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# Persistent chlorinated pesticide residues in selected market vegetables of root and leaf origin

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**ABSTRACT**

*Aldrin, dieldrin, heptachlor and lindane pesticides analyzed in selected root and leaf vegetables. The concentration of total organochlorine pesticides (OCPs) was ranged between, <0.01-6.00 ng/g, with an average of 2.16±0.21 ng/g (wet wt.). The concentration of individual aldrin, dieldrin, heptachlor and lindane was 0.48±0.06 ng/g, 0.13±0.02 ng/g, 1.03±0.11 ng/g and 0.52±0.06 ng/g (wet wt.), respectively. The selected vegetables had residue levels, much below the recommended maximum residue limits (MRLs) set by European Commission and Indian government.*

**Key words**: Vegetables, Pesticides, Aldrin, Dieldrin, Heptachlor, Lindane.

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## INTRODUCTION

Organochlorine pesticides have been widely used in public health and agriculture production in developed and developing countries including India. Organochlorine pesticides (OCPs) are of much concern in the environment because of their prolonged persistence, long range transport nature, toxicity as well as bioaccumulative tendency [1]. Organochlorine pesticide exposure has been associated with human health risk of arthritis, skin disease, bone disorder, cancer and nerve disorder [2,3].

In India, organochlorine pesticides (OCPs) were produced and used in agricultural and public health, their indiscriminate use leads to accumulation in consumable agricultural products. With approximately, 85 TMT (thousand metric tonnes) annual pesticides production, India is the fourth largest producer of pesticides in the world after US, Japan and China. Domestic consumption of pesticides is about 50 TMT annually and 13-14 percent share of pesticides used for vegetables and fruits production [4,5]. The consumption of pesticides in India for agriculture is comparatively low (0.5 kg/ha), against 12.0, 7.0, 6.6, and 3.0 kg/ha in Japan, USA, Korea and Germany, respectively, and only 3.75% of global consumption [6]. During 2004-05, in West Bengal 4100 MT of technical grade pesticides was used on 5.123 million hectares of agricultural land.

Indian diet contains vegetables as important component in food, because majority of Indians are vegetarian and per capita consumption of vegetables is approximately 135 g per day. Therefore, information on pesticide residue in vegetables is very important for human health. This paper presents the results of a study carried out on aldrin, dieldrin heptachlor and lindane residue levels in selected vegetables (*Beta vulgaris* L., *Coriandrum sativum* L. and *Trigonella foenum-graecum* L.) from Kolkata markets and concentration of pesticides in vegetables was compare with recommended maximum residual levels (MRLs) set by European commission [7] and Food Safety and Standard Authority of India [8].

## MATERIALS AND METHODS

***Chemicals and Solvents***

Sodium sulphate, silver nitrate, potassium hydroxide, activated charcoal and sulphuric acid, acetone, methanol, dichloromethane, and hexane were procured from Merck India. Pesticide standard solutions were obtained from Supelco (Sigma, USA). Silica gel 60 was purchased from Sigma-Aldrich.

***Sampling and Pesticide Extraction***

The vegetables selected for analysis were, sugar beet (*Beta vulgaris* L.), coriander leaves (*Coriandrum sativum* L.) and maithee leaves (fenugreek) (*Trigonella foenum-graecum* L.). Samples were collected from local markets of Kolkata in clean polyethylene bags; labelled and transported ice preserved to the laboratory and kept in -20$^0$C till extraction.

After washing the samples were with deionised distilled water, dried on filter paper and cut into small pieces with the help of grater. 20-30 g of thoroughly mixed sample was grinded with 10-15 g anhydrous sodium sulphate to absorb water contents. The grinded sample was extracted with 50 ml acetone on mechanical shaker for one hr. The acetone extract was filtered by employing vacuum suction with all glass filtration assembly, and the process was repeated three times for complete extraction. The extracted filtrate was concentrated to ≈25 ml using Rotatory Vacuum evaporator (Buchi, Germany) and subjected to liquid-liquid portioning with hexane in separatory funnel. Hexane layer containing pesticide residues was collected passing through sodium sulphate to remove traces of water contents. Aqueous phase was again subjected to hexane extraction (three times) for leftover residues. Pooled hexane fractions were concentrated to 10 ml.

