

Insecticide Residues in Market Samples of Brinjal in Imphal, Manipur, India

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Twenty samples of brinjals (1 kg each) are collected from different vegetable markets of Imphal, India at fortnightly interval to assess the extent of pesticide residues. The samples were analyzed using multi-residues methodology (MRM) and residues were estimated by GLC for the presence of organochlorine, organophosphorus and synthetic pyrethroid insecticides. The study reveals that the maximum residue levels was observed for phorate, dimethoate, chloropyrifos, triazophos, β -endosulfan, λ -cyhalothrin and cypermethrin were 0.01, 0.19, 0.12, 0.23, 0.08, 0.12 and 0.13, respectively. None of the samples exceeded their respective maximum residues limit.

Key Words: Insecticides, Residues, Brinjals, Maximum residues limit, GLC.

INTRODUCTION

Brinjals are also commonly available vegetable items in both summer and winter season but in different quantity. In Imphal markets, 60-70 % brinjals are from outside Manipur. The crop is therefore constantly under the threat of attack by brinjal pests and other pathogens which produce diseases and result in economic losses.

Due to brinjal pests, the foods are unfit for human consumption. For the control of insect and other pests at different stages, the farmers commonly used λ -cyhalothrin, cypermethrin, monocrotophos, fenvalerate, phorate, endosulfan, dimethoate, quinalphos, profenofos, chloropyrifos, malathion and methylparathion. The indiscriminate and injudicious application of pesticides and their subsequent persistence contaminate our environment. Surveys conducted in various parts of India showed high incidence of pesticide residues in food commodities¹. As the pesticide use in crop is very common, regular monitoring of residues levels in food commodities is of paramount importance to human being. Further to know the pesticide residues status in brinjals is very important as this type of vegetables are consumed directly

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without much processing. Little information is available on monitoring of insecticide residues in market sample of vegetable items in Manipur. In light of the above facts, this study is undertaken to estimate the level of insecticide residues present in brinjal grown in Imphal, India.

EXPERIMENTAL

The solvents were further purified by distillation. All other chemicals were of analytical grade and were used as such. Running reagent blanks along with the samples during analysis checked the suitability of the reagents/solvents.

Twenty samples of brinjals (1 kg each) were collected from different markets of West Imphal at fortnightly intervals. The samples were processed immediately to avoid any loss of insecticides during storage. A representative sample of brinjals (100 g) was blended in 100 mL acetone: water (65:35), a few mL at the time of blending just for homogenization. It is then transferred in a beaker and added all the solvent completely and stirred well using a glass rod. It was filtered through a buchner funnel using whatman No. 41 and collected the extract.

Liquid-liquid clean up: 80 mL of extract in a 500 mL separating funnel + 100 mL mixture of hexane: DCM, was shaken well for a minute. The layers were allowed to separate and then collected the upper organic layer. 10 mL of saturated NaCl soln + 100 mL DCM was added to solution and shaken well for a minute and collected the entire organic layer from the evaporator to 3 mL then rinsed with hexane and recondensed nearly dryness and then made up the volume up to 3 mL with acetone.

Column clean-up: After plugging the glass column with cotton, packed with 2 g anhydrous Na₂SO₄ and 5 g activated florisil in alternate layers.

Elution: 1 mL of the extract was made up volume upto 10 mL by adding 10 % acetone in hexane, and eluted with eluants 50 mL DCM + 48.5 mL hexane + 1.5 mL acetonitrile at the rate 5 mL/min. It is for OCs/Sp analysis. 2 mL of extract was eluted with eluants acetone: DCM (2:1). Then each extract was collected after elution separately and condensed up to 3-5 mL using hexane for organo chlorines/synthetic pyrethroids and acetone for organophosphorus in final volume.

The residues of insecticides were estimated using gas chromatography model Chemito-86 10 equipped with thermo ionic detector (TID) fitted with capillary column and GC model CP-3 800 equipped with electron capture detector (EDC) fitted with capillary column. The operating condition are given in Table-1.

RESULTS AND DISCUSSION

The results obtained by analysing the samples of brinjals are presented in Table-2. Out of 20 samples of brinjals analyzed, the mean residues levels of phorate and λ -cyhalothrin were 0.01 and 0.06 mg/kg, respectively.

The selected insecticides/isomers/metabolite are methyl parathion, chlorpyrifos, malathion, phorate, quinalphos, monocrotophos, triazophos, dimethoate, profenfos, α -HCH, β -HCH, δ -HCH, ppDDD, ppDDE, endosulfan sulphate, a endosulfan, β -endosulfan, λ -cyhalotrin, diocofol, deltamethrin, cypermethrin and lindane (Table-2).

