

## Risk Assessment of Pesticide Residues Through Selected Vegetables

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### ABSTRACT

Samples of three selected summer and winter vegetables viz, cucumber, okra and cauliflower were collected from field and analysed for pesticide residues. Extraction of samples was done using QuEChERS method and was analysed using gas chromatography using GC-ECD and GC-FTD detectors for the presence of organochlorine, organophosphates and synthetic pyrethroids. Among the three vegetables analyzed, cauliflower was found to be most contaminated (47.5%) followed by okra (40.0%) and cucumber (22.5%) with low but measurable amounts of residues. Organochlorines were not detected in any of the samples. For risk assessment under Indian conditions, theoretical maximum residue contribution (TMRC) was determined for both urban and rural respondents and a comparison with maximum permissible intake (MPI) for Indian women with standard body weight of 55 Kg was done. The TMRC obtained for all the income groups in rural and urban respondents was significantly lower than MPI.

**Keywords:** Pesticides; Vegetables; Risk Assessment; Household Processing

### Introduction

Vegetables are the tender edible parts of the plants and are an essential part of human diet in India. These are low in fat and provide essential vitamins, minerals and fibre for body growth. In order to suffice its population growth and challenge to feed them, India adopts an extensive system of farming. This kind of farming practice includes use of agents like pesticides, fertilizers that help to increase yield. Excess application of pesticides results in the concentration of the pesticides in the vegetables rendering them unfit for consumption due to the hazardous nature (Tomer et al., 2015). Different processing techniques which can be applied feasibly at household level can also prove quite effective in reducing pesticide exposure (Cengiz&Certel, 2012, Kin &Huat, 201). Hence, the study was undertaken with the objective to observe the risk that the people in district Ludhiana were exposed. The impact of different processing techniques on pesticide residue was also observed.

## **Material and Methods**

In order to determine the dietary intake of the cucumber, okra and cauliflower, a total of 240 household women (120 from each group viz rural and urban) were surveyed for their diet pattern. The respondents were further divided in to three income groups namely, lower income group (LIG), middle income group (MIG) and higher income group (HIG) by cube root method. Household women from rural areas of district Ludhiana were divided into three income groups viz., LIG (farm size- 0-1.99 acres), MIG (farm size- 2-4.99 acres) and HIG (farm size- above 5 acres). On similar grounds, respondents randomly selected from urban localities were classified into three income groups namely, LIG - Plot size less than 100 square yards, MIG – plot size 101 to 200 square yards and HIG - plot size more than 200 square yards. To study the extent of contamination in vegetables, forty marketable size samples of each selected vegetable viz cucumber, okra and cauliflower (1 Kg each) were collected vegetable growing areas of district Ludhiana in the crop season during 2011-12.

The analysis of pesticides was carried out in Pesticide Residue Analysis Laboratory, Punjab Agricultural University, Ludhiana. Vegetable samples were extracted by QuEChERS method with slight modifications (Anastassiades *et al* 2003) and the analysis was done with gas chromatography (GC). The process of sample preparation and analysis has been discussed in detail in our previous study (Tomer *et al.*, 2014). Effect of processing on different pesticide residues in selected vegetables was observed through simulated studies. The three vegetables were spiked with different concentrations of pesticides and effect of processing treatments was observed and analysed by the method described above.

## **Results and Discussion**

### **Dietary Intake of Vegetables**

The survey was conducted in rural and urban areas of district Ludhiana. A total of 240 household women (120 from each group viz rural and urban) were surveyed for their diet pattern. The respondents were further divided in to three income groups namely, lower income group (LIG), middle income group (MIG) and higher income group (HIG) by cube root method. It was found that

the all the respondents consumed selected vegetables way below the suggested intake (Table1). As given in table 1, highest consumption for all the vegetables was observed to be in middle and higher income of urban household women in contrast to their rural counterparts except for okra. Consumption of okra was found more among the rural respondents of all the three classes.

### **Pesticide residue content in farmgate samples**

The study was conducted in District Ludhiana of Punjab. Vegetable samples were collected from villages lying in the three blocks from vegetable growing belt of District Ludhiana viz., Sudhar, Pakhowal and Malerkotla. A total of 120 Samples (40 samples of each vegetable) were collected randomly from these locations.