***Sample Extract Clean-Up***

Primarily, pigment were removed were by passing through glass column containing activated charcoal and anhydrous sodium sulphate. Then, silica gel column chromatography was performed to remove other interfering aliphatic and polar compounds. Briefly, glass column (300 mm x 10 mm) was packed from bottom to up with clean glass wool, 10 g activated silica gel, and 2.5 g anhydrous sodium sulphate. The column was pre-rinsed with 50 ml n-hexane before sample was loaded, then sample was loaded with three rinsing of extract flask with 2 ml hexane. The elution of analytes was subsequently carried out using 100 ml hexane and concentrated to 2.0 ml. The extract was transferred to auto sampler vial and sealed for quantification using gas chromatograph equipped with an electron capture detector (GC-ECD).

***Pesticide Analysis and Quantification***

Identification and quantification of aldrin, dieldrin, heptachlor and lindane compounds was carried out using Varian gas chromatograph (Star 3400cx, Australia) equipped with $^{63}$Ni-ECD. Separation of OCP compounds was done on a RTX-5 capillary column (0.25 mm and 30 m and 0.5 µm film of 5% diphenyl-95% dimethylpolysiloxane). The column oven temperature program was as: oven temperature was initially at 170$^0$ C and increases to 220$^0$ C (@ 7$^0$ C min$^{-1}$), then ramped to 250$^0$ C at 5$^0$ C min$^{-1}$ and held for 7.0 min. The injector and detector temperature were maintained at 250$^0$ C and 325$^0$ C, respectively. A purified laboratory grade nitrogen gas was used as carrier at the flow rate of 1.0 ml/min.

***Analytical Quality Control***

Appropriate quality assurance quality control (QA/QC) analysis was performed. A procedural blank was run alongwith each batch of real samples to check the cross contamination and interferences. The blank concentrations were <MDL (method detection limit). Certified reference standard solutions (Sigma, USA) were used for calibration of instrument. Standard deviation of random duplicate sample analysis was <±5%. Linear calibration curves was obtained with the $r^2$ value of 0.999 and calibration verification standard deviation was <±5%. Resolved peaks were integrated using inbuilt Varian Star workstation software. The concentrations of target compounds were determined by external standard method using the peak area of the samples and the five level calibration curves of the standards. The peak identification was conducted by the accurate retention time of each standard. Matrix spiked recovery study was undertaken by spiking the known working standard solutions of OCPs with samples, which were extracted and analyzed in the same way as the real samples. The matrix spiked recoveries were in range of 76-124% (± 9-18%) for studied compounds. Each sample was analysed in duplicate and the average value was used in calculations. The results of the analysis are reported in ng/g wet wt. A reporting limit of > 0.01 ng/g wet wt was

taken for calculation. Levels below reporting limit or below MDL (<0.01 ng/g wet wt) were taken as zero (0) in the calculations.

## RESULTS AND DISCUSSION

A total of 23 samples of different vegetables (*Beta vulgaris* L., *Coriandrum sativum* L. and *Trigonella foenum-graecum* L.) were analysed for persitent organochlorine pesticide compounds (aldrin, dieldrin, heptachlor and lindane). Total concentration of ∑OCPs was ranged between <0.01 to 6.00 ng/g (wet wt) with the mean of 2.16±0.21 ng/g (Table 1).

**Table 1: Concentrations (range and mean) of aldrin, dieldrin, heptachlor and lindane in selected vegetables**

| Pesticides | Min | Max | mean | SD | SE* | % |
|---|---|---|---|---|---|---|
| Aldrin | <0.01 | 1.58 | 0.48 | 0.48 | 0.06 | 22 |
| Dieldrin | <0.01 | 0.80 | 0.13 | 0.16 | 0.02 | 6 |
| Heptachlor | <0.01 | 2.80 | 1.03 | 0.83 | 0.11 | 48 |
| Lindane | <0.01 | 1.59 | 0.52 | 0.46 | 0.06 | 24 |
| ∑OCPs | <0.01 | 6.00 | 2.16 | 1.61 | 0.21 | 100 |

