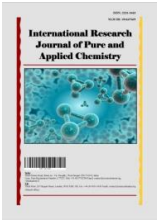

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Dissipation Kinetics of Carbofuran in the Soil and Its Residues in Sugarcane

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Authors' contributions

This work was carried out in collaboration among all authors. This work is a part of Doctoral research of author LKS under the guidance of authors KGP and SS. Authors KDG and VHS assisted in chemical analysis, data interpretation and draft correction. All authors read and approved the final manuscript.

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ABSTRACT

A field experiment was conducted to determine the dissipation kinetics of carbofuran and its metabolite (Carbofuran 3-OH) in the soil and their terminal residue in sugarcane. The experimental plots were subjected to application of carbofuran 3G (1.0 kg a.i./ha) at the time of planting and 60 days after planting of sugarcane variety CoN 07072 grown with recommended agronomic practices. The soil samples were periodically collected at 0 (2 hrs.), 1, 3, 5, 10, 20, 30, 60 and 90 days after last application of carbofuran. QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) based extraction method adopted to quantify the residues of carbofuran on LC-MS/MS (Liquid Chromatography-Tandem Mass Spectrometry) from soil, sugarcane juice and leaves were found accurate, sensitive and precise enough. Residues of carbofuran and total carbofuran (Carbofuran + Carbofuran 3-OH) were observed upto 30 days after application and reached below quantification level at 60 days after application in sugarcane grown soil. Carbofuran and total carbofuran followed the first order dissipation kinetics

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with the half life of 4.18 and 4.17 days, respectively in the experimental soil. Sugarcane leaves and juice contains the residues of carbofuran below quantification limit at the time of harvest of sugarcane.

Keywords: Carbofuran; dissipation; metabolite; sugarcane.

1. INTRODUCTION

Sugarcane is the one of principal cash crop of India with the highest production of sugar after Brazil. In India, sugarcane is cultivated over an area of 5.11 million ha with an annual production of 400.15 million tonnes and productivity 78.3 tonnes/ha [1]. Sugarcane crop losses due to insect and mites pests during pre and post-green revolution era in India was 10% and 20%, respectively, which clearly indicate that after green revolution insect-pests infestation in sugarcane is continuously increases [2]. Like other annual crops of economic importance, several factors are responsible for the low productivity of sugarcane in country. Insect-pests are among the important constraints accounting for about 20 per cent loss in cane yield and significant reduction in sugar recovery [3]. Sugarcane crop is also subjected to ravage by borer and white grub causing widespread damage to roots and underground stem. Under severe incidence, yield loss due to white grub alone has been estimated to be 80 per cent with 5-6 per cent reduction in sugar recovery [4]. For control of these insect-pests, soil applied insecticides are extensively used, which creates a problem of resistance, resurgence and residue. Among these residue problem is one of the most important debating issues for human and soil health.

The edapho-climatic conditions of the cane growing areas also vary widely across the country and ultimately influence the persistence of soil-applied granular insecticides to a greater extent. The duration over which the insecticide remains biologically active in the soil is one of the key factors that influence its toxicity. The existence of biological activity of insecticide does not mean the persistence of parent compound. Sometimes the metabolites of soil-applied insecticides are more toxic to insect-pest and therefore, persistence of biological activity depends on the persistence and joint action of parent compounds and their metabolites. The prediction of pesticides half-life and its persistence in the environment is an important parameter in agronomic practices because it supplies information on the residual activity of agrochemicals which could cause damages to the successive crops and potential contamination of ground water is becoming an important consideration for pesticide use [5]. Carbofuran is one of the broad spectrum N-methyl carbamate insecticide, widely used in agriculture for the control of insects, mites and nematodes in soil or for protection of different crops. Technically carbofuran is 2,3-dihydro-2,2-dimethyl-7-benzofuranyl-N-methylcarbamate which is manufactured by the reaction of methyl isocynate with 2,3-dihydro-2,2-dimethyl-7-hydroxy benzofuran [6]. Although carbofuran is systemic in nature which enable it readily absorption through roots system and then their distribution to other plant parts, yet its non-persistence nature in soil and rapid metabolization to non-toxic metabolites makes it an ideal choice for crop protection. Therefore, the chance of entry of residue of either carbofuran or its metabolites reduced drastically in food chain [7]. Previously phorate, an organophosphate was also used for the control of stem borer in sugarcane but at present it is banned in India as a commercial insecticide [8]. Therefore carbofuran is more popular because it works as an insecticide, miticide as well as nematocide.

