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Harvest Time Residues of Imidacloprid in Cardamom

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Studies were conducted to evaluate harvest time residues of imidacloprid 200 SL on cardamom in Tamil Nadu Agricultural University, Coimbatore during 2006. A field trial was conducted in farmers holding near Kumily, Idukki district, Kerala and three sprays of imidacloprid 200 SL at 25 and 50 g a.i. ha^{-1} were given along with untreated check. Cardamom samples were collected at random on 30 days after last spray for analysis. The results revealed that the harvest time residues of imidacloprid 200 SL at 25 and 50 g a.i. ha^{-1} were below detectable level both in green and cured cardamom capsules.

Key words: Cardamom, residues, imidacloprid, HPLC

Cardamom, *Elettaria cardamomum* (L.) Maton. the Queen of spices is indigenous to the Southern states of India. It is cultivated in Western Ghats (Kerala, Tamil Nadu and Karnataka) in an area of 73, 795 ha. with a production of 12,540 MT (2005-06) and one of the important products fetching enormous foreign exchange (Stanley, 2007). India was the world's largest producer and exporter until it was taken over by Guatemala in the 18th century. One of the major constraints in the production of cardamom is the excessive damage by pests; the major ones being *Conogethes punctiferalis* Guenee and *Sciothrips cardamomi* Ramk. in Tamil Nadu (Rao, 1977). At present, these pests are kept under check with the help of synthetic insecticides. With the strict legislations enforced by the EPA, cardamom capsules with pesticide residues have a chance of being rejected by the hitherto importing countries, which in turn would have a major say in foreign revenues. The pesticide use pattern in the present day situations has led to resistance build-up by pests and pesticide residues, which demands newer and safer pesticides with different modes of action. Thus, there is a greater need to evaluate pesticides that would leave no or lesser residues in the commodity as well as in the environment. Chloronicotinyls/ neonicotinoids are the new group of crop protection agents highly effective against sucking pests which act on receptor protein of insect nervous system. Chloronicotinyls provide excellent control of contemporary resistance pests. Extensive use of these novel compounds inevitably involves the risk of the development of new type of resistance. Keeping all these in views, the present investigation was undertaken to determine the harvest time residues of imidacloprid 200 SL in cardamom capsules.

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Materials and Methods

Field experiment was conducted to determine the harvest time residues of imidacloprid on Green gold variety cardamom during May, 2006 in the farmer's holding at Madhavanganal near Kumily, Idukki district, Kerala. The experiments were conducted with three treatments viz., T1- Untreated control, T2 – imidacloprid 200 SL @ 25 g a.i. ha^{-1} and T3 - imidacloprid 200 SL @ 50 g a.i. ha^{-1}. The crop was maintained properly by adopting standard agronomic practices recommended by Tamil Nadu Agricultural University. The treatments were imposed when the pests crossed the economic threshold level (ETL). Three sprays were given with a pneumatic knapsack sprayer with a spray fluid volume of 500 litres ha^{-1}.

Sampling

Matured and uniform sized cardamom capsules were collected at random on 30 days

after third spray with the help of forceps for residue analysis. From each plot, 150 g of green capsules was collected and from this, a sub sample of 20 g green capsules in duplicate was taken for fresh sample analysis and transferred immediately into the sample container with acetonitrile. The remaining sample of 100 g was divided into two portions and was cured under conventional curing chamber at maximum temperature of 60 - 65°C maintained for 24 h and used as cured samples for residue analysis. The weights of the samples before and after curing were recorded from each plot to workout the residues on moisture free basis and curing loss.

Extraction

The weighed sample of 25 g was soaked in acetonitrile (50 ml) overnight, homogenized and filtered through Buchner funnel. After repeated washing, the pooled acetonitrile extract was evaporated to near dryness.

Clean up

Liquid - liquid clean up

The aqueous remainder was treated with 50 ml of saturated sodium chloride and 150 ml of hexane (three 50 ml portions) in a 500 ml separating funnel. After shaking well, the lower aqueous phase was collected and 100 ml of hexane:ethyl acetate (98:2 V/V) was added and shaken well. Once again, the lower aqueous phase was collected and partitioned with three 50 ml portions of dichloromethane. The pooled dichloromethane extract was passed through anhydrous sodium sulphate. The extract was evaporated to near dryness and the aqueous remainder was dissolved in ethyl acetate.