**SE (standard error) =SD/√n*

The concentration of individual aldrin, dieldrin, heptachlor and lindane in selected vegetable samples was 0.48±0.06 ng/g (wet wt), 0.13±0.02 ng/g (wet wt), 1.03±0.11 ng/g (wet wt), and 0.52±0.06 ng/g (wet wt), respectively. Among the studied pesticides heptachlor alone accounts 48 percent of the total OCPs, followed by lindane (24%), aldrin (22%) and dieldrin (6%) (Figure 1). The concentration of ∑OCPs in sugar beet, fenugreek and coriander ranged between <0.01-4.38 ng/g, <0.01-4.16 ng/g and <0.01-6.00 ng/g, respectively with the mean values of 2.00±0.51 ng/g, 2.08±0.64 ng/g and 2.44±0.69 ng/g, respectively (Table 2). Comparatively, aldrin, dieldrin, heptachlor and lindane concentrations were higher in coriander leaves (Figure 2). The lowest concentration of aldrin/dieldrin, heptachlor and lindane was observed in fenugreek and sugar beet, respectively. The observed contamination pattern of vegetables was coriander > fenugreek > sugar beet.

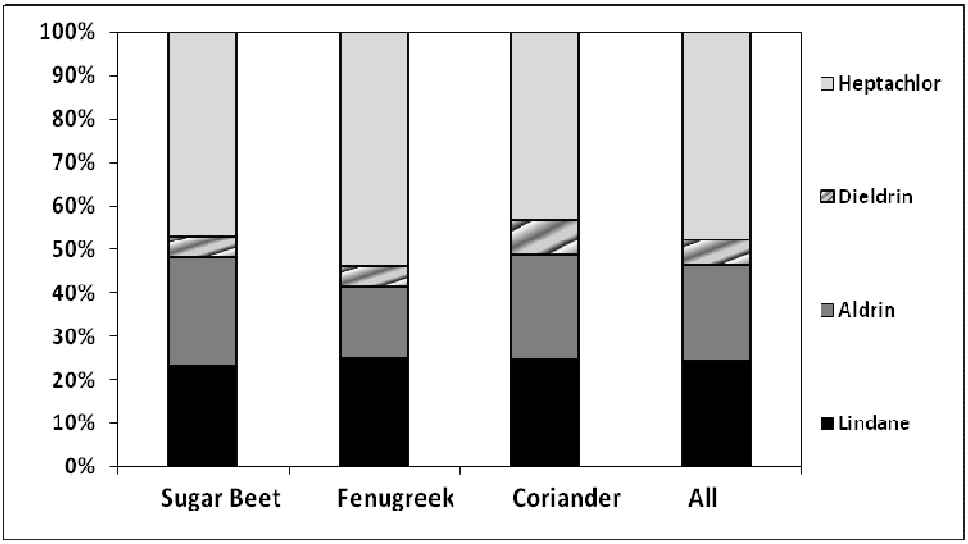


**Figure 1: Distribution in percent of aldrin, dieldrin, heptachlor and lindane**

**Table 2: Concentrations (range and mean) of aldrin, dieldrin, heptachlor and lindane in Sugar beet, Fenugreek and Coriander leaves**

| Pesticides | Name of vegetables | | | | | |
|---|---|---|---|---|---|---|
| | Sugar beet | | Fenugreek | | Coriander | |
| | range | mean±se* | range | mean±se | range | mean±se |
| Aldrin | <0.01-1.58 | 0.49±0.19 | <0.01-0.76 | 0.34±0.11 | <0.01-1.31 | 0.59±0.19 |
| Dieldrin | <0.01-0.19 | 0.10±0.02 | <0.01-0.29 | 0.10±0.04 | <0.01-0.80 | 0.19±0.10 |
| Heptachlor | <0.01-2.80 | 0.94±0.28 | <0.01-2.80 | 1.03±0.39 | <0.01-2.30 | 1.06±0.27 |
| Lindane | <0.01-1.23 | 0.47±0.14 | <0.01-1.20 | 0.52±0.19 | <0.01-1.59 | 0.60±0.20 |
| ∑OCPs | <0.01-4.38 | 2.00±0.51 | <0.01-4.16 | 2.08±0.64 | <0.01-6.00 | 2.44±0.69 |

