INTERNATIONAL JOURNAL OF PLANT, ANIMAL AND ENVIRONMENTAL SCIENCES
IJPAES
Volume-7, Issue-1 Jan-Mar-2017 Coden: IJPAJX-CAS-USA, Copyrights@2015ISSN-2231-4490
Received: 7th Jan-2017 Revised: 25th Jan-2017 Accepted: 25th Jan-2017
DOI: 10.21276/Ijpaes http://dx.doi.org/10.21276/ijpaes Research Article

STUDIES ON THE FATE OF SWING – A READY-MIX FUNGICIDE (EPOXICONAZOLE 12.5% SC & CARBENDAZIM 50 WP) IN PADDY FIELD

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ABSTRACT: To find out the residual fate and dissipation pattern of swing on paddy field under Indian subcontinent, it was applied twice recommended dose at 750 g/ha [187.5 g a.i./ha (93.5 g epoxiconazole + 93.5 g carbendazim)] (T_1) and double the recommended doses at 1500 g/ha [375 g a.i./ha (187.5 g epoxiconazole + 187.5 g carbendazim)] (T_2) along with untreated control (T_3) during pre-monsoon and post monsoon 2004. The field cropped soil and water samples were collected at different day's interval. At harvest, field cropped soil, grain, husk and straw samples were collected. The half-life values were found in the range between 4.5 to 5.76 days for epxicanazole and 18.47 to 23.16 days for carbendazim in paddy cropped soil irrespective of dose and season. This ready-mix fungicide could safely be recommended for application as no residues were detected in the harvest samples, i.e., cropped soil, grain, husk and straw samples.

***Key words*:** Swing, Ready-mix, Epoxiconazole, Carbendazim, Dissipation, Persistence, Half-life, Paddy

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INTRODUCTION

India is one of the largest producers and exporter of rice (*Oryza sativa*) and accounting for 20% of the world rice production. Rice is India's preeminent crops and is the staple food of eastern and southern parts of India. In 2010-11 crop year, India produced 100 million tonnes of rice while in 2011-12 crop year, India's rice production reached to a record high of 104.32 million tonnes. In every year, most of the yields are losses due to various fungal diseases of rice. Sheath blight caused by *Rhizoctonia solani* Kuhn is one of the most important fungal disease of rice during kharif season in almost all rice cultivated areas of India [1, 2]. In some favourable conditions the loss may be upto 50% of the total yield [3,4]. For control of fungal disease, many fungicides are available in market but for better control of fungal disease ready-mix formulation are now available in Indian market.

Swing, a ready-mix formulation, consists of two fungicides, i.e., epoxiconazol 12.5% SC and carbendazim 50 WP. The ready-mix formulation of epoxiconazole [(2RS,3SR)-1-[3-(2-chlorophenyl)-2-(4-fluorophenyl)-oxiran-2-ylmethyl]-1H-1,2,4-triazole; (Mol. Wt. 329.76; m.p. 136.2°C); (Fig.1)] and carbendazim [Methyl benfimidazole-2-yl carbamate; (Mol. Wt. 191.2; m.p. 302-307°C); (Fig.2)] is recently introduced by M/s. BASF. India Ltd., Mumbai, in the Indian-subcontinent, for the control of sheath blight (*Rhizoctonia solani*) of rice mainly and diseases of different crop.

It is highly effective against *Rhizoctonia solani* , *Fussarium* and *Erysiphe* of cereal crpos. Although some information is available on the residual fate of carbendazim in rice tissues [5], bettle leaves [6, 7], fresh and dehydrated brinjal [8], Grapes [9], mango [10] and little information is available of epoxiconazole in soil [11]. Very few information is available about its persistence behaviour in rice. Persistence behaviour of swing is, therefore, an important aspect in order to generate meaningful data from the point of view of plant protection, public health and environmental safety as it is the newly introduced ready-mix fungicide. Because the wide spread use, pesticides have proliferated intensely in the biosphere and thus significantly contribute to the problem of environmental pollution. Persistent and toxic pesticide residues prevailing in the environment ultimately entered into the food chain and retained in the biosphere of the planet earth in some cases. These toxic pesticides and their degraded products have the potentialities of polluting our agro-ecosystem including ground water. Hence, the present study has been initiated to find out the residue and dissipation pattern of Swing, i.e., mixture of epoxiconazole and carbendazim in paddy cropped soil, water, grain, husk and straw and to observe the persistence behaviour and half-life of epoxiconazole and carbendazim under West Bengal agro-climatic condition.

