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Harvest Time Residues of Carbosulfan 6G in Rice Grain, Straw, Husk, Bran and Soil

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A study was conducted to determine the harvest time residues of carbosulfan 6G (NS) in rice grain, straw, husk, bran and soil. Carbosulfon 6G was applied @ 1000 (NS), 2000 (NS), 1000 (ES) and 2000 (ES) g a.i./ha twice *i.e.,* at 21 and 45 days after transplanting in two seasons during *rabi* 2011 and *kharif* 2012 in rice. Rice grain, straw, husk, bran, and soil were analysed for harvest time residues by using HPLC/GC through liquid chromatography. It was found that residues were below detectable level (BDL) in the matrices tested irrespective of the rate of application.

Key words: Carbosulfan, Rice, Soil, Residues.

Rice is a staple food crop and susceptible to pest attack at every stage of its growth. During the early stage, the crop is highly vulnerable to insect pests like thrips, brown planthopper, white backed planthoppers, green leafhopper and stem borer. The effect of these pests poses serious threat to rice cultivation and affect the crop stand and yield adversely. As there is no single tolerant variety of rice available in the country, it is essential to depend more on chemicals to protect the crop (Rajeswaran *et. al.,* 2005). Experiments conducted in India and abroad have revealed the potential of carbosulfan 6G in suppressing the pest complex in different crops including sucking and chewing pests of rice. So, a detailed study was conducted to determine the terminal residues of carbosulfan in rice grain, straw, husk, bran and soil following its repeated applications (2 times) in two seasons on rice crop.

Materials and Methods

Supervised field trials were laid out in randomized block design with three replications at paddy breeding station (PBS) Tamil Nadu Agricultural University Coimbatore during November 2012 and August 2013 with the rice cultivar, Co- 51 to evaluate the bioefficacy of carbosulfan 6G (NS) against rice pests, and determination of terminal residues in rice ecosystem. The plot size adopted was 40 m^2.

Samples were collected at harvest time after last application from each replicate of four treatments applied with carbosulfan 6G (NS & ES) at the rate of 1000 and 2000 g a.i./ha. Control samples were collected similarly from the untreated plots. The samples collected from three replications for each treatment were used for residue analysis. Soil samples were collected at 10 days after application using auger driven to a depth of 15 cm. A minimum of 10 cores were taken across the field and bulked together, from which a single representative sample of 100 g was taken by quartering technique. From this, a sub sample of 25 g of soil was taken for residue studies. Reagents acetonitrile with HPLC grade, methanol with HPLC grade, anhydrous magnesium sulphate, sodium chloride, Tri- sodium citrate dehydrate, disodium citrate sesquihydrate, (reagent grade), primary secondary amines (PSA) and 5 per cent formic acid were used for the residue analysis.

Extraction was done in a representative sample of 10 g each from rice grain, straw, bran, husk, and soil. Samples were taken and blended with 50 ml acetonitrile for 2 min at high speed by using 50 ml centrifuge tube, shaken vigorously for 1 min and 4g magnisum sulphate unhydrous, 1g sodium chloride, 1g tri- sodium citrate dehydrate, and 0.5g di sodium citrate sesquihydrate were added and each tube, shaken directly after the salt addition shortly, then shaken vigorously for 1 min, with phase separation, then centrifuged for 5 min at 3000 rpm. Subsequently X ml of the extract was transferred into a single use PP centrifugation tube, which contained X*25mg primary secondary amines (PSA) and X*150mg anhydrous megnisum sulphate (MgSO4), then shaken well for 30 sec, again centrifuged for 5 min at 3000 rpm. Then Y ml of the extract was transferred into screw cup vial, and acidified with Y*10 micro liter 5 per cent formic acid in acetonitrile (10 micro liter/ml exract) then cleaned and acidified extract was transferred into auto sampler vials and used for the residue determination by GC or HPLC techniques.

The reference standard carbosulfan received

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from FMC India Ltd. was used for the preparation of stock solution, spiking and quantification of residue in the sample matrices. From the technical standard of 93.36 per cent purity, 107 mg was weighed and transferred to a 100 ml volumetric flask with methanol (HPLC grade) and the volume was made up. Then the flask was shaken well to get a homogenous solution of 1000 ppm and was stored in refrigerator at 4^0C.

The concentrated stock solution was brought to room temperature and one ml from the concentrated stock solution was transferred to a 100 ml volumetric flask. The volume was made up and the flask was shaken well to obtain homogenous solution of 10 ppm standard solution. This was utilized for spiking the samples for recovery studies.

From the intermediate stock solution, working standards of 0.05, 0.01 to 0.5 ppm were prepared by diluting one ml of 10 ppm solution to 10 – 100 times. These working standards were derivatised with methonal (HPLC grade) to find out the retention time and for quantitative determination of residues in samples.

Carbosulfan residues were estimated by Cyper lab LC100 model HPLC equipped with UV detector fitted with c-18 column. The following were the operating parameters.

Column : c-18 column

Temperature : Ambient temp. (40^0C)

Detector : UV

Wavelength : 269 nm

Mobile phase : Time (min) Water (%) Methanol (%)

0 50 50

Flow rate : 1.0 ml/min.

