cotton field was divided into nine plots, each measuring 250 m^2. Three replications of control, recommended dosage and double the recommended dosages were maintained. Control was treated with water spray only. The spray fluids used were @ 500 L ha^{-1}. Single foliar application was made at flowering stage using knapsack sprayer fitted with hollow cone nozzle for recommended dose @ 500 g a.i. ha^{-1} and double the recommended dose @ 1000 g a.i. ha^{-1}.

Samples of cotton and soil were collected from each treatment at harvest (PHI=60 days). Samples of cotton/ soil were collected from 5–6 randomly selected spots of each treatment. About 1 kg of cotton and soil sample was collected randomly at harvest from each replicated plot. The cotton samples so collected were air dried and delinted

to get cotton seed and lint to analyze them separately. A representative 5 g sample of cotton lint and 10 g sample of cotton seed were processed immediately and the rest were stored in deep freezer at –20°C. A similar treatment was also given to the soils from each samples.

Extraction and Cleanup. Cotton seed: Representative sample (10 g) of cotton seed was ground along with anhydrous sodium sulfate in a pestle mortar to make a free flowing powder and extracted by Soxhlet apparatus using about 400 mL mixture of hexane + acetone (1+1, VIV). The solvent was concentrated to about near dryness and the resulting fat was dissolved into 50 mL hexane and partitioned into acetonitrile saturated with hexane (3 × 50 mL). The combined acetonitrile fractions were diluted with 600 mL brine solution in 1 L separatory funnel and partitioned the contents three times into 100, 50 and 50 mL dichloromethane. Dichloromethane fractions were dried over anhydrous sodium sulfate and treated with 500 mg activated charcoal powder for about 2–3 hours at room temperature. The clear extract so obtained was filtered through Whatman filter paper No. 1, concentrated to near dryness and again added about 20 mL acetone and concentrated using rotary vacuum evaporator at 30°C. The process was repeated to completely evaporate dichloromethane and the final volume was reconstituted to 5 mL using acetone.

Cotton lint: Representative 5g sample of lint was placed in 150 mL dichloromethane for 24 hours. Dichloromethane extract so obtained was filtered through Whatman filter paper No. 1, concentrated to near dryness under rotary vacuum and residues were dissolved in 5 mL using acetone.

Soil: Representative sample (50 g) of soil was extracted by using about 150 ml of methanol + water (2 + 1, v/v) mixture. The extract was filtered into 1 L separatory funnel was diluted with 600 mL brine solution and contents partitioned three times into 100, 50 and 50 mL dichloromethane. Dichloromethane fractions were dried over anhydrous sodium sulfate and treated with 500 mg activated charcoal powder for about 2–3 hours at room temperature. The clear extract so obtained was filtered through Whatman filter paper No. 1, concentrated to near dryness and again about 20 mL acetone was added and concentrated using rotary vacuum evaporator at 30°C. Process was repeated to completely evaporate dichloromethane and the final volume was reconstituted to 5 mL using acetone.

Estimation of Quinalphos. The residues of quinalphos were estimated on gas liquid chromatography (GLC) (Perkin Elmer) equipped with nitrogen phosphorus detector (NPD). A capillary column Elite–5 (30 m × 0.25 mm i.d. × 0.25 µm film thickness) was used for estimation quinalphos insecticides. GC operating parameters were as follows: Carrier gas flow rate: nitrogen flow rate: 30.0 mL min^{-1}, hydrogen flow rate: 3.0 mL min^{-1} and air flow rate: 145.0 mL min^{-1}, temperature: injection port: 280°C, detector: 310°C. The column temperature was initially maintained at 170°C for 5 min, then increased at the rate of 10°C min^{-1}, to 220°C, kept hold for 3 min and was finally increased at the rate of 5°C min^{-1} to 240°C and kept hold for 3 min. Under these operating conditions the retention time were found to be 7.610 minutes. The residues were estimated by comparison of retention time of insecticides and their peak heights/ peak area with respect to retention time and peak heights/ peak area of reference standards analyzed under identical conditions.

Recovery studies. The per cent recovery of quinalphos in cotton lint, cotton seed and soil were found to be consistent and more than 80 per cent (Table 1).

