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Residue behavior of ready-mix formulation of endosulfan and cypermethrin in cauliflower, *Brassica oleracea* var *botrytis*

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ABSTRACT

The present study was done in 2010-11 to compare the persistence of endosulfan and cypermethrin in ready-mix formulation of Endohyper 40 EC (endosulfan 35% + cypermethrin 5%) with individual insecticide endosulfan (Endocel 35 EC) and cypermethrin (Challenger 25 EC). Endosulfan and cypermethrin were applied at recommended rate 350 g and 50 g ai/ha and at double the recommended rate 700 g and 100 g ai/ha on cauliflower crop. The curds and soil samples were collected at different intervals and analyzed after second spray. Residues of both insecticides were determined by using gas chromatograph, Agilent 6890N having electron capture detector. In curds the endosulfan and cypermethrin residues reached below detectable limit in 15 and 7 days when applied at recommended rate and in 20 and 10 days when applied at double recommended rate. Endosulfan deposits were reduced to half in 1.81-2.32 days and cypermethrin deposits required 1.19-1.44 days to reduce to half as combi product but when applied individually endosulfan initial deposits were reduced to their half in 1.21-1.51 days and cypermethrin deposits became half in 1.38-1.69 days. In soil residues of endosulfan persisted for 0-20 days whereas residues of cypermethrin persisted for 0 day only.

Keywords: Endosulfan; cypermethrin; cauliflower; residues; persistence

INTRODUCTION

Vegetables are an essential component of the human diet and it has been estimated that 85-90 per cent of the pesticides in human bodies are received through foods. Vegetables are of direct concern with respect to the buildup of pesticide residues from point of health hazards to consumers as they are consumed afresh immediately after field harvest without giving much time for dissipation of residues. Among cole crops cauliflower, *Brassica oleracea* var *botrytis* L is an important vegetable in India and is grown extensively in Himachal Pradesh as a cash crop. One of the major constraints in commercial growth of this crop is the heavy damage caused to it by insect pests. Hence various insecticides are being used by the

farmers in the state for their effective control. The crop is susceptible to attack by a large number of pests including diamond back moth, *Plutella xylostella*, leaf eating caterpillars and aphids (Regupathy et al 1985, Patel et al 1999). In order to prevent the damage to the crop the farmers rely heavily on the usage of many pesticides viz quinalphos, phosalone, fenvalerate, cypermethrin, deltamethrin etc (Awasthi 1986). Improper and injudicious use of pesticides besides posing health threat to the farm workers also leaves harmful pesticide residues on the crop and soil and causes development of pest resistance leading to the losses to the crops (Kumar and Singh 2014). To combat this menace usage of insecticide mixtures is a promising option. In India a large number of ready-mix insecticide formulations have been registered for use on various crops (Regupathy et al 2004). Endohyper 40 EC, a combination of endosulfan 35 per cent + cypermethrin 5 per cent is one such ready-mix formulation. Endosulfan has been banned in India after 2011 but these studies were done before it. Ready-mix insecticide formulations have been found effective against insect pests of many vegetables (Dharne and Kabre 2009, Kumar and Shivaraju 2009). In cauliflower, endosulfan and cypermethrin separately or in ready-mix have been found effective in controlling insect pests (Tripathi et al 2003, Mishra 2002, Sarangdevot et al 2010a, 2010b). The effectiveness of these two insecticides on cauliflower has propelled the use of combination product Endohyper 40 EC (endosulfan 35% + cypermethrin 5%) on cauliflower. However information available on the persistence behavior of these ready-mix products in/on cauliflower curds and soil is scare under present environmental conditions. Therefore the present studies were contemplated in 2010-11 with an objective to study the persistence behavior of pre-mix endosulfan and cypermethrin and its comparison with the residue status when applied individually following spray application.

MATERIAL and METHODS

Chemicals: Analytical grade reagents viz acetone, hexane, toluene, dichloromethane, acetonitrile, sodium chloride, anhydrous sodium sulphate, Florisil and Celite 545 were obtained from M/S Merck Specialties Pvt Ltd, Mumbai, Maharashtra, India. Pesticide residue grade charcoal was procured from M/S Fluka Analytical, Sigma-Aldrich Schweiz, Industries straβe 25, CH-9470 Buchs SG, Schweiz. Endosulfan formulation (Endocel 35 EC) and the ready-mix formulation Endohyper 40 EC containing 35 per cent endosulfan and 5 per cent cypermethrin were obtained from M/S Excel Crop Care Ltd and cypermethrin (Challenger 25 EC) from M/S Tropical Agrosystem Ltd.

