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Dissipation of cartap hydrochloride in brinjal (*Solanum melongena*)

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***Abstract*—Spray application of cartap hydrochloride was given twice to brinjal crop at 15 days interval at the recommended dose and double the recommended dose of 14.4 and 28.8 g a.i./ha during rabi 2012-13 to study its residue persistence. Initial residues of cartap hydrochloride on brinjal from the two treatments were 1.11 and 2.10 mg/ kg, respectively. The residues persisted for 5 days in recommended dose @ 0.06 mg/kg and 7 days in double dose @ 0.07 mg/kg and reached below the determination level. Residues of cartap hydrochloride on brinjal dissipated with the half-life of less than 2 days and the pre-harvest interval (PHI) or the time taken for the residues on brinjal to reach the permissible level of 0.05 mg/kg was 7days. Soil samples analyzed 15 days after the last treatment was free from cartap hydrochloride residues.**

Keywords—brinjal, persistence, residues; below the determination level, pre-harvest interval

Introduction

Brinjal or egg plant (Solanum melongena) is one of the most common and popular and economically important vegetables in India. It contributes to 9% of the total vegetable production of the country (Sidhu and Dutta,2007). The average productivity of brinjal in India has been estimated to be only 130.08 q/ha. Brinjal grown throughout the year is subjected to attack by a number of insect and non-insect pests from nursery stage to harvest. Among all insect pests, the most serious and destructive is the shoot and fruit borer (Nair, 1986).In severe infestation it causes up to 70 per cent yield loss of fruit in south and southeast Asia(Srinivasan, 2009).Fruit damage as high as 92.5% and reduction in yield up to 60% due to borer have been reported by Mall et al. (1992). Although India ranks second after China in global eggplant production, crop productivity is relatively low (FAOSTAT, 2006). Cartap hydrochloride is a nereistoxin analogue group of pesticide used to control fruit and shoot borers, jassids and white flies.

MATERIALS AND METHODS

Reference standard of cartap hydrochloride (purity, 99.5%) and its formulation cartap hydrochloride SP were obtained from Hyderabad Chemical Products Limited. Standard solution of cartap hydrochloride was prepared with gradient grade acetonitrile and suitably diluted to obtain the working standards. The residue study of cartap hydrochloride on brinjal was carried out at College of Agriculture, Rajendra nagar, Hyderabad during Rabi 2012-13. The treatments were control (water sprayed), recommended dose of 500 g a.i./ha (1 g/L) and double the recommended dose of 800 g a.i./ha (1.6g/L) and replicated thrice. The spray application was given twice at the interval 10 days and - analysis of brinjal samples was carried out on 0 (2 h after application), 1, 3, 5, 7,10 and 15 days after the second spray. From each plot, approximately 600-700 g brinjal fruits were harvested, pooled together, packed in plastic bags and transported to the laboratory for processing. From each plot 1 kg soil was collected and soils from all 3 replicates were pooled together, mixed thoroughly and sub sampled. A representative 100 g sample (3replications) was taken for analysis. Extraction and clean up of the samples was carried out as per AOAC official method 2007.01 (QuEChERS) after validation of the method at the laboratory. The collected brinjal samples were homogenized with robot coupe blixer, and homogenized 15±0.1 g sample was taken in 50 ml centrifuge tube.The sample tube is then added with 30±0.1 ml acetonitrile.The sample is homogenized at 14000-15000 rpm for 2-3 min using Heidolph silent crusher. The samples then added with 3±0.1 g sodium chloride and mixed by shaking gently followed by centrifugation for 3 min at 2500-3000 rpm to separate the organic layer. The top organic layer of about 16 ml was taken into the 50 ml centrifuge tube and added with 9±0.1 g anhydrous sodium sulphate to remove the moisture content. 8 ml of extract was taken into 15 ml tube, containing 0.4±0.01 g PSA sorbent (for dispersive solid phase d-SPE cleanup) and 1.2±0.01 g anhydrous magnesium sulphate.The sample tube was vortexed for 30 sec, followed by centrifugation for 5 min at 2500-3000 rpm. The extract of about 2 ml was transfered into test tubes and evaporated to dryness using turbovap with nitrogen gas and reconstituted with 1 ml Acetonitrile for HPLC analysis under standard operational conditions.

Extraction and clean up for soil samples

The soil samples were analyzed for cartap hydrochloride residues following the QuEChERS method after validation of the method at the laboratory. 2 kg of soil was collected from each plot in Polythene bags. The soil samples were pooled, mixed well and about 200 g of the representative sample was drawn by quartering method The soil samples were dried at room temperature under shade, ground,

passed through 2 mm sieve and a representative 10 g sample was taken into 50 ml centrifuge tube.The sample tube is then added with 20±0.1 ml acetonitrile.The samples is then added with 1±0.1 g sodium chloride and 4±0.1 g Magnesium sulphate mixed by shaking gently followed by centrifugation for 3 min at 3300 rpm to separate the organic layer. The top organic layer of about 10 ml was taken into the 15 ml centrifuge tube containing 1.5±0.1 g Magnesium sulphate and 0.25 g PSA and sonicate for 1 min to remove air bubbles and centrifuge for 10 min at 3000rpm.The extract of about 2 ml (1 g sample) was transferred into test tubes and evaporated to dryness using turbovap with nitrogen gas and reconstituted 1 ml Acetonitrile for HPLC analysis under standard operational conditions.

