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## RESEARCH ARTICLE

### DISSIPATION PATTERN OF DIMETHOATE ON CHILLI IN POLYHOUSE AND OPEN FIELD

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#### ABSTRACT

Dimethoate is a systemic organophosphorous insecticide, applied on chilli to control sap sucking insects, thrips and mites in India. Use of dimethoate in poly house and open fields is very common. As per the ICAR recent suggestions dimethoate 30%EC was applied twice @300g a.i. ha<sup>-1</sup>, first spray at fruit initiation followed by second spray at 10 days interval as per the farmers practice to compare the dissipation dynamics of pesticide residues in open field and poly house situations so as to suggest PHIs for addressing the food safety issues. Dimethoate residues were quantified through regular sampling till the residues fell below determination level (BDL) of 0.05 mg kg<sup>-1</sup> following the validated QuEChERS method. The qualitative and quantitative analysis of dimethoate was performed on GC-FPD and GC-MSMS (TQD). Initial deposits of 2.42 mg kg<sup>-1</sup> detected in chilli samples collected from poly house, dissipated to BDL by 10th day with a half-life of 2.61 days. In open fields, deposits of 0.19 mg kg<sup>-1</sup> dissipated to BDL by 7th day with half-life of 36.47 days, indicating that dissipation is slow in poly house compared to open fields due to various factors. There are no maximum residue limit for dimethoate in chilli as per Codex Alimentarius Commission (CAC) and hence based on the present study Dimethoate initial deposits (0.19 mg kg<sup>-1</sup>) are less than FSSAI MRL of 2 mg kg<sup>-1</sup>, hence a PHI of 1 day can be recommended. In case of poly house, initial deposit of 2.42 mg kg<sup>-1</sup> was observed and hence a safe waiting period of 7 days can be recommended since FSSAI MRL is 2 mg kg<sup>-1</sup> for food safety.

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## INTRODUCTION

Chilli (*Capsicum annum* L.), also called "red pepper," is an important cash crop in India and is used throughout the world as a spice, an important ingredient in day to day curries, pickles and chutnies. It is a rich source of A, C, E and P and an alkaloid capsaicin, which has high medicinal value and is used in many pharmaceutical preparations. India is the world's largest producer, consumer and exporter of chilli with an area 794.12 Thousand ha and production of 1304.38 m t, respectively. Andhra Pradesh ranks first both in area (210.02 thousand ha) and production (685.15 Thousand m t) (NHB, 2013). In recent years, due to better cost benefit ratio, farmers are growing chillies in controlled atmosphere conditions, majorly in poly house, besides regular open fields during crop seasons. Chilli suffers major quantitative and qualitative loss in production due to chilli thrips, *Scirtothrips dorsalis* Hood and yellow mite, *Polyphagotarsonemus latus* (Banks) and fruit borers *Helicoverpa armigera* (Hubner).

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The overall reduction in fruit yield of chilli due to thrips and mites damage is up to 34% (Thania et al., 2011). These pests not only cause reduction in yield, but also act as vectors for several viral diseases and cause complete failure of crop. A number of pesticides are being frequently used, to combat these pests. However, some of these insecticides leave residues on pods and these residues may persist up to harvest. Presence of pesticide residues in the harvested chillies is posing problem at the time of export and in recent times importing countries have rejected few consignments. Pesticide use has increased rapidly over the last two decades at the rate of 12 % per year (Thacker et al., 2005). Many farm gate chilli samples showed presence of insecticide residues (Singh et al., 1999). Dimethoate [O,O-dimethyl-S-(N-methylcarbamoylmethyl)-phosphorodithioate] is a systemic organophosphorous insecticide, applied on chilli to control thrips and mites. Since chilli is an important component of daily food, food safety issues are very essential. In recent years due to the support of the Government under National Horticulture Mission (NHM), chilli is widely cultivated under poly house conditions. As per Central Insecticides Board and Registration Committee (CIBRC), dimethoate is recommended for use on chilli but no MRLs are set.

Since the persistence and dissipation of insecticides in poly houses will be different from normal conditions, the present study is proposed to monitor the usage on chilli, dissipation studies of dimethoate both in poly house and open field conditions and also evaluation of decontamination methods for removal of dimethoate residues, so as to recommend the safe waiting periods based on the Maximum Residue Limits (MRLs) calculated, as it helps in recommending risk mitigation protocols for food safety.