Design of experiment: Experiments on the persistence behaviour of endosulfan and cypermethrin individually or as ready-mix

were conducted in two years during 2010 and 2011 at the experimental farm of the Department of Entomology, Dr YS Parmar University of Horticulture and Forestry, Solan, HP. Trials were laid out in a randomized block design and each treatment was replicated thrice.

Application of insecticides: Cauliflower, *Brassica oleracea* var *botrytis* L was sprayed at curd formation stage with individual insecticide endosulfan (Endocel 35 EC) and cypermethrin (Challenger 25 EC) and the ready-mix formulation Endohyper 40 EC (containing 35% endosulfan and 5% cypermethrin) at the recommended rate (RR) 350 g and 50 g ai/ha and at double recommended rate (DRR) 700 g and 100 g ai/ha respectively. Two sprays were given at 15 days interval. Control plots with only water spray were maintained simultaneously for comparison.

Collection of samples: After the second spray curd samples (1 kg) from each replication were collected randomly at 0 (2 hours after spray), 1, 3, 5, 7, 10 and 15 days intervals. Soil samples (1 kg) from each replication were collected on 0, 10 and 20 days after application. The cauliflower curds were homogenized and analysed for respective insecticides. Soil samples were shade-dried and sieved.

Extraction and clean up: Cauliflower curd samples were analysed according to Sharma (2007). Homogenized cauliflower curd sample (100 g) was extracted with 200 ml of acetone, filtered through Buchner funnel under low suction and rinsed with 50 ml acetone. From total extract an aliquot of 60 ml (30 g equivalent of sample) was transferred to 1 litre separatory flask and extracted with 200 ml mixture of hexane and dichloromethane (1:1, v/v). The separatory flask was shaken vigorously for 1 min and then allowed the phases to separate into organic and aqueous phase. The lower aqueous phase was transferred to another 1 litre separatory flask and remaining organic phase was retained in the same separatory flask. Ten ml saturated sodium chloride solution was added to the left amount of aqueous phase and again partitioned twice with 100 ml dichloromethane. Lower aqueous phase was discarded and upper organic phase was transferred to the 1st separatory flask. Pooled organic phase was passed through anhydrous sodium sulfate and evaporated to dryness by using vacuum rotary evaporator at 40^{o}C. Finally the residues were taken in 3 ml (1 + 2) acetone for cleanup.

One ml sample extract was diluted with 10 ml of acetone/hexane (1:9) mixture, loaded on 4 g Florisil column (22 mm id), overlaid with 2 g layer of sodium sulphate and eluted with 50 ml solvent mixture (50% dichloromethane:48.5% hexane:1.5% acetonitrile). Another 2 ml fraction of sample was loaded on a charcoal column which was prepared by placing one inch

layer of Celite 545, 6 g absorbent mixture (1:4 w/w charcoal:celite 545) and then overlaid with 2 g sodium sulfate. The sample extract was loaded on to the column and eluted with 200 ml of 2:1 acetone/dichloromethane mixture. Eluate from both the column fractions was pooled and evaporated to dryness in vacuum rotary evaporator at 50°C. The residues were redissolved in 3 ml toluene and one µl was injected into gas chromatograph for residue estimation.

Soil samples were analyzed according to the method given by Brar (2003). A dried and sieved representative soil sample of 20 g was mixed with 0.5 g activated charcoal + 0.5 g Florisil and packed in 2×40 cm glass column containing about 5 cm layer of anhydrous sodium sulphate over a plug of cotton at the bottom. The column was eluted with 50 ml mixture of acetone and hexane (1:4). The eluate was evaporated to dryness and residues were taken in 1 ml toluene. Finally one µl was injected into gas chromatograph for residue estimation.

Residue estimation: Residues were estimated by using Gas Chromatograph (Agilent 6890N) having ECD detector and DB-5 Ultra Performance Capillary column (Cross-linked Methyl Silicon, length 30 m, 0.250 mm internal diameter with 0.25 µm film thickness).