Table 1. Recovery studies of quinalphos on the cottonseed, lint and soil

Substrate	Level of fortification (mg kg^{-1})	*Recovery (%)
Cotton seed	0.40	85.70 ± 3.23
	0.80	85.52 ± 2.73
Cotton lint	0.20	84.95 ± 2.25
	0.50	85.24 ± 3.88
Soil	0.05	85.05 ± 2.89
	0.10	91.70 ± 2.62

* Value (mean) ± standard deviation of three replicates

RESULTS AND DISCUSSION

The samples of cotton seed, lint and soil were collected and analysed at harvest from the treatments of quinalphos 25 EC @ 50 and 100 g a.i. ha^{-1}. The interval between application and the harvest of the crop was of 60 days. The residues of quinalphos on cotton seed, lint and soil were found to be less than its respective limit of quantification (LOQ) i.e., 0.01 mg kg^{-1}.

The results are in agreement with those of Kumar and Regupathy (1999) who determined the residues of quinalphos (Ekalux 20 AF and 25 EC) in cotton seed and lint. The residues in lint samples from plots treated with the highest dose @ 4.5 L ha^{-1} of AF or 4 L ha^{-1} of EC varied from below detectable level (BDL) to 0.06 mg kg^{-1} in the first picking and 0.02 to 0.09 mg kg^{-1} in the third picking. Except in one seed sample from plots treated with highest dose of quinalphos, the residues were BDL in all the samples collected at first and third pickings. In the same manner, Battu *et al.* (2003) studied the residues of different insecticides in cotton seed and lint revealed higher residues as compared to cotton seed. None of the synthetic

pyrethroids *viz.* alphamethrin, cypermethrin, fenvalerate and deltamethrin, and organophosphorus insecticides *i.e.* monocrotophos, quinalphos, fenitrothion, triazophos and ethion showed presence of residues in cotton seed. Residues of cypermethrin, fenvalerate, endosulfan, ethion and chlorpyriphos were detected in lint. Endosulfan residues (0.44 mg kg^{-1}) detected in cotton seed were below its MRL of 1.0 mg kg^{-1}. However, chlorpyriphos residues in cotton seed were more than its MRL of 0.05 mg kg^{-1}.

The residues of chlorpyriphos were 0.045 and 0.063 mg kg^{-1} in lint and 0.014 and 0.069 mg kg^{-1} in seed from the two dosages, respectively. Similarly, Singh *et al.* (2001) studied the residues of ethion (OP compound) in cotton seed and lint following six applications at 10 days interval. The residues of ethion were detected only in lint samples collected at first pick with average levels of 0.18, 0.30 and 0.62 mg kg^{-1} following its application @ 400, 800 and 1600 g a.i. ha^{-1}, respectively. The residues of the insecticide in cotton seed samples remained below levels of 0.08 mg kg^{-1}.

Manimegalai *et al.* (1994) found that the residues of pyraclofos (Voltage 50 EC) in cotton seed, lint and oil 10 days after the eighth spray at four dosages @ 375, 500, 625 and 750 g a.i. ha^{-1} were quite low and hence the insecticide can be safely used on cotton crop. Battu *et al.* (1999) studied the residues of b–cyfluthrin (Bulldock 025 EC) at 7–10 days inverval were made on cotton @ 12.50 and 18.75 g a.i. ha^{-1} and the residues were estimated in cotton seed and lint using GLC equipped with ^{63}Ni ECD. The interval between last spray and harvest was 10 days. The residues of b–cyfluthrin were detected only in cotton lint samples with average values of 0.30 and 0.40 mg kg^{-1} following its application at the above two dosages, respectively. Cotton seed samples did not reveal the presence of residues of b–cyfluthrin at the minimum detectability limit of 0.01 mg kg^{-1}. Dikshit *et al.* (2006) studied the residues of thiacloprid on cotton seed, lint and soil. Thiacloprid was sprayed @ 30, 120 and 240 g a.i. ha^{-1} at the square bud to fruiting stage of the cotton crop. The fluid rate was kept as 500 L ha^{-1}. Residues of thiacloprid were non–detectable in cotton seed, lint and soil samples at harvest. The treatments did not show any phytotoxicity symptoms.

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