Research Article

STUDY OF CYPERMETHRIN RESIDUE IN OKRA LEAVES AND FRUITS ASSESSED BY GC

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ABSTRACT

The use of pesticides in agriculture concern of residue which may affect on human health. Cypermethrin is a relatively toxic and it is used to control moth pests of fruits and vegetable crops. As a consequence pesticide residue can be found in okra fruits and leaves. GC analytical performance were good ,low limit of detection. Instrumental methods are generally more sensitive and selective than the classical methods. Gas chromatography is one of the most powerful method and is used for determination of cypermethrin residue. Cypermethrin were selected in okra leaves and fruits. Cypermethrin were applied separately in three different concentrations i.e .50ppm, 75ppm, 100ppm on okra crops .Extraction of leaves and fruits were done by using solvent mixture Petroleum Ether: Acetone(9:1)by standard method and is estimated by GC. In the present conditions gas chromatography is most frequently used for residue analysis .After the application of Cypermethrin on okra crop the residue were determined 0,1,3,57,9,11,13,15,17,19 and 21 days after application. The results indicate that the residue below the detectable were found after 17 days. The purpose of the determination of cypermethrin residue is to consume these foods which contain pesticides at this level for their whole lives considered safe.

Keywords: Pesticide, Cypermethrin, PPM, GC, BDL.

INTRODUCTION

The use of pesticides has increased several fold in India and it is likely to increase in the forthcoming years. It is a well known fact that indiscriminate use of pesticides has also lead to high residue levels in food .Even small quantities of these residues present in food lead to high levels in the body fat when these food stuffs are consumed over long period of time .It causes a health problem. In recent times there are several measures being taken to regulate the contamination of food with pesticide residue to safe levels.

Instrumental methods are generally more sensitive and selective than classical methods .Gas chromatography is one of the more powerful method for determination of residue analysis. Analytical instruments are needed to determine pesticide residue in vegetables and fruits. The pesticides are generally analysed by spectrometry[1,2], thin layer chromatography[3,4] high performance liquid chromatography(HPLC) & high performance liquid chromatography mass spectrometry (HPLC-MS)[5-7,] gas chromatography (GC)[8-11],GC-MS[12].

Recently serious attentation has been focused on problems associated with the effect of pesticide residue on human life and environmental alteration. Many workers has been reported the toxicity of pesticides. The application of insecticide is a group of pesticide in crop field insecticide &group of pesticides in crop fields for selective control of pests in the modern age has led to serious environmental contamination resulting the greater loss of crop productivity and growth of many beneficial microorganisms, like phytoplanktons etc.

References indicates that the Cypermethrin residue for the samples of jute leaves from the experimental plots & retted fiber were analyzed by Gas-Liquid chromatography .On 0-day of all the three different dates of spraying showed 6.222ppm, 6.385ppm & 6.875ppm of Cypermethrin respectively on jute leaves. No detectable residues were found on 20 days of application on jute leaves. No residue was detected from retted fiber (13).When Cyperrmethrin was sprayed at 0.01and 0.02% concentration in onion it required 7days to reach below tolerance limit (14) and in urinal 0.01% spray required 2.76 days for residue to reach below maximum permissible residue level (15).

India is an agricultural country. It's80% population is dependant on agricultures (16).To achieve economic benefit and to make sufficient supply of food to vast population, it becomes necessary to increase the yield of crops. Sometimes they might cause economic losses. The use of pesticides contaminated the food stuffs, thus instead of supporting health these become a great health hazard(17).The pesticides commonly applied for farmers are Fenvelerate, Endosulphan, Cypermethrin, Monocr -otophos, Parathion etc(18).This research paper deals with the study of pesticide residue Cypermethrin by using Gas Chromatography technique.

MATERIALS AND METHODS
Chemicals: Petroleum ether, acetone is AR grade, Cypermethrin obtained from Rallis India Ltd. Ralchem, Patanchene .During the experiment different materials, equipments and methods applied for study.

Apparatus: Gas chromatograph Hewlett Packward 5840A, equipped with ECD and FID.

Collection of sample: Field experiment with Okra crop (variety Parbhani Kranti) was conducted at the experimental fields.Field experiment was carried out at agricultural farm at Vishnupuri village. The surface soil used as an organic matter content of 82% &PH7.2.The experimental plots were established in an area free from detctable Cypermethrin residues.

Okra fruits& leaves samples were collected from all treatment of Cypermethrin on 0,1,3,5,7,9,11& so on odd days after each spraying. The samples were mixed treatment wise &representative sample was used for extraction.

Extraction method: 25 gm leaves and fruits were blended separately with 100ml of Petroleum ether: Acetone (9:1)along with 10gm of sodium sulphate in a blender for 2 min. it was then re-extracted twice with 50ml of same solvent mix.& filtered, combined filter was then washed with saturated 5% sodium chloride solution in separating funnel &then extracted. The extract was then concentrated and 2gm of activated charcoal was added to it. It was kept 15-20 min. with occasional shaking &filter. The grinded okra leaves &fruits were extracted with Pet. ether: Acetone (9:1) in a sox let extraction apparatus. The extract was then cleaned up by standard procedure used for leaves & fruits.

