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Chlorpyriphos, Quinalphos, and Lindane Residues in Sesame Seed and Oil (*Sesamum indicum* L.)

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Pesticides have become an essential component of the modern agricultural system. The environmental hazards have largely resulted owing to careless use of conventional persistent pesticides and often adoption of poor method of their application. In recent past the most extensive use of organophosphate, pyrethroids pesticides has, however, promoted adequate steps to safeguard the environment.

Sesame (*Sesamum indicum* L.) is one of the oldest rainy season oilseed crops of dry region of India. The crop, however, is highly susceptible to insect-pests and diseases (Mahadevan 1987). Several pesticides are used to control such pests in India. Chlorpyriphos, quinalphos and lindane are prominent among these. Because of the rapid multiplication of pests in sesame under the prevailing conducive environmental conditions repeated application of insecticides frequently become necessary for their effective control. The use of at least certain pesticides in sesame can result in the accumulation of their residues in seeds, oil and oil cake. Earlier studies of monitoring of pesticide residues in food concentrates showed that the samples of sesame oil cake contained HCH residues. The presence of chlorinated pesticide in oil cake could result in their biomagnification in our food chain. The chlorinated pesticides present in the oil cake reach the milk through lactating animals, and eventually into the human body (Yadava 1977, Kapoor and Kalra 1991). Though the persistent insecticides are beneficial for controlling pests for extended period, their residues in consumable parts of the crop in above MRL (Maximum Residue Limit) may be harmful to the consumers. The present study was undertaken to monitor the persistence of chlorpyriphos, quinalphos and lindane residues in sesame mature seeds and their oil.

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MATERIALS AND METHODS

Field experiments were conducted in Kharif 1995 at the Agricultural Research Station, Durgapura, Jaipur to study the residues of three insecticides chlorpyriphos, quinalphos and lindane in sesame seed and oil. The crop was raised as per the package of practices recommended by the Rajasthan Agricultural University. Identical doses of the insecticides at 500 and 1000 g ai/ha were applied twice as foliar spray. The first spray was done four wks after the germination of the crop and the second, three wks later. Sesame seeds from different treatments and replications were collected at harvest.

25 g powdered seed samples were extracted with n-hexane in soxhlet for 8 hrs. The solvent was removed in Rotary Vacuum Evaporator and the oil thus obtained was weighed. Two gram of the oil sample was dissolved in 50 ml n-hexane and partitioned with 50 ml portions of acetonitrile twice. The n-hexane layer was discarded and to the acetonitrile layer 100 ml of saturated NaCl was added and partitioned thrice with 50 ml portions of n-hexane. The n-hexane layer was dried over anhydrous sodium sulphate and concentrated to 50 ml.

The n-hexane extract was taken in a 250 ml separating funnel and 10 ml of conc. H_2SO_4 was added drop by drop and allowed to stand for sometime so that two layers could be clearly separated. The lower acid layer was removed. The upper n-hexane layer was again treated with 5 ml of conc. H_2SO_4. The sulphuric acid treatment was repeated till the lower layer became colourless. After removing the lower acid layer, the n-hexane layer was washed repeatedly with distilled water till the washing was neutral to litmus (Kapoor et al. 1981). The acid free n-hexane layer was dried over anhydrous sodium sulphate and concentrated to 50 ml.

50 ml n-hexane extract of chlorpyriphos was concentrated to 5 ml and chromatographed over a column containing 20 g silica gel sandwitched between 10 g of anhydrous sodium sulphate. The column was eluted with 150 ml of 5% ethylacetate in n-hexane. The elute was concentrated to 10 ml (Luke *et al.* 1975).

50 ml n-hexane extract of quinalphos was concentrated to 5 ml and chromatographed over a column containing activated charcoal and anhydrous sodium sulphate. The column was eluted with 100 ml of n-hexane : acetone (9:l). The elute was concentrated to 10 ml.

Residue estimation was done by gas chromatograph (Model - Tracor.565) equipped with Electron Capture Detector. The parameters used for different insecticides are given in Table-l. The validity of extraction, the clean up and the estimation procedure of lindane, chlorpyriphos and quinalphos in sesame oil were confirmed by fortifying the control samples with 0.5 to 1.0 $mgkg^{-1}$ levels of each of the above insecticide. The recovery percentages were found to be 97, 96 and 92% for lindane, chlorpyriphos and quinalphos, respectively. During the experimental period the minimum and maximum temperature varied from 18.2-25.2°C and 32.3-37.6°C, respectively. The variation in relative humidity was 40-97 per cent.

Table 1: Parameters for the estimation of lindane, chlorpyriphos and quinalphos by gas liquid chromatography.

Parameters	Insecticide		
	Lindane	Chlorpyriphos	Quinalphos
Detector	ECD	ECD	ECD
Column	1.5%OV-17 + 1.95% OV-210	3% OV-1	3% OV-101
Temperature(^{O}C)			
Detector	250	250	250
Column	210	180	200
Inj.port	225	200	210
Flow rate of carrier gas N_2 (ml/min.)	60	40	40
Retention time (min.)	1.34	4.58	3.33
Recovery (%)	97	96	92

RESULTS AND DISCUSSION

The analytical data pertaining to the residues of chlorpyriphos, quinalphos and lindane are given in Table-2. It was found that quinalphos residues did not persist either in oil or in seed of sesame at the time of the harvest at both the doses. Similar findings were also reported by scientists of Bhubneshwar,

Jabalpur and Kanpur (Biennial Progress report 1994-96). Lindane residues persisted both in seed and oil of sesame. At the lower dose of 500 g ai/ha the residues were within tolerance level while at the higher dose the residues were above in MRL (0.05 ppm) both in seed and oil. At Kanpur, the residues of lindane persisted in oil above MRL at both, the doses i.e. 500 and 1000 g ai/ha. The sesame seed and oil showed chlorpyriphos residues at both t e treatments. Sesame seed showed 0.39 and 0.56 $mgkg^{-1}$ chlorpyriphos residues at 500 and 1000 g ai/ha while the corresponding values of residues in oil were 0.95 and 1.30 $mgkg^{-1}$. These values were above MRL (0.05 ppm) of chlorpyriphos in oil seeds.

Table 2 : Average residues of lindane, chlorpyriphos, and quinaolphos in sesame seed and oil at harvest.

Insecticide	Dosages g ai/ha	Average Residues* $mgkg^{-1}$	
		Seed	Oil
Lindane	500	0.02	0.05
	1000	0.73	1.75
Chlorpyriphos	500	0.39	0.95
	1000	0.56	1.30
Quinalphos	500	BDL**	BDL
	1000	BDL	BDL

* Average of three replications
** BDL = Below detectable limit < 0.2 ng.

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