**se (standard error) =SD/√n*

Concentration of Aldrin, dieldrin and heptachlor in vegetables from this study were lower than reported in vegetables from Meerut, Muzaffarnagar and Ghaziabad district of Uttar Pradesh, India [9], Central cities of Uttar Pradesh, India [10], Jaipur, India [11] and Pakistan [12]. The variations in levels of pesticide residues in vegetables grown on Indian soil are due to the disproportionate usage of pesticides in India and the amount of pesticide residue varies from one place to another. The states like Uttar Pradesh, Tamilnadu, Andhra Pradesh, Haryana and Karnataka have highest use of pesticides while the states like Bihar, West Bengal, North eastern states have lowest use of pesticides.

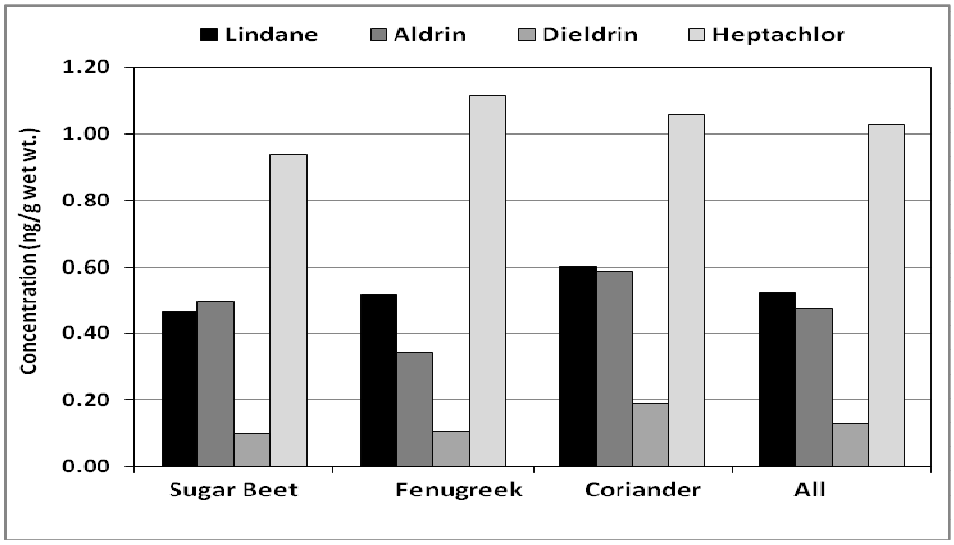


**Figure 2: Comparative distribution of aldrin, dieldrin, heptachlor and lindane**

**Table 3: MRLs for pesticide in vegetables: Comparison with of this study**

| ng/g | Organochlorine Pesticide Compounds | | | | Reference |
|---|---|---|---|---|---|
| | Aldrin | Dieldrin | Heptachlor | Lindane | |
| MRLs (maximum residual limits) | | | | | |
| Europe | 10 | 10 | 10 | 50 | [7] |
| India | 100 | 100 | 50 | 1000 | [8] |
| Present study | 0.48 | 0.13 | 1.03 | 0.52 | - |

The observed concentration of OCP compounds in this study was compared with recommended maximum residue limits (MRLs) set by European commission [7] and Food Safety and Standard Authority of India [8], European commission and Indian government recommended the MRLs of 10 and 100 ng/g, 10 and 100 ng/g, 10 and 50 ng/g, and 50 and 1000 ng/g for aldrin, dieldrin, heptachlor and lindane was, respectively (Table 3). It is clear from the comparison of obtained results with MRLs, that studied vegetables in this study had residue levels of OCPs far much below the recommended MRL for human consumption which indicates minimal risk.

## CONCLUSION

The analysis of market vegetables from Kolkata, West Bengal (India) has demonstrated a quite low level of contamination by organochlorine pesticides, which generally never exceeded the residue levels of OCPs set by European Commission and Indian government, indicating minimal risk to the consumers. However, a frequency of presence of the OCPs in vegetables was observed which may be a matter of concern since organochlorine pesticides are known to accumulate in biota. Therefore, identification and elimination of contamination sources of OCPs in vegetables is recommended for the protection of human health.

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