## MATERIALS AND METHODS

**Chemicals and Reagents:** Certified Reference Materials (CRM) of Dimethoate (96.9% purity) were procured from M/S Sigma Aldrich, Germany, and primary, intermediary and working standards were prepared from the CRMs using GC PR grade acetone and hexane as solvents. Working standards of were prepared in the range of 0.01 ppm to 0.5 ppm in 10 mL calibrated graduated volumetric flask using distilled n-hexane as solvent. Primary Secondary Amine (Agilent), magnesium sulfate anhydrous (Emsure grade of Merck), sodium sulfate anhydrous (Emparta ACS grade of Merck), acetonitrile (HPLC gradient grade of Merck), acetic acid glacial (HPLC grade of Merck), acetone (Emplure grade of Merck), n-hexane (HPLC grade of Merck) were used during the study for sample preparation. Dimethoate 50% EC was procured from local market.

### Analytical Instruments and Limits of Detection

Working standards were injected in Gas Chromatograph (Agilent 7890 B) with Electron Capture Detector (ECD) and Thermionic Specific Detector (TSD) with injector split ratio of 1:10 using VF-5ms Capillary Column) and confirmatory analysis was done on Bruker Scion 436 GC-MS/MS Triple Quadrupole Detector (EI) using Multiple Reaction Monitoring (MRM) method (Qualifier ions: 339>188, 339>251, 339>269, 139>97; Quantifier Ions: 139>97). It is observed that the limit of detection for dimethoate is 0.05 ng in GC-TSD with linearity range of 0.05 ng to 5 ng.

**Method validation:** Prior to field experiments, QuEChERS (Quick Easy Cheap Effective Rugged Safe) method for extraction and clean up was validated as per SANCO/12571/2013 guidelines. Chilli fruits (5 kg) collected from control plots were homogenized with high volume homogenizer (Robot Coupe Blixer 7L) and 15 g was taken in to 50 mL centrifuge tubes. The required quantity of dimethoate intermediary standards were added to each 15 g sample to get fortification levels of 0.50 mg kg<sup>-1</sup> and 0.25 mg kg<sup>-1</sup> in three replications each. 30±0.1 mL acetonitrile was added to the tube, and sample was homogenized for 2-3 min using Heidolph silent crusher (low volume homogeniser). Then 3±0.1g sodium chloride was added to tube and mixed by shaking gently, and centrifuged for 3 min at 2500-3000 rpm 00 xg with Remi R-238 to separate the organic layer. The top organic layer of about 16 mL was taken into the 50 mL centrifuge tube to which 9±0.1 g anhydrous sodium sulphate was added to remove the moisture content. 8 mL of extract was taken in to 15 mL tube containing 0.4±0.01g PSA sorbent (for dispersive solid phase d-SPE cleanup) and 1.2±0.01 g anhydrous magnesium sulphate, and the sample tube was vortexed for 30 sec followed by centrifugation for 5 min at 2500-3000 xg. The extract of

(2mL) was transferred into test tubes and evaporated to dryness using concentration work station (Turbovap LV of Caliper life sciences) with nitrogen gas and reconstituted with 1mL n-Hexane: Acetone (9:1) for dimethoate analysis. Chilli samples fortified with dimethoate at 0.05, 0.25 mg kg<sup>-1</sup> and 0.5 mg kg<sup>-1</sup> were analyzed and the mean recovery of the residues calculated for applying recovery factor while calculating the residues in samples. Fortification and recovery test results are presented in Table 1 and the method followed for qualitative and quantitative estimation of dimethoate is suitable up to 0.5 mg kg<sup>-1</sup> levels as the recoveries obtained are 100.4%,95.74% and 88.70% 0.05, 0.25 and 0.50 mg kg<sup>-1</sup> fortification level. The residues detected below 0.05 mg kg<sup>-1</sup> were mentioned as levels Below Determination Level (BDL) in all cases.

### Field experiments and sample collections

Chilli crop (Popular hybrid pusajwala) was raised in both poly house and open field in Randomized Block Design at spacing of 60×45 cm with plot size of 20 m<sup>2</sup> and all Good Agricultural Practices (GAPs) recommended by University were followed. Dimethoate 30% EC procured from local market was sprayed @300g a.i. ha<sup>-1</sup> twice; first spray at fruit initiation stage followed by second spray at 10 days after first spray, using high volume knapsack sprayer with a spray solution of 500 L ha<sup>-1</sup>. Pest damage free and crack free chilli fruits of 5 kg were collected from each plot in separate polythene bags and brought to laboratory. Samples were collected at regular intervals i.e. 0, 1, 3, 5, 7, 10, 15, 20 days after last spray for dissipation studies.