Instrument conditions: Column temperature was programmed as 100°C for 1 min, 30°C/min up to 150°C (hold time 2 min), 3°C/min up to 205°C and finally temperature was raised up to 260°C at the rate of 10°C/min (hold time 10 min). Temperature of injection port and ECD (detector) were kept at 250 and 300°C respectively. Flow rate of carrier gas nitrogen was 1ml/min and make up gas flow rate was 60 ml/min. Under these gas chromatographic parameters retention time of alpha-endosulfan, beta-endosulfan, endosulfan sulfate and cypermethrin was 19.333, 22.461, 24.590 and 30.771 min respectively. Total of alpha-endosulfan, beta-endosulfan and endosulfan sulfate residues was reported as endosulfan residue.

Method validation: The analytical method employed to estimate residues was validated by spiking the control curd and soil samples at five different concentrations viz 0.01, 0.05, 0.10, 0.5 and 1.0 mg/kg for alpha-endosulfan, beta-endosulfan and endosulfan sulfate. Whereas in case of cypermethrin samples were spiked at 0.05, 0.10, 0.20, 0.50 and 1.0 mg/kg concentrations. The limit of determination (LOD) of alpha-endosulfan, beta-endosulfan and endosulfan sulfate was 0.01 mg/kg and for cypermethrin LOD was 0.05 mg/kg. The residue data were subjected to statistical analysis (Hoskins 1961).

RESULTS and DISCUSSION

The Table 1 depicts reliability of analytical method tested by spiking of untreated cauliflower curds and soil samples at different concentrations. Recovery of endosulfan was between 80.00-93.00 per cent with relative standard deviation (RSD) of 0.040-1.004 per cent in curds and 80.00-91.90 per cent with 0.017-0.936 per cent RSD in soil fortified samples. Recovery of cypermethrin was between 90.00-92.00 per cent with relative RSD of 0.062-0.988 per cent in curds and 87.80-92.00 per cent recovery with 0.049-1.003 per cent RSD in soil fortified samples. The results are in agreement with the work of Pal (2011) who observed recovery of 85.00-91.70 per cent for endosulfan and 86.60-92.31 per cent for cypermethrin in capsicum fruits.

The persistence and degradation of endosulfan and cypermethrin in the ready-mix formulation and individual insecticide formulation were studied on cauliflower crop at the recommended rate (RR) 350 g and 50 g ai/ha and at double recommended

Table 1. Recovery of endosulfan and cypermethrin from spiked cauliflower curds and soil samples

Insecticide	Fortification level (mg/kg)	Curds		Soil	
		Mean recovery (%)	Relative standard deviation (%RSD)	Mean recovery (%)	Relative standard deviation (%RSD)
alpha-endosulfan	0.01	90.00	0.967	80.00	0.797
	0.05	88.00	0.878	88.00	0.547
	0.10	89.00	0.343	87.00	0.484
	0.50	86.00	0.128	87.00	0.104
	1.00	90.00	0.045	90.00	0.017
beta-endosulfan	0.01	80.00	0.987	80.00	0.936
	0.05	86.00	0.875	86.00	0.774
	0.10	87.00	0.847	84.00	0.525
	0.50	86.00	0.181	87.00	0.266
	1.00	88.80	0.040	88.80	0.040
Endosulfan sulfate	0.01	80.00	1.004	80.00	0.907
	0.05	84.00	0.990	82.00	0.873
	0.10	90.00	0.949	90.00	0.541
	0.50	88.20	0.113	88.20	0.097
	1.00	93.00	0.048	91.90	0.065
Cypermethrin	0.05	90.00	0.988	90.00	1.003
	0.10	90.00	0.525	88.00	0.933
	0.20	92.00	0.498	90.00	0.704
	0.50	90.00	0.160	87.80	0.104
	1.00	92.00	0.062	92.00	0.049

rate (DRR) 700 g and 100 g ai/ha. The data on decrease in level of residues in individual and combi-insecticides treatments at different days interval are presented in Tables 2 and 3.