Solid-liquid clean up

For column chromatography, 1.5 cm (id) x 50 cm (length) glass columns were used. Florisil® deactivated with 5 per cent water was used as an adsorbent at 4.5 g per sample. The drip tip of the chromatographic column was plugged with cotton wool. The Florisil® was slurried with 20 ml ethyl acetate and applied quantitatively into the column. This was sandwiched with two cm layers of anhydrous sodium sulphate. The column was prewashed with 20 ml ethyl acetate. The dry residue dissolved in small amount of ethyl acetate was poured on top of the column by means of a pipette and allowed to percolate. The active ingredient was eluted with 20 ml portions of acetonitrile (HPLC grade).

The elutant was concentrated to near dryness, the residue dissolved in acetonitrile and fed into HPLC.

Preparation of standards

The stock solution of 1000 ppm was prepared by dissolving 101 mg of imidacloprid technical material (99.0% purity) in 100 ml of acetonitrile (HPLC grade). From this stock, intermediate stock solutions of 100 and 10 ppm were prepared. Using 10 ppm stock, working standards of 0.5, 1, 2, 3, 5 and 10 ppm were prepared in HPLC grade acetonitrile.

Recovery studies

Samples were fortified with working standards at 0.1, 0.5 and 1.0 ppm level to find out the recovery of imidacloprid. The recovery obtained was 89.32 per cent.

Final quantification

End analysis was done with the aid of High Performance Liquid Chromatography (HPLC), Hitachi model L 6200 with the following operating parameters.

Mobile phase	:	Acetonitrile (HPLC grade): Water (HPLC grade) (35:65 V/V)
Column	:	ODS 2
Flow rate	:	1 ml min^{-1}
Wave length	:	270 nm
Quantity injected	:	20 µl (fixed loop)

The amount of residue was determined by comparing the sample response with the response of standard by using the formula.

$$\text{Residues in ppm} = \frac{H_s}{H_{std}} \times \frac{W_{std}}{W_s} \times \frac{V_{ex}}{V_s} \times \frac{A_s}{A_{std}}$$

where,

H_s - Peak height of the sample

H_{std} - Peak height of the standard

W_{std} - Weight of the standard injected in ng

W_s - Weight of the sample in g

V_{ex} - Volume of the final extract in ml

V_s - Quantity of the sample injected in µl

A_s - Attenuation of the sample

A_{std} - Attenuation of the standard

Results and Discussion

The mean recovery was 89.32 per cent from samples fortified at 0.1, 0.5 and 1.0 ppm level. Hence the recovery factor was not used for working out the residues. The minimum detection limit of the instrument was 0.5 ppm and the determinability level in the sample was 0.05 µg g^{-1} considering the weight of the sample as 20 g and final volume of the extract as 2 ml. The harvest time residues of imidacloprid 200 SL at 25 and 50 g a.i. ha^{-1} as foliar spray were at below detectable level (BDL) in green and cured cardamom capsules (Table 1). The interval between the last spray and sample picking was 30 days. Similar results were obtained by Stanley (2007), who reported that the residues of diafenthiuron dissipated to 0.08 and 0.16 per cent in green cardamom capsules at 15 DAT. Renuka (2001) and Rajabaskar (2003) reported a total loss of profenofos after 15 days of spray in both green and cured cardamom capsules when sprayed at 0.075 per cent. Imidacloprid residues in the cotton lint, seed and oil from first and second picking were at below detectable level (BDL) in the samples collected from the imidacloprid 200 SL treated plots, 12 and 15 days after the last spray in the first and second field trials, respectively (Preetha, 2008). Similar results were obtained by Kumar (1998) and Suganthy (2003) in cotton. In bhendi fruits also, the harvest time residues were at BDL for the samples collected during first and second harvest in two season field trials. This was in confirmation with the findings of Siva veerapandian (2000) and Suganthy (2003).

Table 1. Harvest time residues of imidacloprid 200 SL in/ on cardamom

Treatment	Dose (g a.i. ha^{-1})	Residues in µg g^{-1} at harvest	
		Green capsules	Cured capsules
T1 - Untreated check	-	BDL	BDL
T2 - Imidacloprid 200 SL	25	BDL	BDL
T3 - Imidacloprid 200 SL	50	BDL	BDL

Picking of cardamom capsules was carried out at an interval of 30 - 35 days. As harvest being the focal point for enforcement of residue tolerances, the suggested waiting periods of seven days is safe enough to contain the cardamom pests with imidacloprid without the problem of pesticide residues in harvestable produce.

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