The advice for the use of agrochemicals for a crop cannot be given until its dissipation and residue studies carried out in the particular commodities. Information on degradation rate of pesticides also helps to assess and predict the environmental behaviour of the chemicals [9]. Little information is available on the persistence and dissipation of carbofuran and its metabolites especially under South Gujarat conditions. Therefore, by considering these facts, this study was taken under consideration.

2. MATERIALS AND METHODS

The experiment was carried out at Main Sugarcane Research Station farm of Navsari Agricultural University, Navsari, Gujarat during 2017-18. The experiment farm is located at $20^0$92' N and $72^0$89' E at an altitude of about 10 m above Mean Sea Level. According to climatic condition, Main Sugarcane Research Station farm comes under South Gujarat heavy rainfall zone-I (Agro-ecological situation-III). The climatic condition of region is typically tropical, characterized by humid and warm monsoon with heavy rains, quite cold winter and fairly hot

summer. The average annual rainfall of the Navsari region is around 1500 mm [10]. The soil of experimental field was clay in texture [11] having pH 7.7 [12], EC 0.48 dS/m [12] and organic carbon 0.68% [12]. The experiment was conducted with Large Plot Design having three repetitions. Each plot had 90 m^2 areas.

2.1 Chemicals and Reagents

Reference standards of carbofuran and its metabolite carbofuran 3-OH having purity 99.9 and 98.0%, respectively were procured from Sigma-Aldrich, USA. Solvent like water (LCMS grade), HPLC grade acetonitrile (purity ≥99.9%) and anhydrous magnesium sulfate (purity ≥99.5%) were procured from Merck KGaA, Germany. Primary Secondary Amine (Ethylenediamine-N-propyl, particle size 40 µm) was from SUPELCO, Bellefonte, USA. Acetone, sodium chloride, anhydrous magnesium sulfate and anhydrous sodium sulfate were obtained from Fisher Scientific, UK.

2.2 Instrumentation

LC-MS/MS was used for quantification of carbofuran residues from soil, sugarcane juice and leaves matrix and their details are given below:

uHPLC	:	Thermo Scientific Dionex UltiMate-3000
MS/MS	:	Thermo Scientific Quantum Access Max
Column	:	C_{18} Column (100 mm X 2.1 mm i.d.1.9 µm)
Mobile phase	:	A- Water + 0.1% Formic acid; B- Acetonitrile + 0.1% Formic acid
Column oven temp.	:	25 ^{0}C
Flow rate	:	0.3 mL/min.
Auto sampler	:	Dionex ultimate 3000 R Auto sampler
Injection volume	:	5 µL
Ion spray voltage	:	4500 V
Vaporizer temp.	:	350 ^{0}C
Capillary temp.	:	325 ^{0}C
Sheath gas pressure	:	48 arbitrary unit
Aux gas pressure	:	18 arbitrary unit

2.3 Standard Solution

A technical grade carbofuran and carbofuran 3-hydroxy standard was accurately weighed on Oahu's (maximum capacity 210 g and sensitivity 0.001 g). The reference standards were then transferred to 100 mL capacity volumetric flasks. The content was initially dissolved with acetonitrile and final volume was made up with acetonitrile (Primary standard) and its concentration was calculated with the help of formula given below. From the primary standard, a suitable aliquot was diluted with acetonitrile in volumetric flask to prepare intermediate standard mixture of 10.0 µg/mL. The intermediate standard was further diluted with acetonitrile to obtain working standard.

$$Primary\ standard\ (\mu g/mL) = \frac{\text{Weight of reference standard (mg)}}{\text{Final volume}} \text{ X } \frac{\text{Purity (\%)}}{100} \text{ X } 1000$$

2.4 Method Validation

An analytical method is a sequence of procedures from sample receipt to the final result production. Validation is a process which verifies that the available method is fit for the particular purpose. The method performance verification studies were validated according to criteria given by SANTE guideline [13] in terms of linearity, detection limits, accuracy and precision for carbofuran and carbofuran 3-hydroxy from soils, sugarcane leaves and juice.

2.4.1 Linearity

A linearity study was performed to determine the performance of LC-MS/MS. The linearity of a method is measured of range within which the results are directly, or by well-defined mathematical transformation, proportional to concentration of analyte in a given range. To work out the linearity, response (area) of the detector *vs*. concentration was plotted. To establish the linearity seven different concentrations of the standards viz., 1, 2.5, 5, 10, 25, 50, 100, and 250 ng/mL were injected and their response was recorded.

