ISSN: 2277-9655
Impact Factor: 4.116

IJESRT

INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH TECHNOLOGY

OCCURRENCE OF PESTICIDE RESIDUES IN MARKET AND FARM GATE SAMPLE OKERA CROP , Abelmoschus esculentus (L.) IN MEERUT REGION (U.P.)

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DOI: 10.5281/zenodo.154251

ABSTRACT

A multi pesticide residues analysis was carried out for farmgate samples of okra fruits from four markets places of Meerut region. Collected farmgate samples (20) from Daurala, Lawer, Kesarganj and Partapur markets 40, 45, 30, 35, 50 and 25% samples were exceeded MRL and 30, 40, 35, 45, 35 and 45% samples were recorded below their respective MRL values. Lawer market, 65, 55, 45, 40, 70 and 55% samples contained exceeded MRL and 25, 30, 40, 45, 20 and 25% samples had residues below MRL values, Kesarganj and Partapur markets, 35, 30, 40, 30, 40 and 35% in Kesarganj market and 45, 40, 30, 40, 40 and 40% samples in Partapur market, residues exceeded MRL and 50, 45, 45, 45, 40 and 30% and 40, 40, 45, 45, 35 and 40% samples, respectively contained residues below MRL values of monocrotophos (0.20), chlorpyriphos (0.20), dimethoate (2.00), cypermethrin (0.20), endosulfan (2.00) and imidacloprid (0.05ppm), respectively. So the okra fruits samples with residues above MRL are not safe for human consumption.

KEYWORDS: GLC, HPLC, Monocrotophos, Chlorpyriphos, Dimethoate Cypermethrin, Endosulfan and Imidacloprid.

INTRODUCTION

Agriculture plays an important role in economic structure in India. Okra, *Abelmoschus esculentus*(L). MOENCH, often known as bhindi, lady's finger is valued for its edible green fruit. In India, it is grown over 3.58 lakh ha area with production of 35.25 lakh tones and productivity of 9.84 tones/ha. (Anonymous, 2005). The avoidable losses in yield and fruit damage due to this pest have been estimated as 36-90% (Misra *et al.*, 2002). The jassid is causing damage throughout the growing period of the crop and reduces the plant vigour and fruit yield (Mahal *et al.*, 1994). Insecticides play vital role in management of these pests. Still pest control is largely dependent on the use of many pesticides of different groups as organophosphate, carbamate, organochlorine and synthetic pyrethroides, which cause ill effect on the human health. Synthetic pyrethroid being effective at very low dosage, having low mammalian toxicity and involving less risk of contamination have been recommended to control fruit borers (Gajbhiye *et al*., 1985 and Rai *et al*., 1980). Pesticide, a critical modern input, has become an established global practice. Some people are more vulnerable than others to pesticide impacts. For example, infants and young children are known to be more susceptible than adults to the toxic effects of pesticides. Farm workers and pesticide applicators are also more vulnerable because they receive greater exposures. Immediate health effects from pesticide exposure include irritation of the nose, throat, and skin causing burning, stinging and itching as well as rashes and blisters. People with asthma may have very severe reactions to some pesticides, particularly with pyrethrin / pyrethroid, organophosphate and carbamate pesticides.These chemicals may leave toxic residue in the harvested produce which is consumed by human beings (Babu *et al*., 1996). Pesticide residues come from four sources: on farm pesticide use, post harvest pesticide use, pesticide used on imported food and discarded pesticides that persist in the environment. Above all post harvest pesticide accounts for the largest share of residue detection (Kuchler *et al*., 1996).

On an average 13-14% of total pesticides used in the country are applied in vegetable crops. Since the produce is harvested at short intervals and consume fresh in many cases, the surveys of market samples show high level of pesticide residues in vegetables (Arora and Gopal, 2002, Agnihotri, 1999, Awasthi and Ahuja, 1997).

MATERIAL AND METHOD

Preparation of standard solutions

An accurately weighed 0.0105g amount of an individual analytical grade pesticide in a weighing bottle, was transferred to a volumetric flask (25ml) and dissolving it in distilled acetone and washing the weighing bottle simultaneously with acetone, the volume was made up to the mark of volumetric flask to give 420ppm stock solution. 6ml of this stock solution was diluted with hexane in 25ml volumetric flask to obtain 100ppm solution. Serial dilutions were made in a similar manner so as to get 10ppm and 1ppm solution of each compatible (monocrotophos, chlorpyriphos, dimethoate, cypermethrin, endosulfan and imimdacloprid) pesticide in a 10 ml volumetric flask and making the volume up to the mark with n-hexane. Serial dilutions were made in a similar manner so as to get 10ppm and 1ppm solution for residues analysis. They were stored in a refrigetor at 5^0c.