The standardization of analytical methods was done on the spiked samples (i.e. organochlorines, pyrethroids and organophosphorus insecticides) and followed by the analysis of test samples. Consistent recoveries of pesticides was found in more than 80% of samples at a concentration of 0.01, 0.05, 0.1mg kg<sup>-1</sup>(Table 2). The area of peak so obtained was compared with the area of standard and recoveries were calculated in terms of percentage. As depicted in Table 2, it can be seen that the pesticides detected in all the three samples were either organophosphate or pyrethroids. However, the concentration of pesticides detected in all the samples was below the maximum residue limits.

The analysis of cucumber samples revealed that several commonly used organophosphate and pyrethroid insecticides were present. Total contamination in cucumber was found to be only 22.5% out of the forty samples analysed. However, none of the pesticide exceeded MRL. Among the detected pesticides only malathion is recommended by package of practices, PAU. The presence of other insecticidal residues depicts the irresponsible use of insecticides by farmers.

On analysing okra samples obtained from farmers' field, forty percent of the samples were found to be contaminated. However, like in cucumber none of the samples exceeded maximum residue limits. Highest rate of contamination of okra samples was found to be with cypermethrin (Table 2) which is the recommended pesticide but all the samples had residue level under MRL (0.5 mg/Kg).

Chlorpyrifos, cypermethrin, methylparathion, deltamethrin and profenophos were the pesticides which were present in high concentration in the cauliflower. However, none of the samples were found to contain residues of any of these insecticides above their respective MRL's (Table 3). Many of the insecticides found in cauliflower are banned by the government which reflects the misuse of insecticides by farmers. Among the identified pesticides only quinalphos is allowed to be used on cauliflower.

### **Risk Assessment**

The maximum residue limit for a few pesticides detected in analysed samples has been prescribed by Codex. As discussed above, all the analysed samples had residues below the prescribed MRLs. However, MRL under Indian conditions is not available for majority of the pesticides. Hence, assessment of the risk of pesticide residues detected in the analysed vegetables was made on the basis of the total intake of pesticides on consumption of the vegetables in comparison to the acceptable daily intake (ADI). ADI of pesticides detected for each selected vegetable has been given in table 3, 4 and 5. To calculate the Maximum Permissible Intake (MPI) the ADI was multiplied with the average weight (55 kg) of an Indian woman. TMRC for each pesticide was calculated as a product of vegetable consumption in grams and pesticide level detected in that vegetable.

As indicated in Table 4, TMRC from cucumber of none of the respondents either from rural or urban background exceeded the Maximum permissible intake and was found to be significantly lower. None of the pesticide posed any health risk to the consumers of any income group. The exposure to pesticides increased as the income group increased and this trend was observed in both rural and urban respondents. Highest intake was observed for  $\lambda$ -cyhalothrin but the TMRC was still significantly lower.

Table 5 gives the TMRC of different pesticides through okra and its comparison to MPI. As indicated in Table 4, TMRC from okra of none of the respondents either from rural or urban background exceeded the Maximum permissible intake. Hence, present results indicate that none of the pesticide posed any health risk to the consumers of any income group. Similar results were obtained for cauliflower as well (Table 6). TMRC from cauliflower of none of the respondents either from rural

or urban background exceeded the MRI and was found to be significantly lower. Highest intake was observed for cypermethrin. However, none of the pesticide posed any health risk to the consumers of any income group.

### **Discussion**

Vegetables are short duration crops and in subtropical climate are prone to insect pest infestations at both growth and maturity stages. To save the crops from economic losses, it is necessary to spray pesticides on crop plants repeatedly during the entire period of growth and development (Jeyanthi and Kombairaju, 2005).

It has also been reported that pesticides are repeatedly being sprayed on vegetables several times during growing period, so it is the need of the time to monitor the usage to assess the environmental load of pesticide residues (Gehad, Saleh & Ibrahim, 2012). Therefore, analysis of pesticide residues in food and their removal by traditional processing have become essential requirement (Margriet *al* 2002, Randhawa et al., 2008; Satpathy 2012).