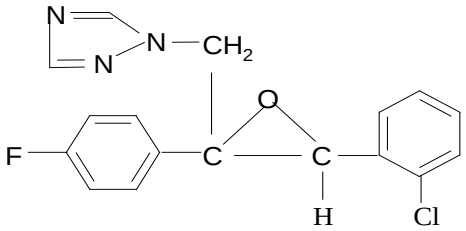

Fig.1. Epoxiconazole

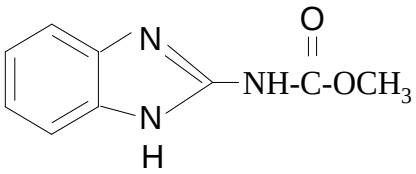

Fig.2. Carbendazim

MATERIALS AND METHODS

A field trial was conducted at Regional Research Station, Bidhan Chandra Krishi Viswavidhyalaya, Chakdah, Nadia, West Bengal, India, located at 23º5.3′ N latitude and 83º5.3′ E longitude and at an elevation of 9.5m from mean sea level for two consecutive seasons (pre-monsoon, 2004 and post monsoon, 2004) on paddy [variety – (i)"Khitish" (IET – 4090): pre-monsoon 2004 (ii) MTU 7029 (Swarna mashuri): post monsoon 2004]. The experimental site was clay loam in soil texture with pH 6.92, organic carbon 0.76%, available N 222.6 kg/ha, P_2O_5 24.3 kg/ha and K_2O 189.7 kg/ha. Swing, a ready-mix formulation was applied twice recommended dose @ 0.75 kg/ha [187.5 g a.i./ha (93.5 g epoxiconazole + 93.5 g carbendazim)] (T_1) and double the recommended doses @ 1.50 kg/ha [375 g a.i./ha (187.5 g epoxiconazole + 187.5 g carbendazim)] (T_2) along with untreated control (T_3) with the help of high volume knapsack sprayer on the paddy field @ 400 L water/ha. Each treatment including control was replicated thrice in a randomly block designed (RBD) with plot size 5m × 4m. The residual fate of the ready-mix formulation, swing, was studied on the substrates, i.e., field soil, field water, grain and straw. For dissipation study, field soil samples were taken at 0 (2 h after application), 5, 10, 30 d after application and field water samples were collected at 0 (2 h after application), 5, 10, 30 d interval. Cropped soil, grain, husk and straw samples were collected at harvest. All the reagents used were of analytical reagent grade and all the solvents were redistilled before use. Water used was double glass distilled.

The samples were extracted immediately after collection, if this was not possible, the substrates were stored at –18^{0}C. The employed analytical methods for determination of epoxiconazole and carbendazim residue were validated at laboratory. For extraction of epoxiconazole and carbendazim residue, samples were taken for analysis from each treatment after quartering.

Extraction of epoxiconazole residues

Soil samples was collected at a depth of 0-15 cm with the help of soil auger and representative amount of samples (100 g) was kept in a conical flask with 200 ml mixture of methanol:water (8:2; v/v). Then it was shaken vigorously for 2 h in a mechanical shaker and celite was then added and filtered through Buchner funnel using Whatman No.1 filter paper followed by washing with 150 mL of same solvent mixture. The filtrate was reduced in a rotary vacuum evaporator at 40^{0}C and 5.0 g $Ca(OH)_2$ was added, stirred for 15 mints and allowed to stand for precipitation and then filtered with same solvent mixture. From the filtrate, methanol was evaporated in a rotary vacuum evaporator at 40^{0}C and transferred to a separatory funnel by 100 mL distilled water. Then it was partitioned thrice with (100+50+50) mL hexane and combined organic layer was collected through anhydrous sodium sulphate. The organic layer was concentrated in a rotary vacuum evaporator at 40^{0}C and transferred to a column packed with 10 g silica gel (60-100 mesh, Qualigen). Column was first eluted with 40 mL mixture of hexane:acetone (93:7; v/v) and discarded. Finally, the column was eluted with 50 mL mixture of hexane:acetone (8:2; v/v) and the solvent mixture was evaporated to dryness. The concentrated extract was reconstituted with mixture of hexane:acetone (9:1; v/v) for GLC analysis.

