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Residues and dissipation of monocrotohos 36% SL in/on brinjal and soil

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Abstract
Field experiment was conducted during *Kharif,* 2016 at University Farm, MPKV, Rahuri, Dist.-Ahmednagar to determine the residues and dissipation of monocrotophos in/on brinjal and cropped soil. Two sprays of monocrotophos 36 SL were given at an interval of 10 days initiating first spray at fruit formation stage @ 250 g a. i./ha (625 ml/ha) and 500 g a. i./ha (1250 ml/ha). Samples of brinjal fruits were collected periodically at 0, 1, 3, 5, 7, 10 and 15 days after second spray. The cropped soil samples were collected at harvest. Collected samples were analyzed for Monocrotophos residues by using validated QuEChERS method on Gas Chromatography. The results revealed that average initial residues of monocrotophos in brinjal fruits were found to be 0.38 mg/kg at recommended and 0.75 mg/kg at double recommended dose and reached BQL (<0.05 mg/kg) on 10^{th} and 15^{th} day at both the doses, respectively. No residues were detected in the untreated control samples. The half life values (DT_{50}) were found to be 2.75 and 3.01 days, for recommended and double recommended dose, respectively. Soil samples collected at harvest did not recorded the residues of monocrotophos.

Keywords: Monocrotophos, brinjal, residues, dissipation

Introduction
Brinjal or eggplant (*Solanum melongena* L.) is an important solanaceous crop grown throughout the country except higher altitudes. It is versatile crop adapted to different agroclimatic regions. Brinjal can be grown throughout the year. The varieties of Brinjal displays a wide range of fruit shapes and colours ranging from oval or egg shaped to long club shaped; and from white, yellow, green through degrees of purple pigmentation to almost black. The global production of brinjal is around 50 million tons annually, with a net value of more than US$10 billion a year, which makes it the fifth most economically important solanaceous crop after potato, tomato, pepper, and tobacco (FAO, 2014) [3]. The top five producing countries are China (28.4 million tons; 57% of world's total), India (13.4 million tons; 27% of world's total), Egypt (1.2 million tons), Turkey (0.82 million tons), and Iran (0.75 million tons). In Asia and the Mediterranean, eggplant ranks among the top five most important vegetable crops (Frary *et al*., 2007) [4]. In India brinjal is grown in area of 7,30,400 ha with production of 1,28,00,800 MT and having a productivity of 17.5 MT/ha.(Anon, 2018) [1].
Monocrotophos is one of the highly toxic organophosphorus pesticides extensively used in agriculture. In India it is banned for use on vegetables since 2005 and is under 'restricted use' category. Monocrotophos 36 SL is a systemic & stomach broad spectrum insecticide and acaricidal action· It control such as bollworms, leaf-eating beetles insects and mites in various crops. The presence of residues in brinjal is matter of serious concern especially when brinjal fruits are consumed as vegetables. Therefore, it becomes mandatory to assess the impact of use of monocrotophos 36 SL from residues point of view for the safety of the consumers. However, very little information is available on persistence of monocrotophos on brinjal. Therefore, the present study was carried out to investigate the dissipation studies of monocrotophos residues in brinjal with the aims to asses risk to the consumer.

Material and Methods
Study was initiated on brinjal (var. Phule Arjun) at University Farm, MPKV, Rahuri, Ahmednagar (M.S.), India during *Kharif,* 2016. The first application of monocrotophos 36 SL @ 625 ml/ha and 1250 ml/ha was given at fruit formation stage by using knapsack sprayer. Subsequent applications were given at 10 days interval. Each treatment was replicated thrice. Control plots were sprayed with water only. One kg brinjal fruits were collected at random from each treatment at 0 (2 hours after spray), 1, 3, 5, 7, 10 and 15 days after second spray and soil samples were collected at harvest. Samples were brought to the pesticide residue laboratory and extracted immediately.

The residues of monocrotophos were estimated on Gas Chromatography.

Chemicals and Reagents

The Certified Reference Material of high purity of monocrotophos (99.8%) and commercial formulation was supplied by M/s. UPL, Mumbai. All the solvents used were of analytical grade. The solvents viz. Ethyl Acetate (HPLC grade), Acetonitrile, (HPLC grade), Sodium Chloride (AR grade), Magnesium Sulphate (AR grade), Sodium Sulphate (AR grade), Acetone (AR grade), were obtained from M/s. Rankem Fine Chemicals, Ltd., New Delhi and Primary Secondary Amines was procured from Agilent Technologies, Bangalore.