Analysis of farmgate samples of cucumber, okra and cauliflower revealed contamination from organophosphates and synthetic pyrethroids and none of them exceeded MRL. However, trend obtained in present study was different from that in 2010 in which organochlorine pesticides like DDT, endosulfan, aldrin etc. were also detected in cauliflower (Mandal and Singh, 2010). With the ban on highly persistent organochlorine pesticides in agriculture, there has been a decline in organochlorine residues in food. However, the incidence of contamination by less persistent but more toxic organophosphates and carbamate pesticides are on the rise in Punjab (Tiwana *et al* 2007). TMRC for all the pesticides in selected vegetables was lower than maximum permissible intake. Takkar *et al* (2011) also found indoxacarb residues to be below the MPI and identified no health risk to Indian populace from Indoxacarb residues.

### **Summary**

A total of one hundred and twenty samples of three selected summer and winter vegetables viz, cucumber, okra and cauliflower were collected from vegetable growing villages of District Ludhiana and analysed for pesticide residues to determine the magnitude and frequency of pesticidal

contamination. Extraction of samples was done using QuEChERS(Quick Easy Cheap Effective Rugged Safe) method and was analysed using gas chromatography using GC-ECD and GC-FTD detectors for the three major chemical groups i.e. organochlorine, organophosphorus and pyrethroids. Residues were estimated by comparison of peak areas of the standards with spiked samples run under identical conditions. Among the three vegetables analyzed, cauliflower was found to be most contaminated (47.5%) followed by Okra (40.0%) and Cucumber (22.5%). with low but above the detectability limits. Organochlorines were not detected in any of the samples. Malathion in cucumber, cypermethrin on okra and quinalphos in cauliflower were the only pesticides detected that were recommended by PAU. Occurrences of residues of other insecticides reflect the misuse of insecticides on cauliflower. For comparison under Indian conditions, theoretical maximum residue contribution (TMRC) was calculated for both urban and rural respondents and then compared with maximum permissible intake (MPI) for Indian women with standard body weight of 55 Kg. The TMRC obtained for all the income groups in rural and urban respondents was significantly lower than MPI. For analysing the efficacy of household treatments, simulated trials were conducted. The vegetables were spiked with known concentration of pesticides as recommended in 'Package of practices' published by Punjab Agricultural University. Different processes commonly used at household level were applied and samples were extracted. Results obtained showed that processing treatments at household were quite effective in reducing pesticide residues in all three vegetables. Peeling was most effective treatment in cucumber and reduced malathion residues by 91.64 percent. In okra and cauliflower, pressure cooking followed by frying was most effective in reducing pesticide residues and cypermethrin and quinalphos residues were reduced by more than 80 percent in both the cases. Washing in tap water was least effective for all the three vegetables. Processing treatments performed at higher temperature were more effective for both cypermethrin residues in okra and quinalphos residues in cucumber.

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**Table 1: Consumption of selected vegetables by urban and rural household women**

Vegetable	Rural			Urban		
	LIG	MIG	HIG	LIG	MIG	HIG
Cucumber	20.94±19.35	26.72±18.05	28.37±18.09	18.68±16.75	28.88±16.52	38.14±14.53
Okra	18.60±18.38	24.08±18.41	26.20±19.08	8.50±7.8	17.28±13.2	23.24±16.27
Cauliflower	17.78±16.89	21.53±17.67	23.26±18.86	16.51±17.76	22.54±15.51	30.55±19.35

\* Values are given in terms of grams (Mean ± SD)

**Table 2: Recovery obtained for different pesticides in cucumber, okra and cauliflower at different spiking levels**

Substrate	Pesticide	Level of Spiking (mg/kg)	Percent Recovery
Cucumber	Malathion	0.05	91.77 ± 6.9
	Chlorpyrifos	0.05	87.17 ± 3.5
	λ-cyhalothrin	0.1	84.20 ± 3.6
	Cypermethrin	0.01	85.13 ± 3.9
	Ethion	0.01	85.90 ± 4.3
Okra	Chlorpyrifos	0.05	86.00 ± 4.0
	Cypermethrin	0.05	89.90 ± 7.4
	Malathion	0.05	88.73 ± 5.3
	Deltamethrin	0.05	90.53 ± 4.4
	triazophos	0.1	85.27 ± 2.4
	λ-cyhalothrin	0.1	87.87 ± 5.8
Cauliflower	Acephate	0.05	86.57 ± 3.1
	Chlorpyrifos	0.01	88.27 ± 4.7
	Cypermethrin	0.05	86.43 ± 4.2
	methyl parathion	0.1	85.67 ± 5.3
	Deltamethrin	0.05	87.47 ± 4.8
	Profenofos	0.1	88.37 ± 7.8
	Triazophos	0.1	85.07 ± 1.6
	cyhalothrin	0.05	83.23 ± 0.4
	β-cyfluthrin	0.1	89.37 ± 6.5
Fenpropathrin	0.1	89.57 ± 5.7	