Collection of samples

Farmgate vegetable samples of okra fruits each (20 in numbers) were collected at random from four different markets *viz*. Daurala, Lawer, Kesharganj and Partapur, located in meerut region (U.P.), during the month of August, 2009. About one kg. Sample each with 20 basket from all four location of markets were collected for pesticides residues analysis. Samples were analyzed for the organochlorine, organophosphate and synthetic pyrethroides group of insecticides based on the information provided by the vegetable growers. Each sample was sub-divided into three replicates from each basket, a sub sample of 50g was withdrawn for processing for residue analysis.

GLC/HPLC estimated instrumation

The collected sub samples of okra fruits were chopped and processed for pesticides residues analysis. The samples were homogenized in a blender (Remei mixie).The extract was treated with 100ml solvent mixture (1:1 hexane: acetone) and was filtered through a Buchner funnel fitted with a Whatman No. 1 filter paper. The residual pieces of okra fruits on the filter paper were again transferred to the blender. This process was repeated two more times with 25ml acetone. After extraction, the blender jar was rinsed with 25ml acetone and rinsate was also filtered the same way and the final volume of the extract, around 100ml was collected.

Solvent from the extract was evaporated off with the help of rotary vacuum evaporator to around 10ml and then transferred to a 250ml separating funnel. 100ml of sodium chloride solution (18%, w/v) followed by 50ml of distilled Dichloromethane were added to it. Separating funnel was thoroughly shaken for 1 minute by slowly releasing pressure through stop cork and allowed to stand (for about 5 minutes) until the two layers were separated. The bottom layer was collected in a conical flask after passing through anhydrous sodium sulphate (5 g) layer. The aqueous solution of the separating funnel was shaken two more times with 50ml DCM. The anhydrous sodium sulphate layer was given additional washing with 10ml DCM and the filtrate was collected in the same flask. Glass column (50cm × 1.5cm) was plugged with cotton at the bottom, then dry packed with 5g of anhydrous sodium sulphate followed by 5g of Florisil and finally again with 5g of anhydrous sodium sulphate. It was prewashed with 20ml hexane. The extract from DCM was taken in 10 ml hexane and was added to the adsorbent column. The column was sequentially eluted with 100ml hexane : acetone (7:3 v/v) and the eluate was collected in a conical flask for final estimation of pesticides using GLC & HPLC.

Analysis of pesticides extract

The samples were analyzed using GLC for determination of organochlorines, synthetic pyrethroides and organophosphates and HPLC for imidacloprid insecticide. GLC (HP 5890 series II) equipped with Ni^{63}, mega bore column (10m, 0.53mm id, 2.65 mm film thickness) and ECD detector. The GLC working conditions were as flows: Nitrogen gas flow rate 12 ml/min Detector, 260°C: and Injector 250°C. The column temperature 150°C maintained for 10 minutes raised @ 5°C/min to 220°C and held for 5 minutes. The retention time (Rt) of monocrotophos 3.0 min, Endosulfan 13.1 min, chlorpyriphos 15.4 min, Dimethoate 18.8 min, Cypermethrin 31.6 min. The HPLC working conditions as were as flows: Column -BEH C-18 (1.7 µm), Column dimensions-100 mm x 2.1 mm id. Column

temperature -50 ºC, Mobile phase -Acetonitrile –water (30:70, v/v),Flow rate -0.5 ml/min, Sample size -5 µl ,Detector -PDA ,λ_{max} -278nm, Retention time -1 min ,BDL-up to the ng.

Insecticides residues formula:

$$\text{Residue (in ppm)} = \frac{A \times B \times C}{D \times E}$$

Where, A = Area of sample peak,B =Concentration of standard ,C =Volume of aliquot,D =Area of standard peak, E=Weight of sample

RESULT

The farmgate samples of okra fruits were collected from four markets of Meerut region for carrying out pesticides residues analysis. The samples of okra fruits were observed to be contaminated with monocrotophos, chlorpyriphos, dimethoate, cypermethrin, endosulfan and imidacloprid. Out of twenty samples of okra fruits for multi-residues analysis of pesticides, for Daurala market, Eight samples were observed to have residues of monocrotophos above MRL (0.20), seven samples below MRL and six samples were BDL (Below Detectable Limits). Nine samples were observed to have residues of chlorpyriphos above MRL (0.20), eight samples below MRL and three samples were BDL. Six samples were observed to have residues of dimethoate above MRL (2.00), seven samples below MRL and seven samples were BDL. Seven samples were observed to have residues of cypermethrin above MRL (0.02), nine samples below MRL and four samples were BDL. Ten samples were observed to have residues of endosulfan above MRL (2.00), seven samples below MRL and three samples were BDL. Five samples were observed to have residues of imidacloprid above MRL (0.05), nine samples below MRL and six samples were BDL (Table-1).