## RESULTS AND DISCUSSION

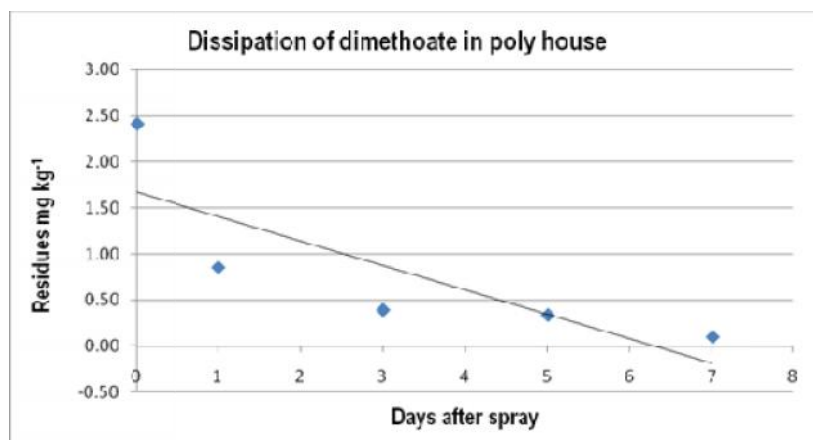
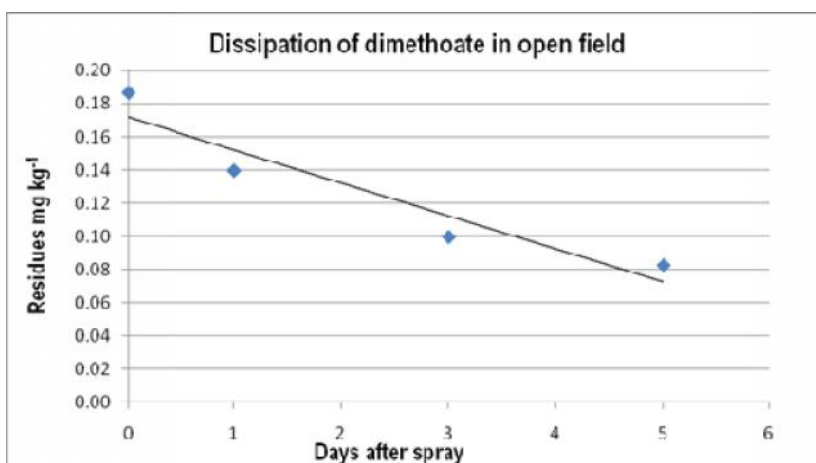
The initial deposit and subsequent residues of dimethoate on chilli in open field and poly house situation at an interval of 0, 1, 3, 5, 7, 10, 15 and 20 days after second spray are presented in Table 2 and depicted in Figures 1 and 2. In open field situation, the initial deposit of 0.19 mg kg<sup>-1</sup> gradually dissipated to 0.14, 0.10 and 0.08 mg kg<sup>-1</sup> at 1, 3 and 5 days, respectively. The per cent dissipation was 26.31, 47.36 and 57.89, respectively. The residues fell Below Determination Level (BDL) of 0.05 mg kg<sup>-1</sup> by 7<sup>th</sup> day. The half-life (RL50) of dimethoate was worked to be 36.47 days. The regression equation was  $Y = 0.171 + (-0.019) X$  with R<sup>2</sup> of 0.902. There are no maximum residue limits (MRL) for dimethoate on chilli as per Codex Alimentarius Commission (CAC), while Food Safety and Standards Authority of India (FSSAI) suggests 2 mg kg<sup>-1</sup> for fruits and vegetables, and hence safe waiting period of 1 day can be suggested, as the initial deposits are less than the MRL. The half-life of dimethoate on chilli was 36.47 days in open field situation. In poly house, initial deposits of 2.42 mg kg<sup>-1</sup> were detected, which dissipated to 0.10 mg kg<sup>-1</sup> by 7<sup>th</sup> day after the second spray. The residues of 0.86, 0.39, 0.34 and 0.10 mg kg<sup>-1</sup> were recorded at 1, 3, 5 and 7 days, respectively and dissipated to 64.46, 83.88, 85.95 and 95.86 per cent on 1, 3, 5 and 7<sup>th</sup> day, respectively. The results showed that the residues of dimethoate reached Below Determination Level (BDL) of 0.05 mg kg<sup>-1</sup> in 10 days while half-life was worked out to be 2.61 days. The regression equation was  $Y = 1.671 + (-0.265) X$  with R<sup>2</sup> of 0.664. There are no maximum residue limit for dimethoate in chilli as per Codex Alimentarius Commission (CAC) while Food Safety and Standards Authority of India (FSSAI) suggests 2 mg kg<sup>-1</sup> for vegetables

**Table 1. Recovery of dimethoate residues in chilli**

Details	Recoveries of dimethoate from fortified chilli samples					
	Fortified level (mg kg <sup>-1</sup> )					
	0.05 mg kg <sup>-1</sup>		0.25 mg kg <sup>-1</sup>		0.50mg kg <sup>-1</sup>	
	Residues recovered (mg kg <sup>-1</sup> )	Recovery %	Residues recovered (mg kg <sup>-1</sup> )	Recovery %	Residues recovered (mg kg <sup>-1</sup> )	Recovery %
R1	0.049	97.3	0.245	97.71	0.450	90.04
R2	0.052	103.4	0.235	92.78	0.432	86.33
R3	0.050	100.5	0.238	93.55	0.449	89.74
Mean		100.4		95.74		88.70
SD		3.060		2.116		2.060
RSD		3.048		2.210		2.323