**Table 3: Residues of various pesticides detected in various samples of selected vegetables**

Vegetable	Pesticide	n (%)	Mean ± SD	MRL (Year of Adoption)
Cucumber (N=40)	<b>Total</b>	<b>9 (22.5)</b>		
	Malathion	7 (17.5)	0.18 ± 0.21	0.2 mg/kg (2004)*
	Chlorpyrifos	5 (12.5)	0.10 ± 0.09	-
	λ-cyhalothrin	3 (7.5)	0.43 ± 0.11	0.5 mg/kg (2011)*
	Cypermethrin	4 (10.0)	0.18 ± 0.11	-
	Ethion	2 (5.0)	0.09 ± 0.04	-
Okra (N=40)	<b>Total</b>	<b>16 (40.0)</b>		
	Chlorpyrifos	6 (15.0)	0.47 ± 0.36	0.5 (2008)#
	Cypermethrin	12 (30.0)	0.49 ± 0.30	0.5 mg/kg (2009)*
	Malathion	4 (10.0)	0.27 ± 0.15	-
	Deltamethrin	4 (10.0)	0.28 ± 0.17	0.3 (2012)
	triazophos	6 (15.0)	0.05 ± 0.04	-
	λ-cyhalothrin	6 (15.0)	0.29 ± 0.21	0.3 mg/kg (2009)*
	Acephate	5 (12.5)	0.34 ± 0.32	-
Cauliflower (N=40)	<b>Total</b>	<b>19 (47.5)</b>		
	Chlorpyrifos	12 (30.0)	0.31 ± 0.23	-
	Cypermethrin	8 (20.0)	0.65 ± 0.28	1 mg/kg
	methyl parathion	2 (5.0)	0.09 ± 0.05	-
	Deltamethrin	7 (17.5)	0.08 ± 0.14	0.1 mg/kg (2004)*
	Profenofos	9 (22.5)	0.37 ± 0.15	-
	Triazophos	7 (17.5)	0.03 ± 0.02	-
	cyhalothrin	6 (15.0)	0.18 ± 0.22	2.0 mg/kg
	β-cyfluthrin	3 (7.5)	0.10 ± 0.06	0.2 (2012)
	Fenpropathrin	4 (10.0)	0.49 ± 0.40	-
	Quinalphos	9 (22.5)	0.07 ± 0.12	0.1 mg/kg

Mean values are in terms of mg/Kg

\* MRLs obtained from FAO/WHO 2013.

#MRLs obtained from European Union Database 2013

**Table 4: Intake of pesticide residues through Cucumber and its risk assessment**

Pesticides	Maximum permissible intake (MPI)	Theoretical Maximum Residue Contribution (TMRC) (µg·person <sup>-1</sup> ·day <sup>-1</sup> )							
		Rural				Urban			
		LIG	MIG	HIG	Total	LIG	MIG	HIG	Total
Malathion	16500	3.72	4.78	4.96	4.49	3.37	5.14	6.73	5.08
Chlorpyrifos	550	2.18	2.81	2.91	2.63	1.98	3.02	3.95	2.98
λ-cyhalothrin	1100	9.03	11.61	12.04	10.89	8.17	12.47	16.34	12.33
Cypermethrin	1100	3.77	4.85	5.03	4.55	3.42	5.21	6.83	5.15
Ethion	110	1.05	1.35	1.40	1.27	0.95	1.45	1.90	1.43
Health Risk		No	No	No	No	No	No	No	No

**Table 5: Intake of pesticide residues through Okra and its risk assessment**

Pesticides	Maximum permissible intake (MPI)	Theoretical Maximum Residue Contribution (TMRC) ( $\mu\text{g}\cdot\text{person}^{-1}\cdot\text{day}^{-1}$ )							
		Rural				Urban			
		LIG	MIG	HIG	Total	LIG	MIG	HIG	Total
Chlorpyrifos	550	8.93	11.28	12.22	10.81	4.23	7.99	10.81	7.68
Cypermethrin	1100	10.03	12.67	13.72	12.14	4.75	8.97	12.14	8.62
Malathion	16500	5.22	6.59	7.14	6.32	2.47	4.67	6.32	4.49
Deltamethrin	550	5.28	6.67	7.23	6.39	2.50	4.73	6.39	4.54
Acephate	550	6.38	8.06	8.74	7.73	3.02	5.71	7.73	5.49
$\lambda$ -cyhalothrin	1100	5.50	6.95	7.53	6.66	2.61	4.92	6.66	4.73
Triazophos	55	0.87	1.10	1.19	1.05	0.41	0.78	1.05	0.75
Health Risk		No	No	No	No	No	No	No	No