For Lawer market, thirteen samples were observed to have residues of monocrotophos above MRL (0.20), five samples below MRL and two samples were BDL. Eleven samples were observed to have residues of chlorpyriphos above MRL (0.20), six samples below MRL and three samples were BDL. Nine samples were observed to have residues of dimethoate above MRL (2.00), eight samples below MRL and three samples were BDL. Eight samples were observed to have residues of cypermethrin above MRL(0.20), nine samples below MRL and three samples were BDL. Fourteen samples were observed to have residues of endosulfan above MRL (2.00), four samples below MRL and two samples were BDL. Eleven samples were observed to have residues of imidacloprid above MRL (0.05), five samples below MRL and four samples were BDL (Table-1). For Kesarganj market, seven samples were observed to have residues of monocrotophos above MRL (0.20), ten samples below MRL and three samples were BDL. Six samples were observed to have residues of chlorpyriphos above MRL (0.20), nine samples below MRL and five samples were BDL. Eight samples were observed to have residues of dimethoate above MRL (2.00), nine samples below MRL and three samples were BDL. Six samples were observed to have residues of cypermethrin above MRL (0.20), nine samples below MRL and five samples were BDL. Eight samples were observed to have residues of endosulfan above MRL (2.00), eight samples below MRL and four samples were BDL. Seven samples were observed to have residues of imidacloprid above MRL (0.05), six samples below MRL and seven samples were BDL (Table-1).For Partapur market, nine samples were observed to have residues of monocrotophos above MRL (0.20), eight samples below MRL and three samples were BDL. Eight samples were observed to have residues of chlorpyriphos above MRL (0.20), eight samples below MRL and four samples were BDL. Six samples were observed to have residues of dimethoate above MRL (2.00), ninesamples below MRL and five samples were BDL. Eight samples were observed to have residues of cypermethrin above MRL (0.20), nine samples below MRL and three samples were BDL. Eight samples were observed to have residues of endosulfan above MRL (2.00), seven samples below MRL and five samples were BDL. Eight samples were observed to have residues of imidacloprid above MRL (0.05), eight samples below MRL and four samples were BDL (Table-1).

For Daurala market 45 replicates were found to be pesticide residues exceeding MRL. For Lawer market 66 replicates were found to pesticide residues exceeding MRL. For Kesarganj market 42 replicates were found to be pesticide residues exceeding MRL. For Partapur market 47 replicates were found to be pesticide residues exceeding MRL (Fig. 1).

ISSN: 2277-9655
Impact Factor: 4.116

Table –1. MRL value of insecticides for okra fruit

S.N.	Insecticides	MRL (ppm)
1.	Monocrotophos	0.20
2.	Chlorpyriphos	0.20
3.	Dimethoate	2.00
4.	Cypermethrin	0.20
5.	Endosulfan	2.00
6.	Imidacloprid	0.05

(Arora, 2008)

