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

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ABSTRACT

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

Key words : Cottonseed, Lint, Quinalphos, Residues, Soil

Cotton (*Gossypium hirsutum* L) an important commercial fibre crop of India is widely cultivated throughout the sub–tropical parts of north India occupying an area of 607 thousand hectares in Punjab with an annual production of 2678 thousands tones bales per annum. As the crop is subjected to high degree of instability 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 more serious pests, several insecticides have been recommended for their control. Under Punjab conditions, Ekalux/GAIC Quinalphos/ Quinguard 25 EC (quinalphos) @ 2 kg ha^{-1} is recommended for the control of bollworm complex on cotton (Anonymous, 2008). 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 cottonseed do not pose any hazard to consumers and are admissible in domestic and international trade. Thus, field trials were conducted to study the persistence of quinalphos in the cottonseed, lint and soil under sub–tropical conditions of Punjab, India.

MATERIALS AND METHODS

Insecticides: Standard compound quinalphos was obtained from Dr. Ehrenstorfer–Schafers. Bgm.–Schlosser–Str.6 A.D. 86 199. Augbur, 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.

Reagents and chemicals: The following reagents and chemicals were used:

Sodium chloride (E.Merck (India) Limited, Mumbai, Sodium sulfate–anhydrous (S.D. Fine Chemicals) and Charcoal decolorizing powder activated (Qualigene Fine Chemicals), Solvents–acetone, dichloromethane, hexane, acetonitrile, methanol (Qualigene Fine Chemicals). All common solvents were redistilled in all–glass apparatus before use. The suitability of the solvents and other chemicals was ensured by running reagents blanks before actual analysis.

Apparatus: Gas liquid chromatography (GLC) (Perkin Elmer) equipped with notrogen 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.

Field experiment: Cotton (var. LH 900) was raised during *kharif* 2008 at the Regional Research Station, PAU, Abohar, Punjab. The crop was raised according to recommended agronomic practices (Anonymous, 2008).

Application of the Insecticide: A cotton field at Regional Research Station, PAU, Abohar, was selected and divided into nine plots, each measuring 250 m^2 so that three replications of the three treatments control, recommended dosage and double the recommended dosages could be maintained. The control treatment was sprayed with water only. The spray flukds 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}.

Sampling procedure: Sample 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 cotton and soil were collected randomly at harvest from each replicated plot. The cotton samples so collected were air dried and delinted to get cottonseed and lint to analyze them separately. A representative 5 g sample of ctton lint and 10 g sample of cottonseed were processed immediately and the rest was stored in deep freezer at –20°C. A similar treatemtn was given to the soil from each sample.

Analysis for Quinalphos Residues: The sample were processed and analyzed at Pesticide Residue Analysis Laboratory, Department of Entomology, PAU, Ludhiana. The procedures followed for analysis of quinalphos residues in the seed, lint and soil of cotton are described below.

Extraction and Cleanup

Cottonseed: Representative sample (10 g) of cottonseed was ground along with anhydrous sodium sulfate in a pestle mortar to make a free flowing powder and extracted by Soxhlet apparatus usingabout 400 mL mixture of hexane + acetone (1+1, v/v). 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 x 50 mL). The combined acetonitrile fractions were diluted with 600 mL brine solution in 1 L separatory funnel and the contents partitioned three times into 100, 50 and 50 mL dichloromethane. Dicholormethane 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 to which about 20 mL acetone was added and concentrated using rotary vaccum evaporator at 30°C. The process was to conmpletely evaporate dichloromethane and the final volume was recontituted to about 5 mL using acetone.

Cotton lint: Representative 5 g 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, diluted with 600 mL brine solution and the 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 about 20 mL acetone was added to it and concentrated using rotary vacuum evaporator at 30°C. The process was repeated to completely evaporate cichloromethane and the final volume was reconstituted to about 5 mL using acetone.

Estimation of Quinalphos: Te 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 of quinalphos insecticides. GC operating parameters were as follows: Carrier gas flow rate: nitrogen flow rate: 30.0 mL min^{-1}, hidrogen 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 22°C, kept constant for 3 min and was finally incrased at the rate of 5°C min^{-1} to 240°C and again constant 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: Cotton lint, cottonseed and soil samples were spiked with quinalphos at different levels and analyzed separately as per the methodology described above. The recoveries of quinalphos in cotton lint, cottonseed and soil were found to be consistent and more than 80 per cent (Table 1); therefore, the results are presented as such without applying any correction factor.

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

*Each value is mean ± standard deviation of three replicate determinations.

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 aplication and the harvest of the crop was found to be 60 days. The residues of quinalphos on cottonseed, lint and soil were found to be less than its respective limit of quantifiation (LOQ) arrived at 0.01 mg kg^{-1} (Table 2).

Table 2. Residues (Mean ± SD) of quinalphos (mg kg^{-1}) in the cottonseed lint and soil at harvest time after the application of quinalphos 25 EC @ 500 and 1000 g.a.i. ha^{-1}

Substrate	Quinalphos @ 500 g.a.i. ha^{-1}		Quinalphos @ 1000 g.a.i. ha^{-1}	
	Replicates	Mean ± S.D.	Replicates ± S.D.	Mean
Cotton seed	BDL		BDL	
	BDL	BDL	BDL	BDL
	BDL		BDL	
Cotton lint	BDL		BDL	
	BDL	BDL	BDL	BDL
	BDL		BDL	
Soil	BDL		BDL	
	BDL	BDL	BDL	BDL
	BDL		BDL	

BDL = Below determination limit of 0.01 mg kg^{-1}.

The results of the present study confirm to 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 the highest dose of quinalphos, the residues were BDL in all the samples collected at first and third pickings. One the same lines, Battu *et al*. (2003) studied the residues of different insecticides in cottonseed and lint following their use as per recommended practices. In general, lint samples revealed higher residues as compared to its counterpart 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 cottonseed. Residues of cypermethrin, fenvalerate, endosulfan, ethion and chlorpyriphos were detected in lint. Endosulfan residues (0.44 mg kg^{-1}) detected in cottonseed 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}.

Working on another organophosphate (OP) compound, chlorpyriphos, Chinniah *et al*. (1999) estimated its residues in cotton lint and cottonseed. Chlorpyriphos @ 400 and 800 g.a.i. ha^{-1} was applied on cotton four times at 20 days interval starting from 25 days after sowing. 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) studies 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 40 EC) in cotton seed, lint and oil 10 days after the 8th spray at four dosages @ 375, 500, 625 and 750 g.a.i. ha^{-1} were quite low and hence suggested that the insecticide can be safetly used on cotton crop. On the same lines, Battu *et al*. (1999) studied the residues of ß–cyfluthrin in cottonseed and cotton lint. Six applications of ß–cyfuthrin (Buldock 025EC) at 7–10 d interval were made on cotton @ 12.50 and 18.75 g.a.i. ha^{-1} and the residues were estimated in cottonseed and lint using GLC equipped with ^{63}Ni ECD. The interval between last spray and harvest was 10 days. The residues of ß–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. cottonseed samples did not reveal the presence of residues of ß–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 cottonseed, lint and soil samples at harvest. The treatments did not show any phytotoxicity symptoms and plant compatibility was good; hence the recommended dose (120 g.a.i. ha^{-1}) of thiacloprid could be considered safe for the protection of the cotton crop.

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