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Short Note

Harvest Time Residues of Thiodicarb in Cardamom

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Studies were conducted to evaluate harvest time residues of thiodicarb 70 WP on cardamom in Tamil Nadu Agricultural University, Coimbatore during 2007. A field trial was conducted in farmer's holding near Kumily, Idukki district, Kerala and three sprays of thiodicarb 70 WP at 700 and 1400 g a.i. ha^{-1} were given along with untreated check. Cardamom capsule samples were collected at random on 30 days after last spray for analysis. The harvest time residues of thiodicarb 70 WP at 700 and 1400 g a.i. ha^{-1} were below detectable level both in green and cured cardamom capsules.

Key words: Cardamom, Residues, Thiodicarb, 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 71,170 ha with an annual production of 11,000 MT (Spices Board, 2009) and is one of the important products fetching enormous foreign exchange. One of the major constraints in the production of cardamom is the excessive damage caused by pests. About 72 insect pests have been recorded on cardamom. Cardamom shoot and capsule borer (CSCB), *Conogethes punctiferalis* Guenee is an economic pest that feeds on the capsules, pseudostems and panicles causing more than 10 per cent yield loss in the field (Thyagaraj, 2002). At present, this pest is kept under check with the help of synthetic insecticides. Of them, the most important organic insecticides used against pests of cardamom belong to organophosphates, carbamates and synthetic pyrethroids. The pesticide use pattern in the present day situations has led to the buildup of resistance in pests and the problem of pesticide residues, which demand 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. Thiodicarb (3, 7, 9, 13 - tetramethyl - 5, 11 -dioxa - 2, 8, 14 -trithia - 4, 7, 9, 12 -tetra-azapentadeca -3, 12-diene-6, 10-Dione) is an important carbamate insecticide that is used to control the shoot and capsule borer in cardamom.

Pesticide residues should be monitored routinely for the safety and health of the farm workers and consumers. Pesticides can remain in as dislodgeable state, be absorbed in to the cuticular layer or translocated in to the inner plant tissues. Moreover cardamom is an export oriented crop that needs to be strictly free from pesticide residues. With the strict legislations enforced by the EPA, cardamom capsules with pesticide residues may face rejection by the importing countries, which in turn would have a major say in foreign revenues. With the above background, research work was carried out to determine the harvest time residues of thiodicarb in cardamom samples.

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Materials and Methods

Field experiment was conducted to determine the harvest time residues of thiodicarb on and in 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 - Thiodicarb 70 WP @ 700 g a.i. ha^{-1} and T3 - Thiodicarb 70 WP @ 1400 g a.i. ha^{-1}. The crop was maintained properly by adopting standard agronomic practices recommended by Tamil Nadu Agricultural University. 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 50 g in duplicate was taken for fresh sample analysis and transferred immediately to the sample container with acetonitrile. The remaining sample of 100 g was divided into two portions and was cured under conventional curing chamber maintained at a maximum temperature of 60 - 65°C for 24 h and used for residue analysis of cured samples. 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 20 g was soaked in acetonitrile (100 ml) overnight, homogenized and filtered through Buchner funnel. After repeated washing, the pooled acetonitrile extract was evaporated to near dryness and the residue was taken up in the 20 ml acetonitrile.

Clean up

Partition: The concentrated extract was partitioned thrice with 100 ml hexane and the collected hexane layer was concentrated, in a 250 ml round bottom flask and evaporated to near dryness and the residue was taken by dissolving in 10 ml High Performance Liquid Chromatography (HPLC) grade hexane.

Silica gel column clean up: Fifty ml of hexane was allowed to drain through a chromatography column packed with 20 g silica gel (activated at 110 ºC for 24h) sandwiched with anhydrous sodium sulphate. The residue taken from partitioning was allowed on the silica gel column at a flow rate of one milliliter per minute. The column material was washed with 50 ml of hexane and the eluate was discarded. Then the thiodicarb residue was eluted from the column using 100 ml of hexane - ether (9:1) mixture. 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 thiodicarb 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 thiodicarb. The recovery obtained was 88.64 per cent.

Final quantification

End analysis was done with the aid of HPLC, Hitachi model L 6200 with the following operating parameters. Mobile phase : Acetonitrile (HPLC grade): Water (70:30 V/V); Column : ODS 2; Flow rate : 0.5 ml min^{-1}; Wave length : 270 nm; Quantity injected : 20 ìl (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 ìl; A_s - Attenuation of the sample; A_{std} - Attenuation of the standard

Results and Discussion

The mean recovery was 88.64 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 ìg 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 thiodicarb 70 WP at 700 and 1400 g a.i. ha^{-1} as foliar spray were below detectable level (BDL) in green and cured cardamom capsules. 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 per cent in green cardamom capsules 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. Vinothkumar *et al.,* (2009), reported 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 at 30 days after last spraying. 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 thirty days is safe enough to control the cardamom pests with thiodicarb without the problem of pesticide residues in harvestable produce.

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