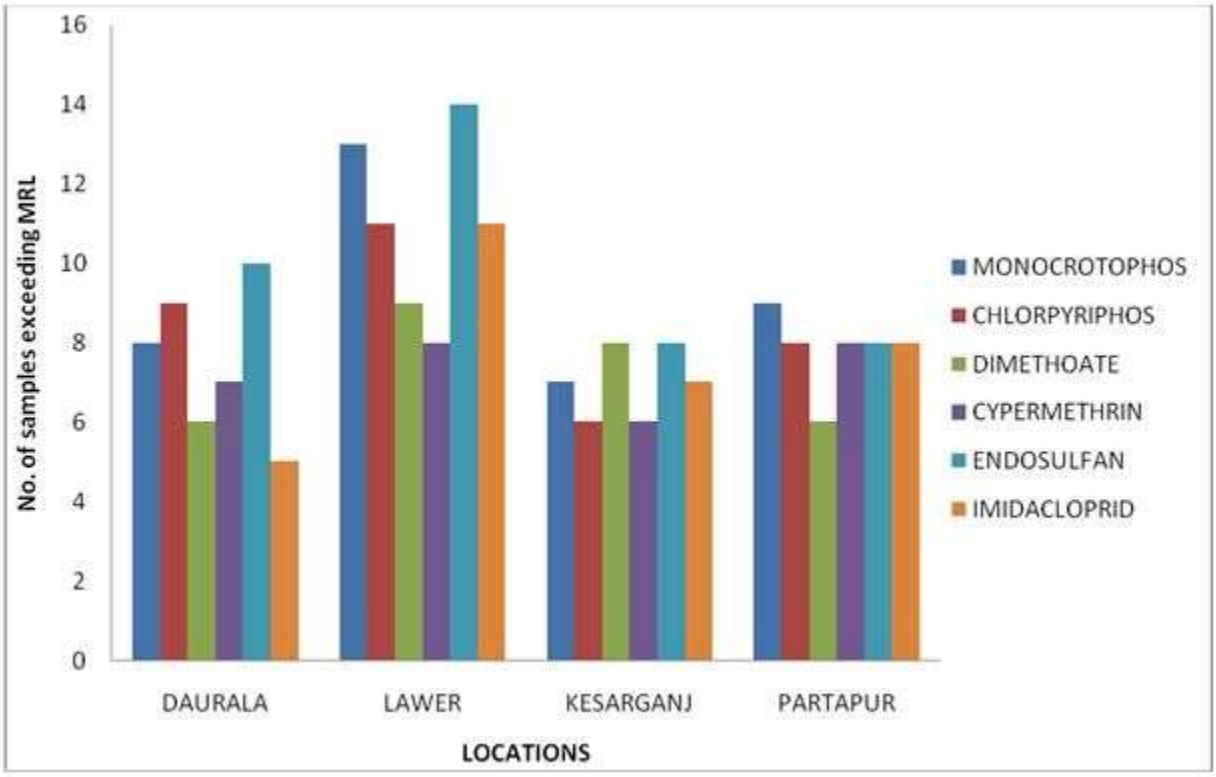

Fig. 1- Samples with pesticides residues exceeding MRL in different locations of Meerut Region

DISCUSSION

The sample of okra fruits collected for multi-residual analysis from for markets of Meerut district, viz. Daurala, Lawer, Kesarganj and Partapur, monitoring of pesticides (monocrotophos, chlorpyriphos, dimethoate, cypermethrin, endosulfan and imidacloprid) residues in farmgate samples (80) to above markets, the farmgate vegetable samples have been done by several workers in different parts of India, however many of them recorded pesticide residues above and below MRL.

The residues in Daurala market was detected to the range of 0.02-5.34ppm, out of twenty contaminated okra fruit samples were 8, 9, 6, 7, 10 and 5 exceeded the MRL and 6, 8, 7, 9, 7 and 9 samples were below MRL values of monocrotophos (0.20), chlorpyriphos (0.20), dimethoate (2.00), cypermethrin (0.20), endosulfan (2.00) and imidacloprid (0.05ppm), respectively. The present observations are in agreement with those of Kole *et al*. (2002) who

reported the range of 0.01-1.26ppm. Out of 12 contaminated okra fruit samples, four were exceeded the MRL values of cypermethrin and monocrotophos (0.20ppm), whereas two samples were found to contain cypermethrin (0.5ppm), the minimum waiting period allowed for cypermethrin and monocrotophos treated crops is only 1 day. According to Kumawat *et al*. (2000) initial deposit of 0.42ppm of monocrotophos was obtain in/on okra fruits from 0.036% sprays, the residues were translocated to the extent of 0.19ppm at three days intervals, which were well below the MRL of 0.20ppm, and the residues reached 0.11ppm at six days of spray which remain undetected after nine days of spray.

The residues in okra fruit samples collected from Lawer markets was detected to the range of 0.01-6.13ppm, out of twenty contaminated okra fruit samples 13, 11, 9, 8, 14 and 11 were exceeded the MRL and 5, 6, 8, 9, 4 and 5 samples were below MRL values of monocrotophos (0.20), chlorpyriphos (0.20), dimethoate (2.00), cypermethrin (0.20), endosulfan (2.00) and imidacloprid (0.05ppm), respectively. Shah *et al*. (2000) reported out of 15 okra fruit samples were contaminated with cypermethrin (1) dimethoate (2) and monocrotophos (4), to have residues 0.040, 0.027-0.052 and 0.033-0.851 mg/kg below MRL 0.20, 2.00 and 0.20ppm respectively, also reported by Khan *et al*. (1999) initial deposit of 1.31 mg/kg of cypermethrin, okra fruit dissipated to 0.78, 0.50, 0.17 and 0.05 mg/kg at 1, 3, 7 and 10 days respectively, corresponding dissipation was 40-46, 61.83-87.02 and 96.18 per cent, reported safe waiting period of 5.81 days when cypermethrin was sprayed on okra crop.

The residues in collected samples from Kesarganj market was detected to have residues in the range of 0.01-6.04ppm, out of twenty contaminated samples 7, 6, 8, 6, 8 and 7 were exceeded MRL and 10, 9, 9, 9, 8 and 6 samples were below MRL values of monocrotophos (0.20), chlorpyriphos (0.20), dimethoate (2.00), cypermethrin (0.20), endosulfan (2.00) and imidacloprid (0.05ppm), respectively. Among the 30 okra fruit samples, one each with cypermethrin (2.3) and endosulfan (2.21) exceeded the MRL of 0.20 and 2.00ppm respectively, the residues of cypermethrin was to the tune of 0.09 and 1.8 mg/kg (Chandrasekaran *et al*., 1997). These findings also presented by Deka *et al*. (2005) five samples of okra fruit contaminated with cypermethrin (3) and dimethoate (2), they have residues 0.009-0.012 and 0.007-0.013 mg/kg below MRL 0.20 and 2.00ppm, respectively.