Endosulfan initial deposits on cauliflower curds from mixture (Endohyper 40EC) and individual insecticide formulation (Endocel 35 EC) were 3.674-3.740 mg/kg which dissipated to 0.070-0.077 mg/kg on 10th day and 3.640-3.704 mg/kg to 0.065-0.069 mg/kg on 7th day at recommended rate. In double the recommended rate initial deposits of endosulfan from mixture were 6.358-6.405 mg/kg which dissipated to 0.075-0.098 mg/kg in 15 days whereas deposits 6.260-6.374 mg/kg declined to 0.073-0.072 mg/kg from endosulfan alone in 10 days. The comparison of initial deposits obtained in the present study suggests that the persistence of combi-formulation was higher than from its individual formulation. This could be due to additive effect and the other adjuvants present in combi-mix formulation which is supported by Sharma et al (2011) who observed a slightly higher level of initial deposits of flubendiamide in combination with thiacloprid (0.499-0.992 mg/kg) than individual insecticide (0.467-0.824 mg/kg) at 60 and 120 g ai/ha doses in chilli crop. Reddy et al (2007) observed 4.62 mg/kg initial deposits of endosulfan on green chillies sprayed with endosulfan alone @ 350 g ai/ha. Shah et al (1999) reported 7.555 mg/kg endosulfan initial deposits on okra fruits sprayed with 0.05 per cent Decidan 32.8 EC (endosulfan 32% + deltamethrin 0.8%).

Initial deposits of cypermethrin on cauliflower curds from mixture with endosulfan applied @ 50 g ai/ha were 1.007-1.015 mg/kg whereas at double dose the initial deposits were 2.075-2.093 mg/kg. When applied individually cypermethrin initial deposits were 0.629-0.681 mg/kg at recommended rate and 1.139-1.167 mg/kg at double recommended rate. Bhupinder and Udeaan (1989) reported 0.65 mg/kg and 1.43 mg/kg initial deposits at 50 g ai/ha and 100 g ai/ha doses of cypermethrin respectively in okra fruits. There was decline in residues with the time lapse at both the level of application (Table 4).

Insecticides were not directly applied to soil but their residues were detected in the soil on the day of application. Endosulfan residues 0.176-0.197 and 0.301-0.348 mg/kg were detected at the respective dosage of 350 and 700 g ai/ha in combi-mix formulation which became non-detectable on 20th day in single as well as in double recommended rate. When endosulfan was applied individually on the crop then residues in soil also persisted for 20 days (Tables 5 and 6). Kanjana and Kannathasan (2007) reported 0.392, 0.421 and 0.505 mg/kg residues of

Table 2. Persistence of endosulfan (350 g ai/ha) and cypermethrin (50 g ai/ha) on cauliflower curds applied in combi-mix and as individual insecticide

Interval (days)	Isomer/ metabolite	Residue (mg/kg)					
		Combination			Individual		
		Endosulfan		Cypermethrin Residue ± SD	Endosulfan		Cypermethrin Residue ± SD
		Residue ± SD	Total ± SD		Residue ± SD	Total ± SD	
2010							
0	*alpha*	2.501 ± 0.226	3.674 ± 0.362	1.007 ± 0.005	2.507 ± 0.057	3.640 ± 0.057	0.629 ± 0.003
	beta	1.173 ± 0.136			1.133 ± 0.002		
1	*alpha*	1.738 ± 0.102	2.554 ± 0.105	0.820 ± 0.012	1.679 ± 0.009	2.466 ± 0.028	0.405 ± 0.002
	beta	0.816 ± 0.032			0.787 ± 0.035		
3	*alpha*	0.645 ± 0.067	1.041 ± 0.106	0.376 ± 0.005	0.599 ± 0.010	0.857 ± 0.008	0.213 ± 0.009
	beta	0.316 ± 0.047			0.190 ± 0.003		
	ES	0.079 ± 0.017			0.068 ± 0.001		
5	*alpha*	0.305 ± 0.034	0.627 ± 0.052	0.054 ± 0.013	0.099 ± 0.001	0.288 ± 0.008	0.050 ± 0.008
	beta	0.204 ± 0.019			0.083 ± 0.007		
	ES	0.118 ± 0.004			0.106 ± 0.001		
7	*alpha*	0.103 ± 0.004	0.318 ± 0.011	BDL	0.021 ± 0.002	0.065 ± 0.005	BDL
	beta	0.109 ± 0.008			0.015 ± 0.004		
	ES	0.106 ± 0.006			0.029 ± 0.002		
10	*alpha*	0.024 ± 0.006	0.070 ± 0.006		BDL	-	
	beta	0.013 ± 0.003			BDL		
	ES	0.033 ± 0.003			BDL		
15	*alpha*	BDL	-				
	beta	BDL					
	ES	BDL					
2011							
0	*alpha*	2.542 ± 0.003	3.740 ± 0.005	1.015 ± 0.003	2.541 ± 0.002	3.704 ± 0.005	0.681 ± 0.006
	beta	1.198 ± 0.003			1.163 ± 0.003		
1	*alpha*	1.751 ± 0.001	2.598 ± 0.004	0.831 ± 0.004	1.703 ± 0.008	2.505 ± 0.004	0.428 ± 0.004