TABLE-1
DETAILS OF GLC PARAMETERS

Detector	ECD (OC/SP)	TID (OP)
Temperature	Column-180-250 °C @ 5 °C/min Injection-250 °C Detector-300 °C	Column-200-220 °C @ 1 °C/min Injection-240 °C Detector-280 °C
Column	DB 1701P; 30 m × 0.32 mm	HP 5; 30 m × 0.32 mm
Nitrogen flow rate (mL/min)	2	4
Hydrogen flow rate (mL/min)	2	4.8
Zero air flow rate (mL/min)	2	90
Contact potential (mV)	150	316
Split mode	Splitless	Splitless
Volume injected (µL)	1-2	1-2

TABLE-2

S. No.	Phorate	Dimethoate	Chloroxyriphos	Triazophos	β-Endosulfan	λ-Cyhalothrin	Cypermethrin
1	BDL	BDL	BDL	0.23	BDL	BDL	BDL
2	BDL	BDL	BDL	BDL	BDL	BDL	BDL
3	BDL	BDL	BDL	BDL	BDL	BDL	BDL
4	BDL	BDL	0.12	BDL	BDL	BDL	0.02
5	BDL	BDL	BDL	BDL	BDL	BDL	BDL
6	BDL	BDL	BDL	0.18	BDL	BDL	BDL
7	BDL	BDL	BDL	BDL	BDL	BDL	BDL
8	BDL	0.02	BDL	0.07	BDL	BDL	0.06
9	BDL	BDL	BDL	BDL	BDL	0.12	BDL
10	0.01	BDL	BDL	BDL	BDL	BDL	BDL
11	BDL	BDL	0.08	BDL	BDL	BDL	BDL
12	BDL	BDL	BDL	BDL	0.08	BDL	0.13
13	BDL	0.16	BDL	BDL	BDL	BDL	BDL
14	BDL	BDL	BDL	0.12	BDL	BDL	BDL
15	BDL	BDL	0.02	BDL	BDL	0.01	BDL
16	BDL	BDL	BDL	BDL	BDL	BDL	BDL
17	BDL	BDL	BDL	0.14	BDL	BDL	0.11
18	BDL	0.19	BDL	BDL	BDL	BDL	BDL
19	BDL	BDL	BDL	BDL	BDL	BDL	BDL
20	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Mean	0.01	0.12	0.07	0.15	0.08	0.06	0.08
MRL	0.05	2.00	0.20	0.50	2.00	0.20	0.20

BDL = Below detectable limit, MRL = Maximum residue limit.

The detection of mixtures of OCs and SP were found good on the capillary column DB 170 IP and Ops found good on capillary column HP.5. When the compounds were injected into the GC as a mixture, the resolution was good.

The individual insecticide standards (10 OCs, 3 SP and 10 OPs) were also injected at specified set of operating conditions of GC to obtain peaks and retention times for each of the insecticides from the mixtures. GC-EDC identified OCs and SP and OPs through GC-TID in a mixture by employing temperature programme along with gas flow.

The insecticides fortified brinjals showed the recovery percentage 19.75, 18.64, 15.67, 54.00 and 63.94 for methyl parathion, chloropyrifos, quinalphos, lindane, λ -cyhalothrin, respectively.

In brinjal, the recovery percentage for chloropyrifos, methyl parathion, quinalphos, lindane, α -endosulfan, β -endosulfan, λ -cyhalothrin, were observed as 28.25, 15.64, 30.86, 58.92, 53.03, 46.08 and 45.00, respectively from Florisil adsorbent.

Lakwah *et al.*² reported pirimiphosmethyl and malathion residues in most of the market samples of tomatoes and cucumber, but none of the samples contained residues above their respective MRL's. Similarly low levels of residues of methyl parathion, triazophos and malathion were detected in farmgate vegetable samples of cauliflower, brinjal, okra and tomato^{3,4}.

The analysis of vegetable samples for pesticide residues under All India Coordinated Research Project on Pesticide Residues revealed that majority of the samples were generally below the prescribed MRL's. In some European countries it was verified that in many cases exceeded the permissible levels⁵. Out of 20 samples of brinjal: analysed; phorate, λ -cyhalothrin, dimethoate, β -endosulfan, chloropyrifos, triazophos, cypermethrin were present in 5, 10, 15, 5, 15, 24 and 20 %, respectively and mean residue levels were 0.01, 0.06, 0.12, 0.08, 0.07, 0.15 and 0.08, respectively.

The average recoveries assessed from spiked samples were found to be more than 80 %. The maximum residue levels was observed for phorate, dimethoate, chloropyrifos, triazophos, β -endosulfan, λ -cyhalothrin and cypermethrin were 0.01, 0.19, 0.12, 0.23, 0.08, 0.12 and 0.13, respectively. None of the samples exceeded their respective maximum residue limit.

Acephate, methylparathion, monocrotophos, malathion, fenitrothion, quinalphos α -HCH, β -HCH, lindane, δ -HCH, α -endosulfan, endosulfan-sulphate, were present below detectable limit (BDL).

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