**Table 2. Dissipation of dimethoate in chilli in poly house in open fields**

Days after last spray	Residues of dimethoate (mg kg <sup>-1</sup> )					Residues of dimethoate (mg kg <sup>-1</sup> )					Dissipation %
	R1	R2	R3	R4	Average	R1	R2	R3	R4	Average	
0	2.26	2.42	5.38	2.47	2.42	0	0.18	0.20	0.18	0.19	0
1	0.86	0.80	0.89	0.88	0.86	64.46	0.14	0.14	0.13	0.14	26.31
3	0.37	0.39	0.42	0.40	0.39	83.88	0.10	0.10	0.11	0.10	47.36
5	0.31	0.32	0.36	0.36	0.34	85.95	0.10	0.08	0.07	0.09	57.89
7	0.11	0.10	0.10	0.10	0.10	95.86	BDL	BDL	BDL	BDL	100
10	BDL	BDL	BDL	BDL	BDL	100	BDL	BDL	BDL	BDL	100
15	BDL	BDL	BDL	BDL	BDL	100	BDL	BDL	BDL	BDL	100
20	BDL	BDL	BDL	BDL	BDL	100	BDL	BDL	BDL	BDL	100
Regression equation	Y = 1.671 + (-0.265) X					Y = 0.171 + (-0.019) X					
R <sup>2</sup>	0.664					0.902					
Half-life	2.61 days					36.47 days					
Safe waiting period	:7 days					: 1 days					
Codex MRL: NA											
FSSAI MRL: 2 mg kg <sup>-1</sup> for Fruits and Vegetables											
BDL	: Below Determination Level (< 0.25 mg kg <sup>-1</sup> )					BDL					: Below Determination Level (< 0.25 mg kg <sup>-1</sup> )
NA: Not available											

**Fig. 1. Dissipation of dimethoate in poly house****Fig. 2. Dissipation of dimethoate in open field**

and hence based on the present study, PHI of 7 days can be recommended as the residues degraded to BDL by 10<sup>th</sup> day. From Tables 2 it is evident that there is clear difference in dissipation pattern of dimethoate in poly house and open fields. Initial deposit of 2.42 mg kg<sup>-1</sup> was recorded in poly house, where as in open fields it was 0.19 mg kg<sup>-1</sup>. Dimethoate reached Below Determination Level (BDL) of 0.05 mg kg<sup>-1</sup> by 10<sup>th</sup> day in poly house, whereas in open field trial by 7<sup>th</sup> day. In open field initial deposit of 0.19 mg kg<sup>-1</sup> was recorded which is in agreement with findings of Reddy *et al.* (2007) who reported dimethoate @ 300 g a.i. ha<sup>-1</sup> sprayed on chilli recorded initial deposit of 0.35 mg kg<sup>-1</sup> and they also suggested safe waiting period of 1 day. In poly house initial deposit of 2.42 mg kg<sup>-1</sup> was recorded which is in line with the results of Pandey *et al.* (2004) who reported that dimethoate sprayed @ 1.5 ml/l on cabbage had initial deposit of 2.46 mg kg<sup>-1</sup> and they suggested half-life of 2.61 days. Since sufficient literature is not available on the dissipation pattern on dimethoate in chilli or on related crops in poly houses, discussion part for poly house data is not done. However, it can be attributed that the dissipation is slow in poly houses compared to open fields due to very common factors such as cool climatic conditions and less sun light penetration in poly house. The studies are very helpful for the fixation of MRLs and recommendation of MRLs based on the dose recommendations of dimethoate in poly houses / controlled environmental conditions and based on the present scientific studies 7 day PHI can be recommended where as in case of open field situations 1 day PHI can be recommended taking in to consideration initial deposits and dissipation dynamics of dimethoate in both open field and poly house.

### Conclusion

The dissipation pattern of insecticides varied in poly houses and open fields, where initial deposits were comparatively higher and dissipated slowly in poly houses.

There are no maximum residue limit for dimethoate in chilli as per Codex Alimentarius Commission (CAC) and hence based on the present study. Dimethoate initial deposits (0.19 mg kg<sup>-1</sup>) are less than FSSAI MRL of 2 mg kg<sup>-1</sup>, hence a PHI of 1 day can be recommended. In case of poly house, initial deposit of 2.42 mg kg<sup>-1</sup> was observed and hence a safe waiting period of 7 days can be recommended since FSSAI MRL is 2 mg kg<sup>-1</sup> for food safety.

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