Field water sample (500 mL) was filtered through Buchner funnel using Whatman No.1 filter paper and was taken in a separatory funnel and partitioned thrice with (100+50+50) mL hexane and proceed further following the same procedure as described above.
Representative amount of grain, husk and straw (25 g each) samples were dissolved with 200 mL mixture of methanol:water (8;2; v/v) for overnight and blended in a Remi automix blender for 2 min and filtered through buchner funnel using 100 mL methanol:water (8:2; v/v) as washing solvent. Then the similar steps were followed as described above.

Extraction of carbendazim residues

Representative amount of samples (100 g) was kept in a conical flask with 200 mL ethyl acetate. Then it was shaken vigorously for 2 h in a mechanical shaker and filtered through Buchner funnel using Whatman No.1 filter paper followed by washing with 100 mL ethyl acetate. The filtrate was concentrated in a rotary vacuum evaporator at 40^0C and transferred into a separating funnel with addition of 25 mL 0.1 (N) HCl. After partitioning the acidic fraction was collected and was again partitioning with (50 + 50) mL n-hexane by using separating funnel. The hexane layer was discarded and the acidic fraction was collected and was adjusted with 0.1(N) NaOH to pH 6.5. Then the resulted aqueous phase was partitioned with (50 + 50) mL ethyl acetate and aqueous phase was discarded. The ethyl acetate fraction was collected through anhydrous sodium sulphate and concentrated by the rotary vacuum evaporator at 40^0C. After that, the concentrated ethyl acetate fraction was partitioned with 10 ml of 0.1(N) HCl. The aqueous portion was collected and was ready for spectrophotometric analysis.
Field water sample (500 ml) was filtered through Buchner funnel using Whatman No.1 filter paper and was taken in a separatory funnel and partitioned thrice with (100+50+50) mL ethyl acetate and proceed further following the same procedure as described above.
Representative amount of grain, husk and straw (25 g each) samples were dissolved with 100 mL ethyl acetate for overnight and blended in a Remi automix blender for 2 min and filtered through buchner funnel using 100 mL ethyl acetate as washing solvent. Then the similar steps were followed as described above.

Analysis

The residues of epoxiconazole were analyzed by Gas Liquid Chromatography (Hewlett-Packard, Model 5890) equipped with ^{63}Ni electron capture detector was coupled to a data processor (Chemito 5000; supplied by Chemito Instruments, Nasik, Maharastra, India). The column used for analysis of epoxiconazole was a DB-5 fused silica megabore (J&W, USA), 30 m × 0.53 mm i.d., 1.5 µm film thickness. The operating mode is split less. The operating parameters of the gas chromatograph were; oven temperature: 250^0C, injector temperature: 270^0C, detector temperature: 300^0C, flow rate of carrier gas ((N_2): 50 mL/min, retention time: 3.1 min, limit of quantification (LOQ): 0.05 ppm, limit of detection (LOD): 0.01 ppm.
The residues of carbendazim were analysed by UV-VIS spectrophotometer (Model DU-6, Beckman) at wavelength (λ_{max}) 281 nm and 0.1 (M) HCl was used as reference. Limit of quantification (LOQ) and limit of detection (LOD) were 0.1 ppm and 0.01 ppm respectively.
A stock solution of 1 ppm of epoxiconazole (analytical grade, 99.9%; supplied by M/s. BASF. India Ltd., Mumbai) and carbendazim (analytical grade, 99.9%; supplied by M/s. BASF. India Ltd., Mumbai) was prepared in hexane and acetone respectively and used as an external standard. One microliter each of 1 ppm of analytical grade epoxiconazole and carbendazim was iniected into the gas chromatograph and spectrophotometer respectively using the above mentioned parameters. The retention time of epoxiconazole in different samples were compared with external standards and for carbendazim, wavelength in different samples compared with external standards. In both the cases data were recorded.
The recovery experiment was carried out by fortifying the different substrate with 0.5, 1.0 and 5.0 ppm epoxiconazole and carbendazim respectively. Average recoveries of epoxiconazole in cropped soil, water, grain, straw and husk were found in the range of 85.0% - 89.6%, 86.0 – 90.8%, 85.5 – 89.8%, 84.0 – 93.0% and 87.2 – 92.0% respectively and in the case of carbendazim the range were 83.5 – 87.0%, 83.0 – 86.6%, 84.2 – 90.0%, 86.0 – 89.2% and 86.2 – 94.2% respectively. The obtained dissipation data were subjected to regression analysis [12] for computing residual half–lives [13].