Reported that to initial deposit of dimethoate @ 0.06%, 2.93 mg/kg were detected, this degraded to 0.31 mg/kg by 10 days after five spraying on okra crop. The residues of 2.45, 1.90, 0.83 and 0.31 mg/kg were recorded after 1, 3, 7 and 10 days, respectively, the residues dissipated to 16.38, 35.16, 71.67 and 89.42% on 1, 3, 7 and 10 days respectively, to required waiting period of two days for safe consumption at harvest (Khan *et al*., 1999).

The residues in okra fruit samples at Partapur market was detected to have residues in the range of 0.01-5.20ppm, out of twenty contaminated samples 9, 8, 6, 8, 8 and 8 were exceeded MRL and 8, 8, 9, 9, 7 and 8 samples were below MRL values of monocrotophos (0.20), chlorpyriphos (0.20), dimethoate (2.00), cypermethrin (0.20), endosulfan (2.00) and imidacloprid (0.05ppm), respectively. Singh et *al.* (2004) who reported deposit of cypermethrin on okra fruit were 0.274 and 0.382 mg/kg when the application of insecticides used @ 50 and 100 gram ai ha^{1}, the safe waiting period was worked out as 2 days. Deen *et al*. (2009) reported dose of cypermethrin dissipated by 39.4, 56.4, 72.5 and 85% at 2, 4, 6 and 9 days after treatment, respectively. Half life and safe waiting period were 3.3 and 4.7 days respectively in case of endosulfan deposit of 5.33 µg/g while dissipated by 29.5, 42.5, 66.0 and 81.0% at 2, 4, 6 and 9 days of treatment, respectively. Residues reached below MRL values for 2.0 µg/g in 4 days, half life and safe waiting period were 3.8 and 8.3 days, respectively. The reduction of residues was observed in the range of 16-37% and 16-24% for cypermethrin and endosulfan, respectively.

Goswami *et al.* (2002) recorded 0.057 and 0.081ppm residues in lower and higher dosage, respectively at 20 days after the insecticidal treatment. According to Chahal *et al*. (2006) recorded monocrotophos level on brinjal fruits were 1.93 and 2.59 mg/kg at single and double recommended rate respectively, dissipated to non-detectable level and 0.24 mg/kg respectively in four days.

Collected farmgate samples (20) from Daurala market 40, 45, 30, 35, 50 and 25% samples were exceeded MRL and 30, 40, 35, 45, 35 and 45% samples were recorded below their respective MRL values of monocrotophos (0.20), chlorpyriphos (0.20), dimethoate (2.00), cypermethrin (0.20), endosulfan (2.00) and imidacloprid (0.05ppm), respectively. The residues were found in 79.17% samples of okra, 43.75% samples of brinjal, 73.68% samples of tomato and in 91.30% samples of cauliflower (0.35-2.10ppm), the residues of monocrotophos were found in 75%

samples of okra, 78.12% samples of brinjal, 68.42% samples of tomato and 78.76% samples of cauliflower (Shukla *et al.*, 1996). According to Chahal *et al.* (1997) who reported that farmgate samples of vegetables for insecticide residues, revealed that 67% of samples were contaminated with insecticides last sprayed and about 7% of these had residues above their respective MRL values.

From lower market, 65, 55, 45, 40, 70 and 55% samples contained exceeded MRL and 25, 30, 40, 45, 20 and 25% samples had residues below MRL values of monocrotophos (0.20), chlorpyriphos (0.20), dimethoate (2.00), cypermethrin (0.20), endosulfan (2.00) and imidacloprid (0.05ppm), respectively. Anonymous,(2001) reported out of 40 farmgate samples of vegetables including brinjal, okra, tomato, chilli, cabbage, pea, reddish, spinach and singhara, collected from different locations of Kanpur city, on an average 65% vegetable samples were found contaminated with either insecticides viz. lindane, endosulfan and cypermethrin, out of contaminated samples, 12.5% vegetable samples contained pesticides residues more than their respective MRL values. The reported residues of 0.58 mg/kg deltamethrin in okra fruit declined by 56.89% in 3 days, 82.75%in 5 days and reached to non-detectable limit after 7 days, okra fruits were safe for consumption after 5 days of insecticidal application (Dixit *et al.*, 2005).