HPLC PARAMETERS

The analysis of the samples was carried out using the following parameters: HPLC, Shimadzu, Prominence LC 20 AT with a PDA detector operated at a wavelength of 270 nm. The column used was Kinetex 5µ –C 18 column,100 mm X 4.6 mm ID. The mobile phase was acetonitrile :water (65+35, v/v), with a flowrate of 1 mL /min, injection volume was 20µl and run time 25 min. With these operating parameters the retention time of the cartap hydrochloride was at 10.44 min (Fig. 2). The recovery study cartap hydrochloride was carried out at the fortification level of 0.05, 0.25 and 0.50 mg/kg of brinjal and soil and processed as per the method described above to get the recovery percent before carrying out sample analysis. The residue data were subjected to statistical analysis according to Hoskins (1961) to compute the residual half-life (t1/2) and safe pre-harvest interval (PHI).

RESULTS AND DISCUSSION

The recovery study carried out as per the method described showed the recovery of cartap hydrochloride as 96.6% from the samples fortified at 0.05 mg/kg, while the samples fortified at 0.25 mg/kg have shown the recovery of 90.6%. The samples fortified at 0.5 mg/kg have shown the recovery of 93.3%. Hence, the limit of determination (LOQ) is 0.05 mg/kg for cartap hydrochloride in brinjal samples.

Dissipation of cartap hydrochloride on brinjal was studied over a period of 15 days. The results of the residue study are given in Table 2. Initial residue deposits of 1.11 and 2.10 mg/kg were recovered from the treatment at recommended and double the recommended dose. The residues dissipated very fast and reached below the determination limit of 0.05 mg/kg by the 7^{th} and 10^{th} day in treatment at recommended and double the recommended dose The half-life of cartap hydrochloride from treatment at 14.4 g a.i. /ha was 19 hrs and at 28.8g a.i. /ha it was 20 hrs. The maximum residue limit (MRL) of cartap hydrochloride residues on brinjal is not fixed by either PFA (Prevention of Food Adulteration, India) or Codex (Codex Alimentarius Commission, FAO/WHO). However, a MRL value of 0.02 mg/ kg has been proposed by UK and other European Union countries http://www.nda.agric.za/doaDev.) Based on the persistence pattern andMRL value of 0.02 mg/kg the safe preharvest interval (PHI) or waiting period of cartap hydrochloride on brinjal has been worked out as 2.62 and 2.95 days. Soil samples analyzed on the10th day were free from any cartap hydrochloride residue. From earlier studies it has been reported that cartap hydrochloride was used at low rates and degraded rapidly (having a half-life of four to six hours) when exposed to light, especially when applied as a thin film on inert surfaces or leaves (Wislocki *et al.,* 1989). Despite its rapid photodecomposition following application, cartap hydrochloride provides residual activity in the field because of its translaminar action and rapid penetration of leaf tissue. It is not only highly effective but as it comes from a biological source, it is a bio-rational product that can be used in an environmentally friendly manner in integrated pest-management programs. Cartap hydrochloride rapidly degrades in soil and at the soil surface it is subjected to rapid photo degradation, with half life ranging from 8 hours to 1 day. Loss of cartap hydrochloride from the soils is thought to be due to microbial degradation because it is undegraded in sterile soil (Wislocki *et al.,* 1989). Cartap hydrochloride rapidly degrades on plant surfaces (Mousa, 2004). This was also observed in the present study, as no residues were detected in brinjal after 5 days and in soil after 10 days. Since brinjal is harvested at regular intervals, the pre-harvest interval of 3 days suggested can be easily followed and cartap hydrochloride can be safely used on brinjal.

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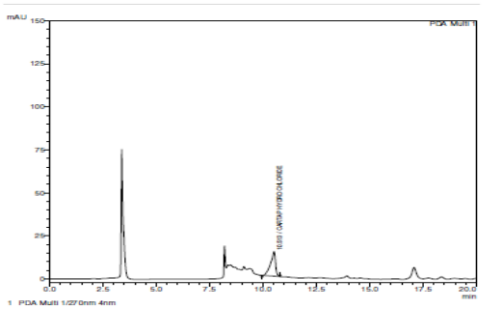
Table 1. Recovery study cartap hydrochloride on brinjal

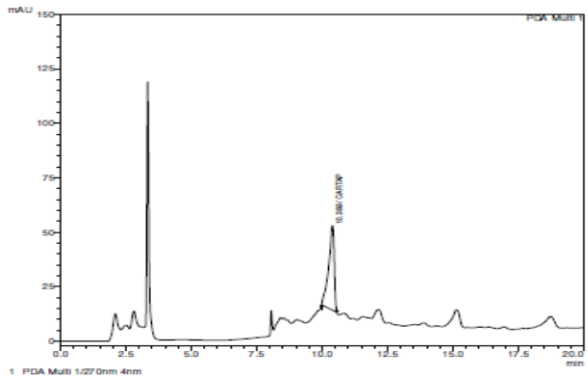
and soil at various fortification levels

fortified concentration (mg/kg)	concentration (%)* ± SD	
	brinjal	soil
0.05	96.6±8.08	90.6±3.05
0.25	90.6±2.31	90.6±4.62
0.5	93.3±1.15	83.3±0.58

***Mean of three replicate**



Cartap hydrchloride Standard Chromatogram



Field Sample Chromatogram Of Cartap Hydrochloride

Table 2: Residues of cartap hydrochloride in Brinjal and soil

Days after treatment	Cartap hydrochloride residues (mg/kg)	
	X Dose- 500 g ai/ha	2X Dose – 800 g ai/ha
	Mean	Mean
0	1.11±0.05	2.10±0.13
1	0.71±0.07	1.49±0.05
3	0.12±0.01	0.32±0.01
5	0.06±0.01	0.15±0.01
7	BDL	0.07±0.01
10	BDL	BDL
15	BDL	BDL
Soil at Harvest	BDL	BDL
Regression equation	Y=0.977+0.212X	Y=1.755+0.290X
DT 50	3.10	2.27

BDL- Below Determination Level (< 0.05mg/kg)
***Mean of three replicate.**