Column preparation: Column for adsorption was prepared by packing the glass column (2*40cm) with one inch non adsorbent sterilized cotton wool. Over this layer one inch layer of anhydrous sodium sulphate layer, one inch layer of silica gel, one inch layer of activated charcoal and one inch layer of sodium sulphate. Lastly the column was packed with one inch cotton layer. The column was saturated with the required solvent for individual pesticide. The extract was passed through the column obtained clear & colourless eluate.

GC Analysis: Cypermethrin residue was determined by GLC. The efficiency of extraction cleans up &estimation procedure was checked by fortifying the samples with known amount of cypermethrin. The recovery was 89-99.5%.The limit of detection was 0.001ppm

Residue of cypermethrin were estimated Hewlett Packard gas chromatograph (USA)equipped with electron capture detector coupled with model 3392 intergrator.The column injector&detector temp.were255&275 degree celcius resp.flow rate of carrier gas N was 70ml/min.

The efficency extraction clean up &estimation procedure was checked by fortifying the samples with known amount of Cypermrthrin.The recovery was 89.99% The limit of detection was 0.001ppm.

RESULT AND DISCUSSION
Cypermethrin in leaves of Okra extracted in petroleum ether: Acetone (9:1) estimated by GC.

Table 1:

Days after application	Conc.50ppm residue in ppm	Conc. 50ppm reduction in %	Conc. 75ppm residue in ppm	Conc. 75ppm reduction in %	Conc. 100ppm residue in ppm	Conc. 100ppm reduction in %
0	3.619	-	4.019	-	4..531	-
1	2.163	40.23	2.385	40.65	2.678	40.89
3	1.209	66.57	1.326	67.00	1.472	67.5
5	0.761	78.97	0.843	79.00	0.944	79.15
7	0.398	89.00	0.436	89.15	0.474	89.52
9	0.179	95.05	0.190	95.25	0.212	95.32
11	0.014	99.61	0.012	99.70	0.033	99.25
13	0.010	99.51	0.007	99.81	0.028	99.36
15	0.001	99.96	0.004	99.90	0.009	99.79
17	BDL	-	0.001	99.96	0.005	99.86
19	-	-	BDL	-	0.001	99.96
21	-	-	-	-	BDL	-

Cypermethrin residue in fruit of Okra extracted in petroleum ether: Acetone (9:1) estimated.

Table 2:

Days after application	Conc.50ppm residue in ppm	Conc.50ppm reduction in %	Conc.75ppm residue in ppm	Conc.75ppm reduction in %	Conc.100ppm residue in ppm	Conc.100ppm reduction in %
0	3.471	-	3.916	-	4.219	-
1	1.980	42.95	23.446	43.00	2.387	43.41
3	0.880	74.64	0.977	75.05	1.028	75.62
5	0.532	84.67	0.591	84.90	0.632	85.00
7	0.210	93.94	0.225	94.10	0.232	94.50
9	0.110	96.83	0.117	97.00	0.103	97.51
11	0.090	97.40	0.052	98.65	0.084	97.99
13	0.050	98.55	0.007	99.80	0.042	98.99
15	0.002	99.94	0.001	99.96	0.020	99.55
17	BDL	-	BDL	-	0.006	99.86
19	-	-	-	-	0.001	99.96
21	-	-	-	-	BDL	-

Awasthi (18) reported that 0.0075&0.01 percent cypermethrin spray resulted in the initial deposit of 0.93 &1.21 ppm & reached below 0.5ppm as detected by GC. The initial deposits were higher as compaired to those in present studies. Sinha et al (19) could record the initial deposit of only 0.40 ppm on brinjal when cypermethrin was sprayed at 0.01 percent showing a great variation in the initial deposit which may be due to plant type and surface of the fruit which does not permit deposition (20,21,22,23,24,25).

The estimation residue was done by extraction of the samples in Pet.Ether: Acetone (9:1). Okra leaves and fruits applied with three different concentration 50ppm, 75 ppm & 1000 ppm of cypermethrin were estimated for the level of residue. The leaves and fruits were extracted in Pet.Ether: Acetone (9:1). After 0,1,3,5,7,9,11,13,15,17,19 & 21 days after application. The residue was determined by GC.

MRL's pf cypermethrin for the studies is in the table no. 1 &2, it indicates that the presence of 3.619 ppm&3.471 ppm initial residue at 0 hours in leaves and fruit sample respectively. The residue below detectable level was found in 17 days in concentration 50 ppm. The residue 4.019 ppm &3.916 ppm initial residue gone below detectable level in 17 & 19 days in 75 ppm . The initial residue 4.531 & 4.219 was below detectable level in 21 days in 100 ppm in leaves and fruit respectively.

The minimum residue detected was 0.001 ppm where as the maximum reduction was 99.96 %, 99.96 %, 99.96 %& 99.94 %, 99.96%, 99.96% in 50 ppm, 75 ppm & 100 ppm respectively in both leaves and fruit.

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