To work-out the predictable strength of calibration curve per cent residual was calculated.

$$\%\ residual = \frac{Actual\ response - Anticipated\ response}{Actual\ response}\ X\ 100$$

Anticipated response is calculated by using calibration curve where, y is anticipated response and x is actual response.

2.4.2 Detection limits

The limit of detection **(**LOD) is the lowest concentration of analyte detects by an analytical instrument and limit of quantification (LOQ) is the lowest concentration that can be determined with acceptance precision and accuracy under the stated experimental condition. LOD and LOQ were determined on the basis of signal to noise ratio (S:N) of 3 and 10, respectively by using the following formulae [14].

LOD = 3 X Mean Standard Deviation in the response of standard / Slope of regression equation of linearity

LOQ = 3.33 X LOD

2.4.3 Accuracy and precision

In order to ensure quality assurance information such as accuracy or trueness and precision of the analytical method, the recovery study was carried out for different matrices *viz*., soil, sugarcane leaves and juice before taking up analysis of test sample for each treatment. As per SANTE guideline [13], per cent recovery and per cent relative Standard Deviation (RSD) is the indicator of trueness and precision of any analytical method employed for the quantitative estimation of insecticides. A representative sample was fortified with mixture of carbofuran and carbofuran 3-hydroxy at 5, 10 and 25 ng/g level. The fortified samples were kept at room temperature for 2 hrs and residues were estimated.

2.5 Application of Carbofuran

300 g of carbofuran 3G was mixed thoroughly with dry sand of very fine texture and uniformly distribute in the each experimental plot at the time of planting and 60 days after planting of sugarcane.

2.6 Sampling

Periodic soil sampling was started at 60 days after planting of sugarcane from 0-15 cm soil depth with the help of soil auger. About 2.5 kg soil was collected from each plot in a zigzag manner covering whole experimental plot and carried out at Food Quality Testing Laboratory, Navsari Agricultural University, Navsari. The samples were processed on the same day for residues study. The soil samples was taken at 0 (2 hrs), 1, 3, 5, 10, 20, 30, 60 and 90 days after last application of carbofuran. Plant samples were taken at time of harvest for terminal residue analysis. At the time of harvest from each plot, randomly 10 sugarcane plants were harvested and leaves and cane were separated. About 2 kg green leaves and 2 kg juice from cane after extraction were taken for residue analysis.

2.7 Extraction and Clean Up

2.7.1 Soil

The method adopted for the multi-residue analysis from soils is popularly known as QuEChERS method [15]. A 10 g of representative soil was transferred in 50 mL capacity centrifuge tube. A 20 mL of acetonitrile added and shaken it for 1 minute. 4 g $MgSO_4$ and 1 g NaCl were added in centrifuge tube and vortex followed by centrifuged at 3500 rpm for 2 minute. After it, 10 mL of solution was transferred in the 15 mL capacity centrifuge tube containing 1.5 g $MgSO_4$ and 0.250 g PSA (Primary Secondary Amine) and again centrifuged for 2 minute at 3500 rpm. 4 mL of aliquot was transferred in test tube and evaporated it to dryness with N- dryer at 40°C. Finally 1 mL volume was make-up using HPLC grade acetonitrile and filtered it in to the glass vial for quantification on LC-MS/MS.

2.7.2 Sugarcane leaves

Modified QuEChERS method [was adopted for insecticides residue analysis from sugarcane leaves samples [16]. About 1 to 2 kg of sugarcane leaves was chopped and homogenized in high volume homogenizer. A 10 g of representative leaves sample was transferred in 50 mL capacity polypropylene centrifuge tube, to which 20 mL of acetonitrile was added and homogenized the sample at 5000 rpm for 3 minute. After this 3 g NaCl was added and shake well for 2 minute followed by centrifuged for 3 minute at 3000 rpm to separate organic layer. 12 mL of organic layer was transferred into 50 mL capacity centrifuge tube and added about 10 g sodium sulphates and shake well for 1 minute again centrifuged at 3000 rpm. From this 6 mL upper organic layer was transferred to 15 mL capacity centrifuge tube containing 0.3 g PSA and 0.9 anhydrous $MgSO_4$ and centrifuged for 5 minute at 3000 rpm. 2 mL of supernatant aliquot was transferred in test tube and evaporated it to dryness with TurboVap at 40°C. Finally 2 mL volume was make-up by using acetonitrile (HPLC grade) and filtered it in to the glass vial for quantification on LC-MS/MS.