	beta	0.847 ± 0.004			0.803 ± 0.004		
3	*alpha*	0.700 ± 0.004	1.133 ± 0.003	0.409 ± 0.008	0.631 ± 0.002	0.922 ± 0.019	0.246 ± 0.016
	beta	0.351 ± 0.005			0.210 ± 0.010		
	ES	0.082 ± 0.002			0.081 ± -0.010		
5	*alpha*	0.341 ± 0.003	0.700 ± 0.004	0.053 ± 0.010	0.110 ± 0.002	0.331 ± 0.005	0.050 ± 0.003
	beta	0.220 ± 0.002			0.099 ± 0.001		
	ES	0.139 ± 0.002			0.123 ± 0.004		
7	*alpha*	0.105 ± 0.005	0.318 ± 0.010	BDL	0.021 ± 0.005	0.069 ± 0.003	BDL
	beta	0.107 ± 0.007			0.017 ± 0.002		
	ES	0.106 ± 0.004			0.031 ± 0.001		
10	*alpha*	0.026 ± 0.006	0.077 ± 0.006		BDL		
	beta	0.015 ± 0.002			BDL		
	ES	0.037 ± 0.003			BDL		
15	*alpha*	BDL					
	beta	BDL					
	ES	BDL					

BDL= Below determination limit

Table 3. Persistence of endosulfan (700 g ai/ha) and cypermethrin (100 g ai/ha) on cauliflower curds applied in combi-mix and as individual insecticide

Interval (days)	Isomer/ metabolite	Residue (mg/kg)					
		Combination			Individual		
		Endosulfan		Cypermethrin Residue ± SD	Endosulfan		Cypermethrin Residue ± SD
		Residue ± SD	Total ± SD		Residue ± SD	Total ± SD	
2010							
0	*alpha*	4.248 ± 0.159	6.358 ± 0.169	2.075 ± 0.057	4.328 ± 0.004	6.260 ± 0.201	1.139 ± 0.005
	beta	2.110 ± 0.010			1.931 ± 0.200		
1	*alpha*	2.572 ± 0.182	4.418 ± 0.189	1.468 ± 0.251	2.327 ± 0.015	3.497 ± 0.079	0.782 ± 0.003
	beta	1.845 ± 0.028			1.170 ± 0.094		
3	*alpha*	1.408 ± 0.161	2.290 ± 0.203	0.927 ± 0.003	0.800 ± 0.057	1.348 ± 0.034	0.468 ± 0.019
	beta	0.774 ± 0.062			0.405 ± 0.022		
	ES	0.107 ± 0.009			0.143 ± 0.002		
5	*alpha*	0.512 ± 0.006	1.227 ± 0.008	0.415 ± 0.003	0.168 ± 0.014	0.442 ± 0.010	0.227 ± 0.006
	beta	0.512 ± 0.006			0.098 ± 0.002		
	ES	0.203 ± 0.004			0.175 ± 0.003		
7	*alpha*	0.166 ± 0.043	0.533 ± 0.041	0.056 ± 0.053	0.053 ± 0.030	0.157 ± 0.016	0.054 ± 0.042
	beta	0.176 ± 0.004			0.051 ± 0.004		
	ES	0.191 ± 0.002			0.053 ± 0.012		
10	*alpha*	0.036 ± 0.010	0.134 ± 0.045	BDL	0.026 ± 0.002	0.073 ± 0.002	BDL
	beta	0.053 ± 0.050			0.013 ± 0.001		
	ES	0.046 ± 0.005			0.034 ± 0.003		
15	*alpha*	0.027 ± 0.005	0.075 ± 0.004		BDL	-	
	beta	0.015 ± 0.002			BDL		
	ES	0.034 ± 0.003			BDL		
20	*alpha*	BDL	-				
	beta	BDL					
	ES	BDL					