In the samples collected from Kesarganj and Partapur markets, each (20), 35, 30, 40, 30, 40 and 35% in Kesarganj market and 45, 40, 30, 40, 40 and 40% samples in Partapur market, residues exceeded MRL and 50, 45, 45, 45, 40 and 30% and 40, 40, 45, 45, 35 and 40% samples, respectively contained residues below MRL values of monocrotophos (0.20), chlorpyriphos (0.20), dimethoate (2.00), cypermethrin (0.20), endosulfan (2.00) and imidacloprid (0.05ppm), respectively. Similar findings were also reported, when 84 farmgate samples of vegetable were analyzed, revealed that 26% samples contained residues above MRL values. The contamination was mainly with organophosphate followed by synthetic pyrethroid and organochlorines. Among organophosphate, residues of monocrotophos, quinalphos and chlorpyriphos exceeded the MRL values in 23% samples. Residues of monocrotophos were higher than MRL values in three samples of brinjal and one sample of okra and quinalphos one sample in okra. Among synthetic pyrethroid, cypermethrin was the major contaminant and its residues exceeded MRL values in one sample each of brinjal and okra (Kumari *et al.*, 2004).

According to Dethe *et al.* (1995) endosulfan, cypermethrin dimethoate, monocrotophos and mancozeb, detectable level of residues were observed in 33.3% of tomato (endosulfan, dimethoate and monocrotophos), 73.3% of brinjal (endosulfan, cypermethrin, fenvalerate, quinalphos, dimethoate and monocrotophos), 14.3% of okra (endosulfan), however, the level of pesticides residues were lower than recommended maximum residue limit.

The present pattern of insecticides detected in the vegetables samples collected from the four sampled areas does not seem to contribute toward excessive residues. However, the use of insecticides should be need based only and recommended insecticides should be applied as and when required.
Further to safeguard the consumer's interest, proper waiting period must be practiced by the producer before marketing vegetables.

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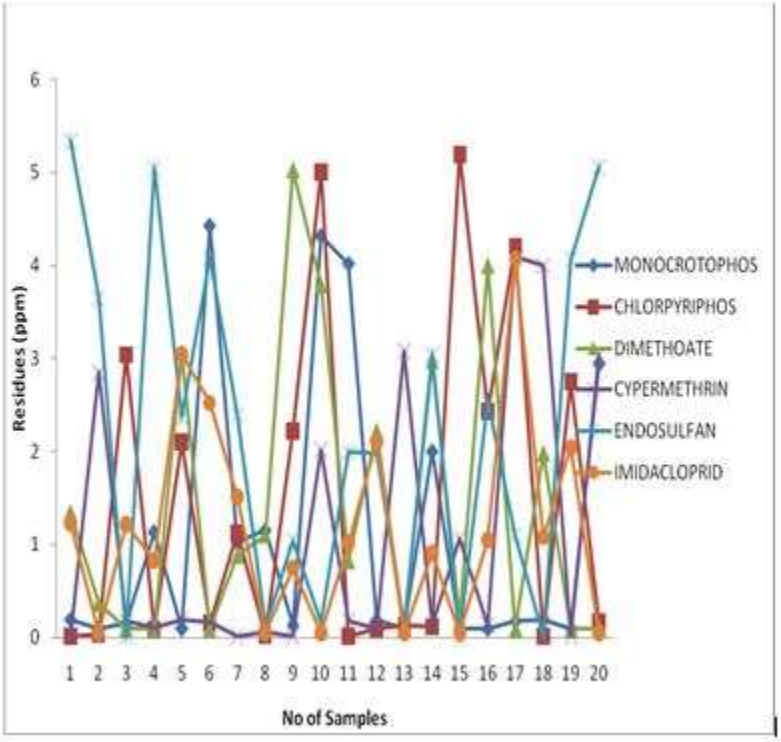

