Biological Performance of Phosalone and Malathion in Grapes (*Vitis vinifera* L. cv *Anab-e-shahi*)

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Abstract

The experiment was conducted during February-March, 2006 with grape variety *Anab-e-shahi*. Two sprays of malathion (0.05%) and phosalone (0.07%) were applied with first spray at berry formation stage and second spray at 15 days after first spray. The dissipation pattern of malathion revealed that initial deposit of malathion 1.24 mg kg^{-1} was dissipated to 0.01 mg kg^{-1} on 15 days after second spray when malathion (0.05%) was sprayed twice at berry formation stage. The dissipation pattern of phosalone revealed that the initial deposit of phosalone 0.33 mg kg^{-1} was dissipated to 0.01 mg kg^{-1} on 10 days after second spray when phosalone (0.07%) was sprayed twice at berry formation stage. The maximum residue limit for malathion and phosalone is 8 and 5 mg kg^{-1}, respectively. The initial deposits of malathion and phosalone were below maximum residue limits. The waiting period for safe harvest of grape berries after two spray of malathion (0.05%) and phosalone (0.07%) when given at berry formation stage is one day.

1. Introduction

Grape (*Vitis vinifera* L.) is an important fruit crop in India occupying an area of about 40,842 ha with an annual production of 9.69 lakh tonnes out of which the state of Andhra Pradesh accounts for 1,765 ha area and production of 0.35 lakh tonnes. A large number of insect pests on grapes, viz. flea beetle (*Scelodontia stingicollis* Mots.), thrips (*Retithrips syriacus* Mayet.), gram caterpillar (*Helicoverpa armigera* Hb.), tobacco caterpillar (*Spodoptera litura* Fb.) and mealy bug (*Maconellicoccus hirsutus*) (Reddy, 2001) are responsible for both qualitative and quantitative losses. Table grapes are not only consumed fresh but also eaten as a whole without peeling. The insecticides like malathion and phosalone are extensively used for controlling above pests. The information on safe or waiting period for the harvest of berries after insecticidal spray is lacking. Keeping the above facts in mind the present study was initiated.

2. Materials and Methods

Field experiment was conducted during February-March, 2006 with grape variety *Anab-e-shahi*. Each treatment was replicated thrice in a randomized block design. Two sprays of Malathion (0.05%) and Phosalone (0.07%) were given with first spray at berry formation stage and second one at fifteen days after first spray with foot sprayer. For the residue analysis of malathion, the representative grape samples (500 g) were collected from each plot at 0 (2 h), 1, 3, 5, 7, 10, 15, 20 and 30 days after last spray. The grape samples were chopped and blended and a representative sample of 50 g was taken and extracted with solvent mixture of 100 ml of acetone: hexane (1:1). The extract was transferred to separatory funnel, diluted with water and partitioned with dichloromethane. The lower dichloromethane phase was passed through anhydrous sodium sulphate and concentrated to about 5 ml. The concentrated extract of malathion was passed through a glass column containing 5 g florisol, 5 g of neutral alumina grade III over laid with a 20 mm layer of anhydrous sodium sulphate. The column was then eluted with solvent mixture of n-hexane: acetone (9:1) and the elute was concentrated to 5 ml for Gas chromatograph (GC) analysis. For residue analysis of phosalone, the grape berries were chopped into pieces and a representative 50 g sample was withdrawn. The sample was blended thrice with 100 ml followed by, 50 and 50 ml acetone in a high-speed blender, filtered and the combined extract concentrated to 50 ml using a rotary vacuum evaporator. The concentrated extract was transferred to separatory funnel (1 l) and diluted with 5% aqueous sodium chloride solution and partitioned thrice into 100 followed by 50 and 50 ml of dichloromethane. The dichloromethane layer was passed

through anhydrous sodium sulphate. The dichloromethane extract was evaporated to near dryness. The complete removal of dichloromethane was ensured by repeatedly adding acetone to the residues followed by evaporation under vacuum. The dried phosalone residues thus obtained was dissolved in 5 ml of acetone and transferred into glass column containing adsorbent mixture of charcoal, celite and magnesium oxide (2:2:1). The transferred extract in the column was eluted with 150 ml of chloroform. The elute was concentrated to near dryness, which was finally dissolved in 5 ml of n-hexane for GC analysis.