2.7.3 Sugarcane juice

The modified QuEChERS method was adopted for insecticides residue analysis from sugarcane juice [17]. 10 mL of juice sample was transferred in 50 mL capacity centrifuge tube followed by10 mL of acetonitrile and vortex it for 1 minute. After this 4 g $MgSO_4$ + 1 g NaCl was added and mixed again for 1 minute followed by centrifuged it for 10 minute at 1200 rpm. From this 4 mL upper layer of aliquot was transferred in 15 mL capacity centrifuge tube having 600 mg $MgSO_4$ and 200 mg PSA and vortex for 30 second followed by centrifuged for 3 minute at 3500 rpm. 2 mL of aliquot was transferred in test tube and evaporate to dryness with N- dryer at 40°C. Finally 2 mL volume was make-up with HPLC grade acetonitrile and filtered it in to the glass vial for quantification on LC-MS/MS.

2.8 Dissipation Kinetics

The dissipation kinetics of carbofuran in experimental soils were determined by three dissipation kinetic models *viz*., zero order, first order and second order kinetic models. The detail information of kinetic models are given in Table 1.

3. RESULTS AND DISCUSSION

3.1 Method Validation Parameters

3.1.1 Linearity

The calibration curve of carbofuran and carbofuran 3-hydroxy showed linear response in the concentration range of 1.0 to 250 ng/mL. Coefficient of determination (R^2) values of carbofuran and carbofuran 3-hydroxy standard curve was >0.99. The mean per cent residual of carbofuran at 5, 10 and 25 ng/mL was 10.10, 3.65 and 1.55%, respectively. However, mean per cent residual of carbofuran 3-OH at 5, 10 and 25 ng/mL was 4.63, 1.71 and 1.92%, respectively.

3.1.2 Detection limit

The limit of detection (LOD) values obtained for carbofuran and carbofuran 3-OH were 0.905 and 1.624 ng/g, respectively. The corresponding limit of quantification (LOQ) values of this method was worked out to be 3.02 and 5.41 ng/g for carbofuran and carbofuran 3-hydroxy, respectively.

3.1.3 Accuracy and precision

Accuracy and precision was determined by calculating per cent recovery and per cent Relative Standard Deviation (RSD). The average recovery value and RSD of carbofuran and carbofuran 3-hydroxy from soil, sugarcane juice and leaves are given in Table 2.

QuEChERS based extraction procedure and measurement on LC-MS/MS instrument for analysis of the residues of carbofuran and carbofuran 3-hydroxy had R^2 >0.99, residual <20%, recovery range between 70-120%, RSD <20% and BQL value of carbofuran <MRL (500 ng/g in sugarcane) which fulfilled the criteria of method validation as prescribed in SANTE guidelines [13].

Table 1. Dissipation kinetics models

Model	*k*	DT_{50}
Zero order $C_t = C_0 - kt$	$k = \frac{C_0 - C_t}{t}$	$DT_{50} = \frac{C_0}{2k}$
First order $C_t = C_0 e^{-kt}$	$k = \frac{2.303}{t} \log \frac{C_0}{C_t}$	$DT_{50} = \frac{\ln 2}{k}$ *or* $\frac{0.693}{k}$
Second order $\frac{1}{C_t} = \frac{1}{C_0} + kt$	$k = \frac{C_0 - C_t}{t(C_t X C_0)}$	$DT_{50} = \frac{1}{k C_0}$

k is dissipation rate constant, C_t is concentration at t time, C_0 is initial concentration and DT_{50} is 50% dissipation time

Table 2. Accuracy and precision of extraction method for the insecticides and their metabolites in soil, sugarcane juice and leaves

Compound	Level (ng/g)	Soil		Sugarcane juice		Sugarcane leaves	
		*Mean Recovery (%)±SD	% RSD	Mean Recovery (%)±SD	% RSD	Mean Recovery (%)±SD	% RSD
Carbofuran	5	84.38±9.16	10.86	85.33±6.59	7.72	92.11±8.37	9.09
	10	97.49±7.65	7.85	91.31±6.53	7.15	96.84±11.88	12.27
	25	87.23±4.34	4.98	77.62±3.34	4.30	90.02±3.92	4.36
Average recovery across levels		89.70±9.04	10.07	84.75±7.88	9.30	92.99±8.75	9.41
Carbofuran 3-OH	5	94.42±13.66	14.47	89.41±9.33	10.43	96.89±5.67	5.85
	10	95.35±7.38	7.74	87.17±5.80	6.66	99.28±5.10	5.14
	25	88.61±10.44	11.78	82.43±5.27	6.39	89.70±9.46	10.54
Average recovery across levels		92.79±10.69	11.52	86.34±7.31	8.47	95.29±7.85	8.24