RESULTS AND DISCUSSIONS

The residue data of epoxiconazole and carbendazim in paddy cropped soil and water at different day's intervals and the residue data in cropped soil, grain, husk and straw at harvest were presented in the Tables 1-6. The corresponding dissipation rates, regression equations and half-life values have been calculated on the basis of residue data. It has been observed from the results that the residues of epoxiconazole and carbendazim in paddy cropped soil and water declined progressively with time irrespective of any dose and season.

The initial deposite of epoxiconazole after 2 h of spraying in field soil and water was found in the range of 0.14 – 0.30 ppm and 0.06 – 0.15ppm irrespective of any dose and season whereas in case of carbendazim, the corresponding values were 0.48 – 1.05 ppm and 0.12 – 0.17ppm respectively. Afterwards, there is a sharp decline in epoxiconazole and carbendazim for both the doses. In case of soil samples, after 30 d application, more than 60% of epoxiconazole was dissipated whereas in case of carbendazim, residue was below detectable limit. In field water samples, after 10 d of application, residues of epoxiconazole and carbendazim were below detectable limit. No residue was detected in untreated control samples throughout the entire study. The dissipation rate of epoxiconazole and carbendazim followed first order kinetics irrespective of dose and season. The calculated half-life values ($T_{1/2}$) were found to be in the range of 4.50 – 5.76 d for epoxiconazole and 18.47 – 23.16 d for carbendazim in field soil samples irrespective of dose and season. In the present study, the persistence of carbendazim is also comparable with earlier reported persistence data of carbendazim in rice tissues [5]. In harvest samples, no residues of epoxiconazole and carbendazim were detected in the cropped soil, grain, husk and straw samples.

The MRL value of epoxiconazole in rice has not established by the Codex Alimentarus Commission where as in case of carbendazim the MRL value in rice straw and fodder, dry was 15 ppm and in rice, husked was 2 ppm [14].

Table-1: Persistence of Epoxiconazole in cropped soil for consecutive two seasons at recommended and double the recommended dose

Season	DAT	Recommended dose			Double the recommended dose		
		Residue in ppm (M ± S.D.)	Dissipation (%)	Regression equation (Half life (d))	Residue in ppm (M ± S.D.)	Dissipation (%)	Regression equation (Half life (d))
Pre-monsoon (2004)	0	0.16 ± 0.03	-	Y= 1.35 – 0.0593X (5.08)	0.30 ± 0.04	-	Y= 1.44 – 0.0523X (5.76)
	5	0.05 ± 0.02	64.29		0.13 ± 0.03	56.67	
	10	0.03 ± 0.01	78.57		0.09 ± 0.01	70.00	
	30	BDL	-		BDL	-	
Post Monsoon (2004)	0	0.14 ± 0.01	-	Y= 1.14 – 0.0669X (4.50)	0.27 ± 0.06	-	Y= 1.41 - 0.0528X (5.70)
	1	0.06 ± 0.03	57.14		0.13 ± 0.05	51.85	
	3	0.03 ± 0.02	78.57		0.08 ± 0.01	70.37	
	7	BDL	-		BDL	-	

Table-2: Persistence of Carbendazim in cropped soil for consecutive two seasons at recommended and double the recommended dose

Season	DAT	Recommended dose			Double the recommended dose		
		Residue in ppm (M ± S.D.)	Dissipation (%)	Regression equation (Half life (d))	Residue in ppm (M ± S.D.)	Dissipation (%)	Regression equation (Half life (d))
Pre-monsoon (2004)	0	0.48 ± 0.03	-	Y= 1.64 – 0.0160X (18.81)	0.71 ± 0.06	-	Y= 1.83 – 0.0145X (20.76)
	5	0.34 ± 0.02	29.17		0.54 ± 0.03	23.94	
	10	0.29 ± 0.01	39.58		0.48 ± 0.06	32.39	
	30	0.15 ± 0.02	68.75		0.25 ± 0.02	64.79	
Post Monsoon (2004)	0	0.71 ± 0.05	-	Y= 1.80 – 0.0163X (18.47)	1.05 ± 0.03	-	Y= 1.98 - 0.0130X (23.16)
	5	0.46 ± 0.04	35.21		0.76 ± 0.15	27.62	
	10	0.41 ± 0.03	42.25		0.68 ± 0.06	35.24	
	30	0.21 ± 0.06	70.42		0.40 ± 0.03	61.90	