**Table 6: Intake of pesticide residues through Cauliflower and its risk assessment**

Pesticides	Maximum permissible intake (MPI)	Theoretical Maximum Residue Contribution (TMRC) ( $\mu\text{g}\cdot\text{person}^{-1}\cdot\text{day}^{-1}$ )							
		Rural				Urban			
		LIG	MIG	HIG	Total	LIG	MIG	HIG	Total
Chlorpyrifos	550	5.49	6.71	7.02	6.41	5.19	7.02	9.46	7.22
Cypermethrin	1100	11.66	14.25	14.90	13.61	11.01	14.90	20.08	15.33
methyl parathion	165	1.61	1.97	2.06	1.88	1.52	2.06	2.77	2.12
Deltamethrin	550	1.50	1.84	1.92	1.76	1.42	1.92	2.59	1.98
Profenophos	1650	6.64	8.12	8.49	7.75	6.27	8.49	11.44	8.73
$\beta$ -cyfluthrin	165	1.86	2.27	2.38	2.17	1.76	2.38	3.20	2.45
Triazophos	55	0.50	0.62	0.64	0.59	0.48	0.64	0.87	0.66
cyhalothrin	1100	3.29	4.02	4.21	3.84	3.11	4.21	5.67	4.33
Fenpropathrin	1650	8.73	10.67	11.16	10.19	8.25	11.16	15.04	11.48
Quinalphos	1650	2.63	3.21	3.36	3.06	2.48	3.36	4.52	3.45
Health Risk		No	No	No	No	No	No	No	No

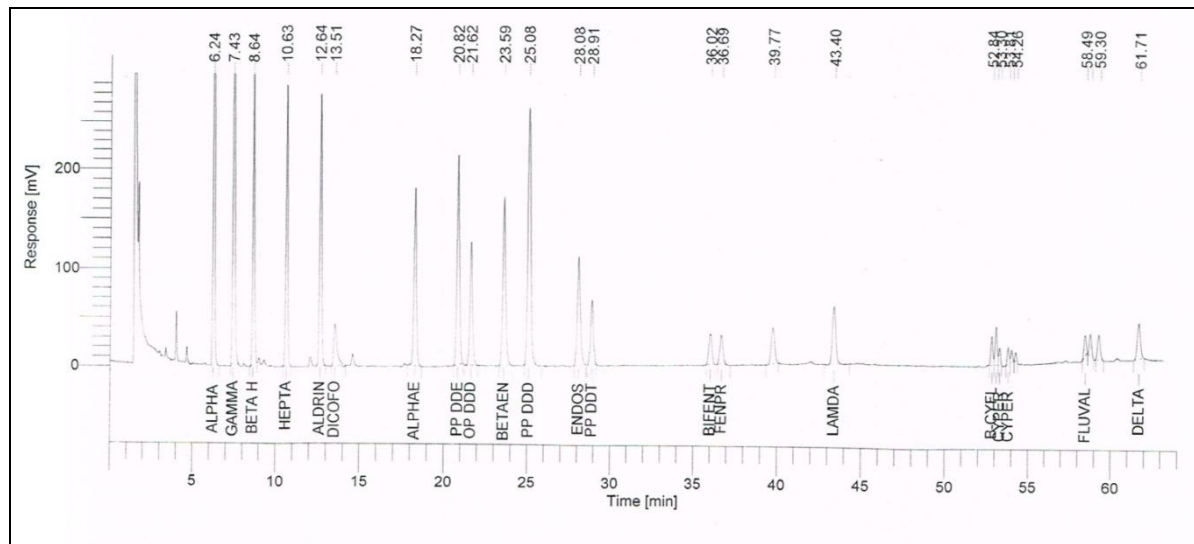


Figure 1: GC Chromatogram for standard mixture of organochlorines and synthetic pyrethroids