Fig. 2- Insecticides residues of okra farmgate samples from Daurala

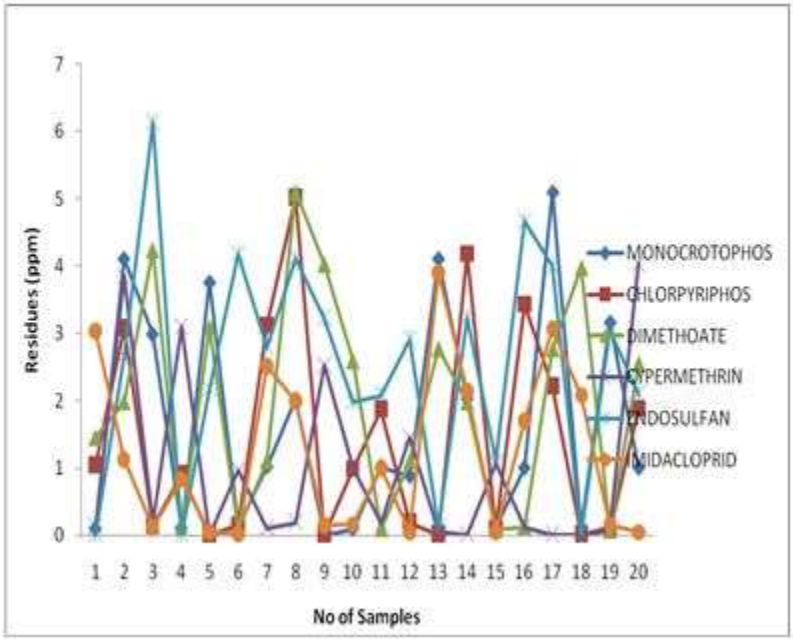

Fig. 3- Insecticides residues of okra farmgate samples from Lawer

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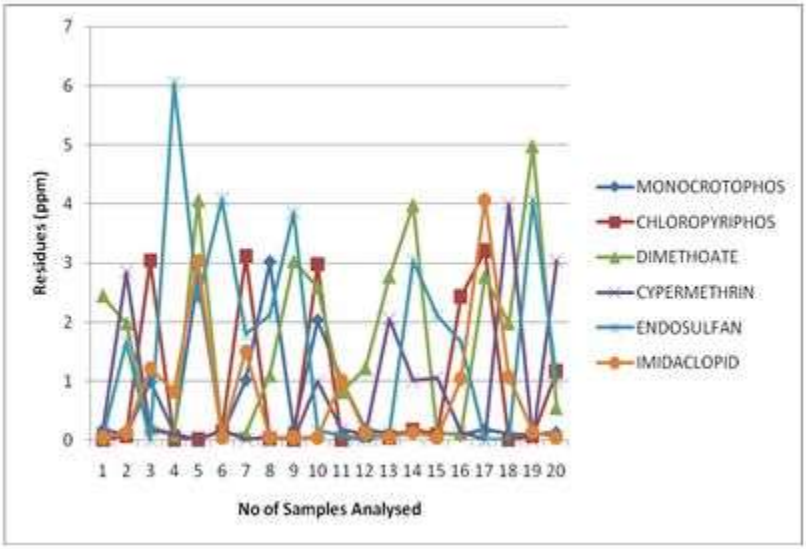

Fig – 4. Insecticides residues of okra farmgate samples from Kesarganj

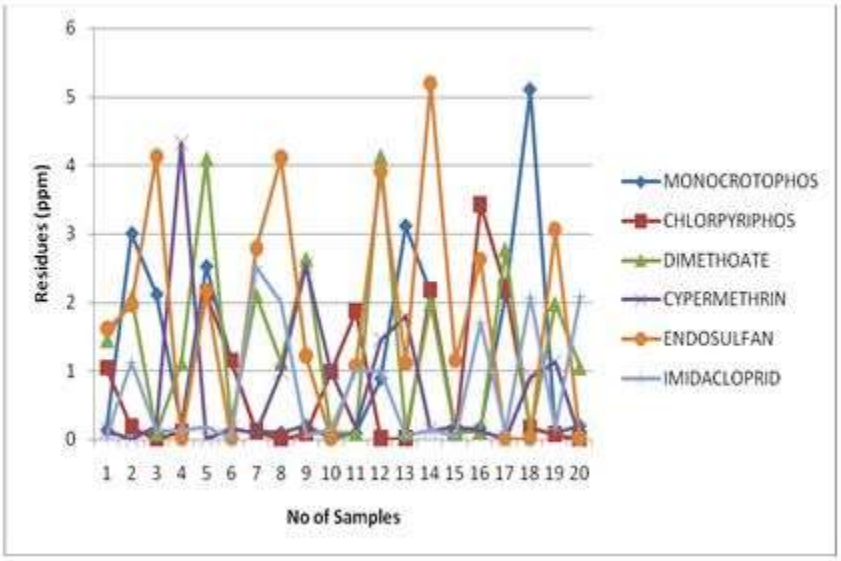

Fig. 5. Insecticides residues of okra farmgate samples from Partapur

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Table – 2. Insecticides residues of okra farmgate samples from Daural, Lawar, Kesarganj and Partapur