2011							
0	*alpha*	4.274 ± 0.003	6.405 ± 0.002	2.093 ± 0.003	4.402 ± 0.005	6.374 ± 0.005	1.167 ± 0.005
	beta	2.131 ± 0.003			1.972 ± 0.003		
1	*alpha*	2.591 ± 0.003	4.589 ± 0.005	1.487 ± 0.004	2.410 ± 0.003	3.593 ± 0.002	0.791 ± 0.003
	beta	1.998 ± 0.002			1.182 ± 0.002		
3	*alpha*	1.431 ± 0.008	2.336 ± 0.015	0.975 ± 0.003	0.832 ± 0.001	1.661 ± 0.006	0.483 ± 0.002
	beta	0.791 ± 0.008			0.443 ± 0.004		
	ES	0.114 ± 0.005			0.387 ± 0.005		
5	*alpha*	0.551 ± 0.004	1.304 ± 0.002	0.420 ± 0.007	0.182 ± 0.001	0.488 ± 0.003	0.239 ± 0.012
	beta	0.530 ± 0.003			0.110 ± 0.002		
	ES	0.223 ± 0.005			0.196 ± 0.004		
7	*alpha*	0.172 ± 0.005	0.525 ± 0.009	0.054 ± 0.003	0.064 ± 0.004	0.194 ± 0.003	0.055 ± 0.008
	beta	0.160 ± 0.002			0.061 ± 0.001		
	ES	0.193 ± 0.004			0.068 ± 0.001		
10	*alpha*	0.056 ± 0.004	0.164 ± 0.003	BDL	0.023 ± 0.004	0.072 ± 0.009	BDL
	beta	0.041 ± 0.003			0.014 ± 0.004		
	ES	0.067 ± 0.002			0.035 ± 0.003		
15	*alpha*	0.040 ± 0.006	0.098 ± 0.012		BDL		
	beta	0.016 ± 0.002			BDL		
	ES	0.042 ± 0.006			BDL		
20		BDL	-				
		BDL					
		BDL					

BDL= Below determination limit

Table 4. Degradation kinetics of endosulfan and cypermethrin on cauliflower

Individual/ combination	Dosage	Endosulfan			Cypermethrin		
		Regression equation (y)	r	RL_{50}	Regression equation (y)	r	RL_{50}
2010							
Combination	RR	0.575-0.166X	-0.995	1.81	0.121-0.252X	-0.961	1.19
	DRR	0.750-0.137X	-0.984	2.19	0.429-0.208X	-0.953	1.44
Individual	RR	0.625-0.248X	-0.996	1.21	-0.163-0.212X	-0.983	1.41
	DRR	0.729-0.200X	-0.992	1.50	0.113-0.178X	-0.978	1.68
2011							
Combination	RR	0.589-0.164X	-0.996	1.83	0.132-0.252X	-0.953	1.19
	DRR	0.742-0.130X	-0.981	2.32	0.440-0.210X	-0.949	1.43
Individual	RR	0.636-0.243X	-0.993	1.23	-0.124-0.217X	-0.973	1.38
	DRR	0.766-0.199X	-0.996	1.51	0.123-0.178X	-0.975	1.69

RR= Recommended rate, DRR= Double recommended rate, r= Correlation, RL_{50}= Residue half-life

endosulfan in soil when applied at 0.035, 0.070 and 0.140 per cent concentrations respectively on tomato crop.

Cypermethrin residues were not detected on 0 day in soil at recommended rate (50 g ai/ha) but in double recommended rate (100 g ai/ha) residues were observed 0.081-0.087 mg/kg in combi-mix formulation treatment. However in individually applied cypermethrin its residues were detected in soil on 0 day and became below detection limit in 10 days. Gupta et al (2011) observed cypermethrin residues below detection limit in soil samples after the application of Roket 44 EC @ 1 and 2 l/ha on tomato crop.