The primary stock solution of monocrotophos was prepared at 1000 mg/kg in toluene. The stock solution was further diluted to obtain working concentrations of 0.05, 0.1, 0.25, 0.4, 1.0 mg/kg.

Method Validation

Prior to analysis of samples, linearity of monocrotophos was established on Gas Chromatography. Accuracy and precision of the method was determined by per cent mean recovery and per cent relative standards deviation (RSD). Linearity was studied by injecting standard solutions of insecticide under study at six linear concentrations i.e. 0.05, 0.10, 0.25, 0.40, 0.50 and 1.00 mg/kg.

The linearity curve was established with concentration of the standard and corresponding peak area. Recovery study was conducted in order to establish the reliability of the method of analysis. The brinjal samples from control plots were used for recovery studies. Ten g homogenized sample was taken in 15 ml polypropylene tube. The Sample was spiked with three different concentrations *viz.* 0.05 mg/kg (LOQ), 0.25 mg/kg (5×LOQ) and 0.5 mg/kg (10×LOQ) in triplicate. The extraction and cleanup of sample was performed as described hereunder. Per cent recovery was calculated by using following formula.

$$\text{Per cent recovery} = \frac{\text{Quantity of pesticide recovered}}{\text{Quantity of pesticide added}} \times 100$$

Sampling

The brinjal fruits (1 kg) were extracted by QuEChERS method (Sharma, 2013) [6]. The entire laboratory sample (1 Kg) was crushed thoroughly in a mixture cum grinder. Approximately 10 g homogenized sample was weighed in a 50 ml polypropylene tube and the tube was kept in deep freezer for 10 min. To this, 10 ml ethyl acetate and 10 g anhydrous sodium sulphate was added, hand shaken vigorously and content was centrifuged at 3500 rpm for 5 min. Transferred 2 ml supernatant to 15 ml tube containing 50 mg PSA. The content was vortexed for 30 Sec and centrifuged for 2 min. at 2500 rpm. The supernatant was filtered through 0.2 micron filter in GC vials and performed GC analysis. The operating parameter were as below.

Column : DB-1 30m x 0.25 µ x 0.25 µ
Column Temperature : 170 ^{0}C to 220 ^{0}C @ 6.50 ^{0}C/min.3 min hold, 220 ^{0}C
min. to 280 ^{0}C@ 10 ^{0}C/min, 6 min hold
Injector Temperature : 250 ^{0}C
Column Temperature : 170 ^{0}C
FPD Temperature : 300 ^{0}C
Injection Volume : 1 µl
Column Flow : 0.96 ml/min
Retention Time Approx. : Monocrotophos : 7.10 min.

Results and Discussion

Method validation

The detector response to the neat standard of the monocrotophos was studied by injecting five linear concentrations. The graph was plotted with detector response against respective concentrations and linearity line was drawn. The response of the instrument was linear over the range tested and an R^2 value was 0.99. (Fig.1). These results indicated that the GC analysis is valid method for residue determination of the monocrotophos in brinjal. Accuracy of the analytical method was determined by recovery studies. The per cent recovery was within acceptable range of 70-120 per cent prescribed by SANCO (SANCO, 2011) [5] and mentioned in Table 1. Residues were estimated by comparison of peak area of the standard with that of the unknown or spiked samples run under identical conditions.

Dissipation of monocrotophos

Residues of monocrotophos (mg kg^{-1}) on brinjal at different time intervals after application @ 250 g a. i./ha (625 ml/ha) and 500 g a. i./ha (1250 ml/ha) are presented in Table 2. The average initial residues of monocrotophos in brinjal fruits were found to be 0.38 mg/kg at recommended dose and 0.75 mg/kg at double the recommended dose and reached BQL (<0.05 mg/kg) on 10th and 15th day, respectively. No residues were detected in the untreated control samples. The half life values (DT_{50}) were found to be 2.75 and 3.01 days, for both the doses, respectively. The residues of monocrotophos in soil were found to be below quantification limit of 0.05 mg/kg. These results are in agreement with findings of Subhash *et.al*. 2014 [7], who reported initial deposits of monocrotophos as 0.388, 0.689 and 0.891mg kg^{-1} when applied 100, 200 and 300 g a.i.h^{-1} on first day. On the contrary, Arora 2009, observed the harvest time residues of 1.25 µg/g of monocrotophos in non IPM brinjal plots. Residues of monocrotophos reached below detection limit (BDL) showing complete dissipation on 13th, 15th, 17th days respectively when it was applied at 100, 200 and 300 g a.i.h^{-1}, on brinjal.