S.N.	Insecticides Residues (ppm)																							
	Monocrotophos(0.20)*				Chlorpyriphos (0.20)*				Dimethoate (2.00)*				Cypermethrin (0.20)*				Endosulfan (2.00)*				Imidacloprid (0.05)*			
	Daurala	Lawar	Keshargan j	Partapur	Daurala	Lawar	Keshargan j	Partapur	Daurala	Lawar	Keshargnj	Partapur	Daurala	Lawar	Kesharganj	Partapur	Daurala	Lawar	Keshargan j	Partapur	Daurala	Lawar	Keshargnj	Partapur
1	0.19	BDL	0.18	0.13	BDL	1.04	BDL	1.04	1.34	1.45	2.45	1.45	BDL	0.13	0.11	0.13	5.34	0.01	0.01	1.61	1.23	3.03	BDL	BDL
2	BDL	4.11	BDL	3.00	0.03	3.08	0.08	0.18	0.36	1.98	1.98	2.08	2.85	3.85	2.85	BDL	3.65	2.66	1.66	1.96	BDL	1.13	0.13	1.13
3	0.15	2.99	0.99	2.11	3.04	0.14	3.04	BDL	BDL	4.24	0.24	BDL	0.17	0.19	0.17	0.19	BDL	6.13	BDL	4.13	1.22	0.12	1.22	0.12
4	1.13	0.10	0.13	0.19	0.08	0.91	0.01	0.11	BDL	0.11	BDL	1.11	0.10	3.10	0.10	4.33	5.04	BDL	6.04	BDL	0.83	0.83	0.83	0.13
5	BDL	3.76	2.76	2.52	2.10	BDL	BDL	2.13	3.06	3.09	4.06	4.09	0.19	BDL	BDL	BDL	2.37	2.17	2.37	2.17	3.04	BDL	3.04	0.19
6	4.43	0.14	BDL	1.14	0.15	0.15	0.15	1.15	BDL	BDL	BDL	0.22	0.16	0.96	0.16	0.16	4.08	4.18	4.08	BDL	2.52	0.02	BDL	0.02
7	1.02	1.02	1.02	0.12	1.12	3.12	3.12	0.12	0.88	1.10	0.10	2.10	BDL	0.10	BDL	0.10	2.39	2.79	1.79	2.79	1.51	2.51	1.51	2.51
8	1.15	2.02	3.02	BDL	0.02	5.02	0.02	BDL	1.10	5.10	1.10	1.12	0.06	0.19	0.06	0.99	0.11	4.11	2.11	4.11	BDL	1.99	BDL	1.99
9	0.13	0.01	0.19	0.19	2.22	BDL	BDL	0.08	5.03	4.03	3.03	2.63	BDL	2.53	BDL	2.53	1.05	3.22	3.85	1.22	0.75	0.15	0.05	BDL
10	4.32	BDL	2.03	0.01	5.01	0.98	2.98	0.98	3.79	2.59	2.59	BDL	2.02	1.00	0.99	1.00	0.16	1.98	0.16	BDL	BDL	0.16	BDL	0.16
11	4.02	1.02	1.02	BDL	BDL	1.87	BDL	1.87	0.82	BDL	0.82	BDL	0.18	0.18	0.18	0.18	1.99	2.08	0.08	1.07	1.02	1.00	1.02	1.00
12	0.18	0.88	0.18	0.88	0.09	0.19	0.09	BDL	2.22	1.10	1.22	4.13	0.09	1.45	0.09	1.45	1.98	2.91	BDL	3.91	2.11	BDL	0.13	0.99
13	BDL	4.11	0.10	3.11	0.13	0.01	0.04	0.01	BDL	2.77	2.77	0.10	3.08	0.05	2.05	1.80	0.12	0.12	0.12	1.11	BDL	3.90	BDL	BDL
14	2.00	2.03	0.13	2.13	0.11	4.18	0.18	2.18	2.98	1.98	3.98	1.98	0.13	BDL	1.00	0.13	3.04	3.24	3.04	5.20	0.91	2.14	0.14	0.14
15	BDL	0.12	0.12	0.11	5.20	0.09	0.09	0.09	0.18	BDL	BDL	BDL	1.06	1.06	1.06	0.19	BDL	1.11	2.11	1.15	BDL	BDL	BDL	BDL
16	BDL	1.00	BDL	0.16	2.43	3.43	2.43	3.43	4.00	0.12	0.12	0.12	0.13	0.13	0.13	0.13	2.55	4.66	1.66	2.63	1.05	1.70	1.05	1.70
17	0.17	5.10	0.18	2.10	4.21	2.21	3.21	2.21	BDL	2.77	2.77	2.77	4.09	BDL	BDL	BDL	1.09	3.99	BDL	0.01	4.07	3.07	4.07	0.11
18	0.19	0.10	0.10	5.10	BDL	BDL	BDL	0.17	1.98	3.98	1.98	BDL	4.00	0.01	4.00	0.91	BDL	BDL	BDL	BDL	1.08	2.08	1.08	2.08
19	BDL	3.16	BDL	BDL	2.75	0.07	0.07	0.07	BDL	BDL	4.98	1.98	BDL	0.13	BDL	1.13	4.06	3.06	4.06	3.06	2.04	0.15	0.15	0.15
20	2.94	1.00	0.13	0.19	0.17	1.88	1.17	BDL	BDL	2.55	0.55	1.05	3.04	4.04	3.04	BDL	5.06	2.10	1.00	BDL	BDL	BDL	BDL	2.09
Number of samples																								
>MRL	8	13	7	9	9	11	6	8	6	9	8	6	7	8	6	8	10	14	8	8	5	11	7	8
<MRL	6	5	10	8	8	6	9	8	7	8	9	9	9	9	9	9	7	4	8	7	9	5	6	8
BDL	6	2	3	3	3	3	5	4	7	3	3	5	4	3	5	3	3	2	4	5	6	4	7	4