In an effort to compare the persistence of endosulfan and cypermethrin in pre-mix formulation Endohyper 40 EC (endosulfan 35% + cypermethrin 5%) and in individual insecticides endosulfan formulation (Endocel 35 EC) and cypermethrin (Challenger 25 EC) when applied at recommended rate and at double recommended rate in cauliflower curds and soil it was observed that the pre-mix formulation showed slightly higher persistence than from its individual

Table 5. Residues of endosulfan (350 g ai/ha) and cypermethrin (50 g ai/ha) in cauliflower cropped soil

Interval (days)	Isomer/ metabolite	Residue (mg/kg)					
		Combination			Individual		
		Endosulfan		Cypermethrin Residue ± SD	Endosulfan		Cypermethrin Residue ± SD
		Residue ± SD	Total ± SD		Residue ± SD	Total ± SD	
2010							
0	*alpha*	0.117 ± 0.002	0.176 ± 0.004	BDL	0.234 ± 0.007	0.333 ± 0.007	0.077 ± 0.007
	beta	0.060 ± 0.006			0.100 ± 0.002		
10	*alpha*	0.018 ± 0.005	0.047 ± 0.006		0.070 ± 0.004	0.155 ± 0.005	BDL
	beta	0.013 ± 0.003			0.032 ± 0.005		
	ES	0.016 ± 0.004			0.053 ± 0.004		
20	*alpha*	BDL	-		0.017 ± 0.006	0.043 ± 0.009	
	beta	BDL			0.012 ± 0.002		
	ES	BDL			0.014 ± 0.002		
30	*alpha*				BDL	-	
	beta				BDL		
	ES				BDL		
2011							
0	*alpha*	0.132 ± 0.006	0.197 ± 0.006	BDL	0.255 ± 0.024	0.364 ± 0.026	0.080 ± 0.007
	beta	0.065 ± 0.002			0.109 ± 0.004		
10	*alpha*	0.019 ± 0.003	0.046 ± 0.005		0.076 ± 0.003	0.169 ± 0.007	BDL
	beta	0.011 ± 0.002			0.033 ± 0.006		
	ES	0.016 ± 0.002			0.061 ± 0.002		
20	*alpha*	BDL	-		0.018 ± 0.002	0.043 ± 0.002	
	beta	BDL			0.011 ± 0.002		
	ES	BDL			0.014 ± 0.003		
30	*alpha*				BDL	-	
	beta				BDL		
	ES				BDL		

BDL= Below determination limit

Table 6. Residues of endosulfan (700 g ai/ha) and cypermethrin (100 g ai/ha) in cauliflower cropped soil

Interval (days)	Isomer/ metabolite	Residue (mg/kg)					
		Combination			Individual		
		Endosulfan		Cypermethrin Residue ± SD	Endosulfan		Cypermethrin Residue ± SD
		Residue ± SD	Total ± SD		Residue ± SD	Total ± SD	
2010							
0	*alpha*	0.202 ± 0.004	0.301 ± 0.006	0.081 ± 0.002	0.388 ± 0.009	0.542 ± 0.015	0.096 ± 0.003
	beta	0.100 ± 0.002			0.154 ± 0.006		
10	*alpha*	0.020 ± 0.003	0.053 ± 0.001	BDL	0.089 ± 0.006	0.210 ± 0.010	BDL
	beta	0.014 ± 0.002			0.038 ± 0.006		
	ES	0.018 ± 0.002			0.083 ± 0.002		
20	*alpha*	BDL	-		0.020 ± 0.002	0.051 ± 0.009	
	beta	BDL			0.013 ± 0.002		
	ES				0.018 ± 0.006		
30	*alpha*				BDL		
	beta				BDL		
	ES				BDL		
2011							
0	*alpha*	0.241 ± 0.007	0.348 ± 0.004	0.087 ± 0.002	0.411 ± 0.004	0.602 ± 0.014	0.105 ± 0.004
	beta	0.106 ± 0.004			0.191 ± 0.013		
10	*alpha*	0.023 ± 0.007	0.057 ± 0.008	BDL	0.092 ± 0.001	0.219 ± 0.004	BDL
	beta	0.014 ± 0.002			0.044 ± 0.002		
	ES	0.021 ± 0.002			0.083 ± 0.004		
20	*alpha*	BDL	-		0.019 ± 0.001	0.046 ± 0.001	
	beta	BDL			0.012 ± 0.003		
	ES	BDL			0.015 ± 0.003		
30	*alpha*				BDL	-	
	beta				BDL		
	ES				BDL		

BDL= Below determination limit

insecticide formulation which is supported by Dharumarajan et al (2009).

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