Table-3: Persistence of Epoxiconazole in cropped water for consecutive two seasons at recommended and double the recommended dose

Season	DAT	Recommended dose			Double the recommended dose		
		Residue in ppm (M ± S.D.)	Dissipation (%)	Regression equation (Half life (d))	Residue in ppm(M ± S.D)	Dissipation (%)	Regression equation (Half life (d))
Pre-monsoon (2004)	0	0.06 ± 0.01	-	-	0.12 ± 0.03	-	-
	5	0.02 ± 0.01	66.67		0.05 ± 0.01	58.33	
	10	BDL	-		BDL	-	
	30	BDL	-		BDL	-	
Post Monsoon (2004)	0	0.08 ± 0.03	-	-	0.15 ± 0.06	-	-
	5	0.03 ± 0.01	62.50		0.08 ± 0.02	46.67	
	10	BDL	-		BDL	-	
	30	BDL	-		BDL	-	

Table-4: Persistence of Carbendazim in cropped water for consecutive two seasons at recommended and double the recommended dose

Season	DAT	Recommended dose			Double the recommended dose		
		Residue in ppm(M ± S.D.)	Dissipation (%)	Regression equation (Half life (d))	Residue in ppm(M ± S.D)	Dissipation (%)	Regression equation (Half life (d))
Pre-monsoon (2004)	0	0.12 ± 0.03	-	-	0.17 ± 0.01	-	-
	5	0.05 ± 0.01	58.33		0.08 ± 0.01	52.94	
	10	BDL	-		BDL	-	
	30	BDL	-		BDL	-	
Post Monsoon (2004)	0	0.15 ± 0.02	-	-	0.16 ± 0.02	-	-
	5	0.06 ± 0.01	60.00		0.07 ± 0.01	56.25	
	10	BDL	-		BDL	-	
	30	BDL	-		BDL	-	

Table-5: Harvest residue of Epoxiconazole in cropped soil, grain, husk and straw samples for consecutive two seasons at recommended and double the recommended dose

Season	Recommended dose				Double the recommended dose			
	Residue in ppm				Residue in ppm			
	Soil	Grain	Husk	Straw	Soil	Grain	Husk	Straw
Pre-monsoon (2004)	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Post Monsoon (2004)	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL

Table-6: Harvest residue of Carbendazim in cropped soil, grain, husk and straw samples for consecutive two seasons at recommended and double the recommended dose

Season	Recommended dose				Double the recommended dose			
	Residue in ppm				Residue in ppm			
	Soil	Grain	Husk	Straw	Soil	Grain	Husk	Straw
Pre-monsoon (2004)	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Post Monsoon (2004)	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL

a.i. = active ingredient, DAT = Day's after treatment, M = Mean of three replicates, BDL = Below detectable level

CONCLUSION

The study clearly shows that epoxiconazole 12.5% SC and carbendazim 50 WP has moderate persistence in soil and water. Interestingly, it was found that no residue was detected in the harvest samples, it might be stated that the ready-mix formulation of epoxiconazole and carbendazim may not pose any residual toxicity problems in rice production ecosystem and might be safely consumed. From the present investigation it was conclude that the ready-mix formulation of Swing (mixure of epoxiconazole 12.5% SC and carbendazim50 WP) is safe to use in paddy field.

ACKNOWLEDGEMENT

The authors are grateful to the Department of Agricultural Chemicals, Bidhan Chandra Krishi Viswavidyalaya, India for providing the necessary instrumental facilities.

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INTERNATIONAL JOURNAL OF PLANT, ANIMAL AND ENVIRONMENTAL SCIENCES

ISSN 2231-4490

International Journal of Plant, Animal and Environmental Sciences