Table 1: Recovery of monocrotophos in different substrates

Substrate	Recovery (%)											
	Fortification Level											
	0.05 mg/kg				0.25 mg/kg				0.5 mg/kg			
	R-I	R-II	R-III	Mean	R-I	R-II	R-III	Mean	R-I	R-II	R-III	Mean
Brinjal fruits	99.57	93.56	102.07	98.40	103.28	102.33	102.90	102.84	100.79	96.37	100.96	99.37
Soil	92.15	94.78	92.49	93.14	94.79	92.41	91.12	92.77	83.37	85.61	84.34	84.44

Table 2: Residues of monocrotophos in brinjal fruits and in soil

Days after last application	Residues (mg/kg)											
	Control				Monocrotophos 36% SL @250 g a.i./ha				Monocrotophos 36% SL @500 g a.i./ha			
	R-I	R-II	R-III	Mean	R-I	R-II	R-III	Mean	R-I	R-II	R-III	Mean
Brinjal fruits												
0	ND	ND	ND	ND	0.36	0.40	0.39	0.38	0.74	0.73	0.77	0.75
1	ND	ND	ND	ND	0.24	0.26	0.29	0.26	0.39	0.43	0.42	0.41
3	ND	ND	ND	ND	0.18	0.19	0.17	0.18	0.28	0.30	0.36	0.32
5	ND	ND	ND	ND	0.11	0.12	0.10	0.11	0.23	0.21	0.24	0.23
7	ND	ND	ND	ND	0.08	0.05	0.07	0.06	0.13	0.14	0.14	0.14
10	ND	ND	ND	ND	BQL	BQL	BQL	BQL	0.05	0.05	0.06	0.06
15	ND	ND	ND	ND	BQL	BQL	BQL	BQL	BQL	BQL	BQL	BQL
Soil (At final harvest)	ND	ND	ND	ND	BQL	BQL	BQL	BQL	BQL	BQL	BQL	BQL
DT_{50}	--				2.75				3.01			

* ND-Not Detected LOQ- 0.05 mg/kg BQL-Below Quantification Limit

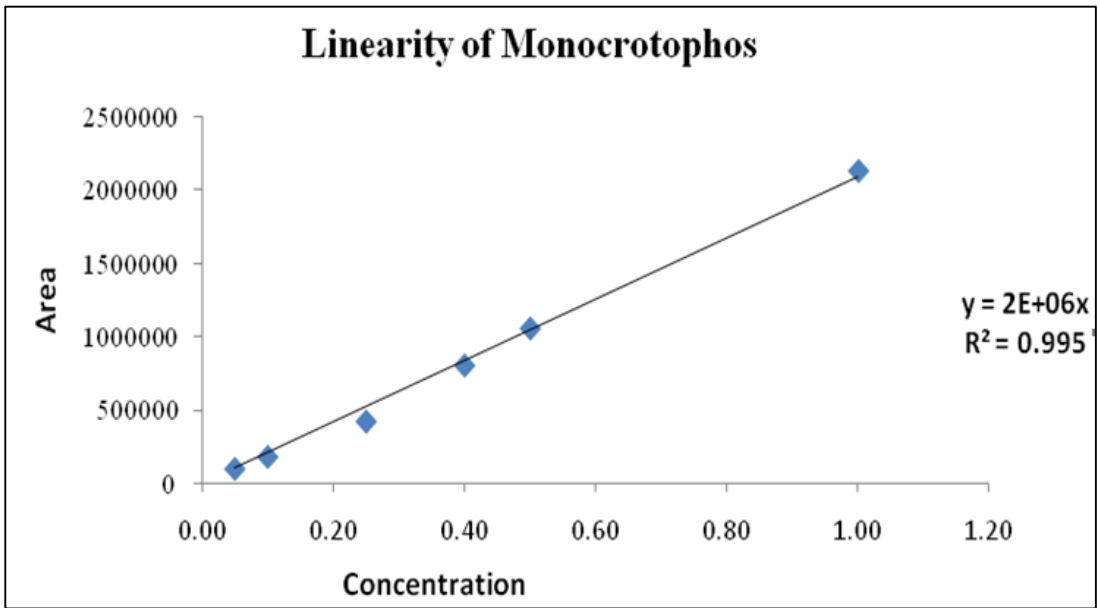

Fig 1: Linearity of monocrotophos

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