**mean of seven replications*

3.2 Persistence and Dissipation

Carbofuran and total carbofuran persist in sugarcane grown soil upto 30 days after application. The extractable residues of carbofuran and total carbofuran (Carbofuran and carbofuran 3-OH) dissipated in similar fashion tabulated in Table 3. The loss of carbofuran and total carbofuran residues was faster in initial phase because solubility of carbofuran in water is high (320 mg/L) so conversion of carbofuran into their metabolite was observed during initial days [6,18]. In our study metabolite of carbofuran *i.e.* carbofuran 3-OH was observed upto 1 day after application. Other scientists were also observed that 3-hydroxy carbofuran dissipated rapidly in soils it was not detect 7 days after application [19]. The residues of carbofuran and total carbofuran lost but at considerably slower pace in the latter phase of this study. The carbofuran residue in soil degraded steadily up to 15 days (86.63-97.62%) but later the rate of degradation was negligible [20].

Among the dissipation kinetic models, three dissipation kinetic models are taken under consideration and found that first order model was best fit for carbofuran and total carbofuran, judging from the significant of the coefficient of determination *i.e.* 0.876 and 0.875, respectively (Table 4). Therefore, half-life of the carbofuran (4.18 days) and total carbofuran (4.17 days) was assessed from the first order kinetics rather than zero and second order kinetics.

Table 3. Persistence and dissipation behaviour of carbofuran and its metabolite in sugarcane grown soil

Days after application	Carbofuran			Metabolite	*Total carbofuran		
	Residue (ng/g)	Loss (%)	Persistence (%)	Carbofuran 3-OH (ng/g)	Residue (ng/g)	Loss (%)	Persistence (%)
0 (2 hrs)	4059.01	-	100.00	12.79	4071.80	-	100.00
1	2322.77	42.77	57.23	44.73	2367.50	41.86	58.14
3	534.05	86.84	13.16	BQL	534.05	86.88	13.12
5	286.86	92.93	7.07	BQL	286.86	92.96	7.04
10	147.84	96.36	3.64	BQL	147.84	96.37	3.63
20	79.37	98.04	1.96	BQL	79.37	98.05	1.95
30	12.39	99.69	0.31	BQL	12.39	99.70	0.3
60	**BQL	-	-	BQL	BQL	-	-
90	BQL	-	-	BQL	BQL	-	-
At harvest							
Sugarcane juice	BQL	-	-	BQL	-	-	-
Sugarcane leaves	BQL	-	-	BQL	-	-	-

**Total carbofuran = Carbofuran + Carbofuran 3-OH; **BQL is <LOQ*

Table 4. Dissipation kinetics and half-lives of carbofuran and total carbofuran in sugarcane grown soil

Compound	Model	Kinetic equation	R^2	*k* (Per day)	DT_{50} (days)
Carbofuran	0 order	y = -85.5300x + 1906	0.384	85.5300	23.73
	1st order	y = -0.16602x + 7.334	0.876	0.1660	4.18
	2nd order	y = 0.002276x - 0.007	0.763	0.0023	0.11
Total carbofuran	0 order	y = -86.2219x + 1921	0.385	86.2219	23.61
	1st order	y = -0.16628x + 7.340	0.875	0.1663	4.17
	2nd order	y = 0.002276x - 0.007	0.763	0.0023	0.11

3.3 Residues in Sugarcane

Terminal or harvest time residue of carbofuran and its metabolite carbofuran 3-hydroxy in sugarcane juice and leaves were found below quantification level at the time of harvest (Table 3). In the earlier section, it was observed that persistence of carbofuran and its metabolite in soil was up to 30 days after application and after 60 days it was found BQL (Table 3). It indicates that carbofuran residues available for absorption by sugarcane plants are only up to 30 days after application and after 60 days it was not available for the plant. Similar finding were also reported by other scientists for carbofuran [20].

4. CONCLUSION

Residues of carbofuran persist in sugarcane grown soil upto 30 days after application. Carbofuran and total carbofuran (carbofuran + carbofuran 3-hydroxy) followed first order dissipation kinetics with the half-life of 4.18 and 4.17 days, respectively. Sugarcane leaves and juice had carbofuran residues below quantification level at the time of harvest.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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