Total run time : 10 min

The final quantification was worked out using the formula

$$\text{Residues} = \frac{A_s W_{std} V_s}{A_{std} W_s A_{sj}}$$

where,

$A_{s\,-}$ Peak area of the sample

$A_{std\,-}$ Peak area of the standard

$W_{std\,-}$ Weight of the standard in ng

Ws - Weight of the sample in g

Vs - Volume of the sample (final extract in ml)

Asj - Aliquot of the sample injected in ml

The recovery studies using control samples of rice grain, straw, husk, bran and soil were conducted by fortifying with known quantities of carbosulfan at three concentrations *viz.,* 0.05, 0.01 and 0.5 ppm to find out the correctness of analytical procedure.

Results and Discussion

Fortification of control samples revealed that, the recoveries ranged between 80.65 to 87.65 % for rice grain, 79.71 to 88.42 % for husk, and 80.90 to 83.10 % for bran, 82.52 to 90.98 for straw, and 85.52 to 88.94 for soil with an average recovery of 84.60, 84.43, 81.79, 86.48 and 87.48%, respectively (Table 1).

Table 1. Recovery studies of carbosulfan 6G (NS) in rice grain, husk, bran, straw and soil

Substrate	Quantity fortified (ppm)	Recovery percentage (%)	Mean recovery percentage (%)
Rice grain	0.01	85.52	84.60
	0.05	80.65	
	0.5	87.62	
Husk	0.01	85.18	84.43
	0.05	79.71	
	0.5	88.42	
Bran	0.01	81.38	81.79
	0.05	83.10	
	0.5	80.90	
Straw	0.01	85.94	86.48
	0.05	90.98	
	0.5	82.52	
Soil	0.01	88.94	87.48
	0.05	87.98	
	0.5	85.52	

The results on the determination of carbosulfan 6G residues in rice grain, straw, husk, bran and soil from the harvested samples revealed that the residues were below detectable level (BDL) in all the matrices irrespective of the rate of application (Table 2). In the present study, the time lapse between final application and harvest was 90 days. The observed BDL at harvest might be due to sufficient time interval between applications and harvest which allowed degradation of carbosulfan. Moreover, the grains were not fully mature when the final insecticide application was given, which might have prevented the direct contamination of straw, husk and bran with carbosulfan.

The results are in agreement with findings of Mukerjee and Gopal (2001), who reported that imidacloprid, a systemic insecticide at 6, 9, 12, 18 and 24 mg kg^{-1} seed treatment left residue of below detectable level (<0.05 mg kg^{-1}) in cotton seed at harvest. George *et. al.,* (2002) reported that contamination of seeds with lindane and deltamethrin was the least when the bolls are in closed condition and maximum (10-18 times higher) when bolls are partially or fully opened. Carbosulfan residue analysed for different doses *viz.,* 250, 500 and 1000 g a.i.ha^{-1} were below

Table 2. Harvest time residue of carbosulfan 6G in rice grain, straw, husk, bran and soil

Treatment	Residue in (mg kg^{1})				
I season harvest	Rice	straw	Husk	Bran	soil
Carbosulfan 6G (NS) 1000 g a.i. ha^{-1}	BDL	BDL	BDL	BDL	BDL
Carbosulfan 6G (NS) 2000 g a.i. ha^{-1}	BDL	BDL	BDL	BDL	BDL
Carbosulfan 6 G (ES) 1000 g a.i. ha^{-1}	BDL	BDL	BDL	BDL	BDL
Carbosulfan 6 G (ES) 2000 g a.i. ha^{-1}	BDL	BDL	BDL	BDL	BDL
II season harvest					
Carbosulfan 6G (NS) 1000 g a.i. ha^{-1}	BDL	BDL	BDL	BDL	BDL
Carbosulfan 6G (NS) 2000 g a.i. ha^{-1}	BDL	BDL	BDL	BDL	BDL
Carbosulfan 6 G (ES) 1000 g a.i. ha^{-1}	BDL	BDL	BDL	BDL	BDL
Carbosulfan 6 G (ES)) 2000 g a.i. ha^{-1}	BDL	BDL	BDL	BDL	BDL

NS- New Source, ES- Existing Source, BDL- Below Detectable Level

detectable limit in the brinjal fruit sampled for residues at first and third harvest (Sheeba Jasmine, 2002). Bhandari and Ujagir (2004) observed no residue of three insecticides used in leaves and pods of pigeon pea after third spray. Suman *et al*. (2007) observed that carbosulfan 25 EC at 187.5 and 375 g a.i. ha $^{-1}$was below detectable limit at 7 days after spraying in brinjal. Suganthi (2012) noticed that, carbosulfan 25 EC 250 and 500 g a.i. ha $^{-1}$was below detectable level in the chilli fruits and soil at harvest.

The result of the terminal residue analysis of the soil samples collected from the respective plots received different doses of carbosulfan 6G *viz.,*1000 (NS), 2000 (NS), 1000 (ES), and 2000 (ES) g a.i/ha, revealed no detectable residue (Table 2). Similar results were reported by Prasad and Singh (2004), who analysed the harvest time residue of pyrethroids in chickpea leaves till harvest after seed treatment carried out about six months earlier; Sanyal and Kulshrestha (2004). At harvest time carbosulfan 25 (DS and ES) at 15 and 30 g a.i/ha, recorded no terminal residue in cotton lint, seed, oil and soil (Abinaya 2012). In the present study, degradation of carbosulfan 6G in soil might have occurred faster and hence, the residue was not detected during the harvest time residue estimation.

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