The residues of malathion and phosalone were determined using Varian 3800 Gas chromatograph equipped with electron capture detector and capillary column VF-1 MS, 15 m, 0.25 mm, id 0.25 mm film thickness. The operating temperatures were detector 300°C, injector 280°C, column oven programmed at 70°C for 1 min, increased @ 20°C min^{-1} to 150°C for 5 min, increased @ 2°C min^{-1} to 240°C for 5 min (total time 66 min). The carrier gas (nitrogen flow) was 1 ml min^{-1} and make up flow was 15 ml min^{-1}. The retention time for malathion and phosalone was 19.10 and 40.8 min, respectively. The residue data was subjected to regression analysis and waiting periods (T_{tol}) and half life ($T_{1/2}$) were calculated (Gunther and Blinn 1955; Hoskins, 1961).

3. Results and Discussion

The recovery test was carried out at 0.1 and 0.01 mg kg^{-1} levels for fortification of malathion and phosalone on grape berries and soil. The percent recovery of malathion was 85.02 at 0.01 ppm and 89.37 at 0.1 ppm level of fortification in grape berries, while it was 88.09 and 91.75%, respectively at 0.01 and 0.1 ppm level of fortification in the soil. The percent recovery of phosalone was 83.52 at 0.01 ppm and 86.19 at 0.1 ppm level of fortification in grape berries, while it was 88.49 and 86.72%, respectively at 0.01 and 0.1 ppm level of fortification in the soil (table 1).

Malathion and phosalone residues in grape berries and soil are presented in table 2. The initial deposit of malathion 1.24 mg kg^{-1} was dissipated to 0.01 mg kg^{-1} on 15th day after second spray with corresponding dissipation of 99.19%. Based on the first order kinetics, the half-life of malathion was 2.76 days.

Table 1: Percent recovery of malathion and phosalone in grape berries and soil

Fortification level (mg kg^{-1})	Percent recovery of malathion		Percent recovery of phosalone	
	Grape berries	Soil	Grape berries	Soil
0.1	89.37	91.75	86.19	86.72
0.01	85.02	88.09	83.52	88.49

The initial deposits of malathion residues were below MRL (maximum residue limits) of 8 (Codex alimentarious, 1998). Hence it is pertinent from the above study that grape berries can be harvested one day after two sprays of malathion @ 0.05% for safe consumption. The present study is in agreement with the results of Prem Chand Kashyap et al. (1999) who reported that initial deposits of malathion were below tolerance limit and hence no safe waiting period was required when malathion 0.05% was sprayed in tomato. Singh et al. (2006) reported that waiting period of 5 days is recommended for the safe consumption of fenugreek as a vegetable as well as green fodder when malathion was sprayed at 625 ml ha^{-1}, while Bandral and Sharma (2007) investigated that almost all the residues of malathion in leaves and pods dissipated on the 7th day after second spray on rapeseed crop. The variation in results of the present study may be due to variation of the crop and surface exposure of the pesticide to the crop.

The initial deposit of phosalone 0.33 mg kg^{-1} was dissipated to 0.02 mg kg^{-1} on 10 days after second spray with corresponding dissipation of 96.96%. Based on the first order kinetics, the half-life of phosalone was 4.69 days. The initial deposits of phosalone residues were below MRL of 5 (Codex alimentarious, 1998). Hence it is recommended grape berries can be harvested one day after two sprays of phosalone @ 0.07% for safe consumption. Pareek and Kavadia (1990) reported that waiting period for phosalone on round gourd is 7 days while Kandasamy et al. (1998) reported that waiting period for

Table 2: Dissipation of malathion (0.05%) and phosalone (0.07%) residues in grapes

Day after second spray	Malathion		Phosalone	
	Residues mg kg^{-1}	Dissipated (%)	Residues mg kg^{-1}	Dissipated (%)
0	1.24	-	0.33	-
1	0.94	24.19	0.22	33.33
3	0.50	59.67	0.14	57.57
5	0.15	87.90	0.06	72.72
7	0.05	95.96	0.02	93.93
10	0.02	98.38	0.01	96.96
15	0.01	99.19	-	-
20	-	-	-	-
30	-	-	-	-
MRL	8.00		5.00	
T_{tol} (days)	1.00		1.00	
$T_{1/2}$ (days)	2.76		4.69	
Regression equation	Y=2.374-0.0641		Y=2.7926-0.0632x	

phosalone in chillies is 2 days. Talebi (2006) reported that the half-life of phosalone in fresh and dried alfalfa was 1.8 and 3.3 days, respectively. The variation in the half-life values and waiting periods for safe harvest of the grape berries to other workers may be due to variation in exposure of surface area to insecticidal spray in grapes when compared to other crops.

4. Conclusion

The initial deposits of malathion and phosalone when sprayed twice at 0.05% and 0.07% during berry formation stage was below the maximum residue limits prescribed by the Codex alimentarious. The waiting period for safe harvest of grape berries when sprayed malathion 0.05% and phosalone 0.07% twice at berry formation stage was one day only. Malathion showed faster dissipation while phosalone dissipated slowly.

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