*Available online www.jocpr.com*

**Journal of Chemical and Pharmaceutical Research, 2014, 6(6):751-755**

**Research Article**

**ISSN : 0975-7384**
**CODEN(USA) : JCPRC5**

# Monitoring of organochlorine pesticide residues in mango *(Mangifera indica)*, papaya *(Carica papaya)* and bottle gourd *(Lagenaria siceraria)* by gas chromatography

**Devendra Kumar* and Anjali Kashyap**

*Department of Chemistry, Institute of Basic Sciences, Dr. B. R. Ambedkar University, Khandari Campus, Agra*

---

**ABSTRACT**

*Residual levels of organochlorine pesticides (OCPs) were determined in two fruits (mango and papaya) and one vegetable (bottle gourd) purchased from local market. Ninteen organochlorine pesticides including isomers of benzene hexachloride or BHC (α-BHC, β-BHC, γ-BHC and δ-BHC), heptachlor, heptachlor epoxide, aldrin, γ-chlordane, endosulfan+α-chlordane, dieldrin+p,p'-DDE, endrin, endrin aldehyde, endrin ketone, endosulfan II, endosulfan sulphate, p,p'-DDT, p,p'-DDD, methoxychlor and decachlorobiphenyl (DCBP) were monitored by using Gas Chromatography coupled with Electron Capture Detector (ECD). Mango and papaya fruits were found contaminated with heptachlor, endrin aldehyde, endosulfan sulphate, endrin ketone and methoxychlor while β-BHC, δ-BHC, heptachlor, dieldrin+p,p'-DDE, p,p'-DDT and endrin ketone were found in bottle gourd. The concentration of the detected pesticides were below the maximum residue limit (MRL) values but continuous consumption of such fruits and vegetable even with moderate contamination level can accumulate in the body and may prove fatal for human in the long term.*

**Key words:** Organochlorine pesticides, Mango, Papaya, Bottle gourd, GC-ECD

---

## INTRODUCTION

In India, farmer uses about 41822 metric tonnes pesticides [Directorate of Plant Protection and Quarantine **2011**] to control pests and to increase the yield and the quality of the crops. The use of toxic chemicals 'pesticide' has resulted to cause much dangerous diseases to public health. It is the main international serious trade problem [1]. Their residues have been reported in various environmental matrices [2-4]. In the modern world, environmental pollution is one of the serious predicaments [5]. During the last decade, increasing environmental pollutants and lack of safety measures of the environmental regulations, it has become a global problem [5-8]. Their exposure has been known to be associated with long term human health problems like respiratory, memory disorders, dermatological conditions [9,10] cancer [11] depression, neurological deficiencies [12,13] miscarriages and birth defects [14]. To protect consumer's health, many countries have established legal directives to control their levels in food, through Maximum Residue Levels, MRLs [FAO/WHO, Food standards programme. Codex Alimentarius Commision. **2004**; Council Directive. **2003**.]. Thus, in continuation of our previous work [15,16] an effort has been made to estimate the residual concentration levels of selected organochlorine pesticides in mango, papaya and bottle gourd.

## EXPERIMENTAL SECTION

All glassware were thoroughly washed by deionized water and then rinsed with acetone and dried in oven (150$^{o}$C) for overnight before use. Solvents like acetone, acetonitrile, dichloromethane, n-hexane and ethyl acetate were distilled before use. Adsorbent neutral alumina, florisil and charcoal were activated before use. Anhydrous sodium sulphate was purified with acetone and heated for 4h at 600$^{o}$C in muffle furnace to remove possible phthalate

impurities. The extracted and purified samples of fruits and vegetables were analyzed by GLC equipped with capillary columns using $^{63}$Ni electron capture detector (ECD) [17]. Minor equipments such as mechanical shaker, rotatory evaporator and warring blender etc. were also used during extraction. Stock solution of standard was prepared in hexane in 5 ppm concentration.

**(I) Estimation of Pesticides in Fruits**

**1.1. Sample Collection and Preparation:** The sample consists of 1kg of each fruit i.e. mango and papaya was collected from local market. Each sample was refrigerated at 5ºC and analyzed within two days of collection. In order to measure the right concentration of pesticides reaching within human body, the household processing like-washing and peeling off covering etc. were carried out. Each fruit was washed for few minutes under tap water and dried with the help of filter paper. After drying each fruit was peeled carefully, cut into small pieces and blended with the high speed warring blender to make a fine paste.

**1.2. Extraction and Clean up:** 50 g of fine paste was taken out from the fruits sample and subjected to extraction with 100 ml acetone [18] for three times. The extract was filter with the help of Buchner funnel. The filtrate was concentrated in vacuum up to 5 ml and then transferred to a 500 ml separating funnel. Saline water (2%, w/v, 50 ml) was added to it and shake for 50 min. The extract was exchanged into dichloromethane layer by liquid-liquid partitioning (3×50 ml). The organic layer was separated out from the separating funnel and passed through a layer of sodium sulfate (5 gm). The residue was again evaporated to dryness (2-5 ml) in rotatory evaporator. Clean up was performed by the column chromatography. Glass column (30 × 1.5 × 1.5 cm) was packed with anhydrous sodium sulfate (2 g) + neutral alumina (5 g) + florisil (1 g). The column was prewashed with n-hexane. The concentrated extract was dissolved in 10 ml hexane–acetone (9:1) and subjected to column cleanup. The column was eluted with 25 ml dichloromethane-acetone (8:2). The extract was again evaporated to dryness (2-5 ml) in rotatory evaporator and finally dissolved in 10 ml hexane-acetone (9:1) for the chromatographic analysis.

**(II) Estimation of pesticides in vegetable**

**1.3. Sample collection and preparation:** 1 kg bottle gourd was collected from local market, refrigerated at 5ºC and analyzed within two days of collection. Sample was washed for few minutes under tap water and dried with filter paper. After drying bottle gourds were chopped into small pieces. A subsample of 50 g was separate out and homogenized with 100 ml acetonitrile [19] in a high speed blender.

**1.4. Extraction and Clean up:** The mixture was then transferred into a separating funnel and shake gently for 1 h with 100 ml mixture of n-hexane and dichloromethane (3:2 v/v). The separating funnel was allowed to be held at vertical position for about 30 min to have distinct layer. n-hexane- dichloromethane layer was separated and collected. This process was repeated three times. The collected extract was dried up to dryness (2-5 ml) in a rotatory evaporator. The dry residue was dissolved in 10 ml n-hexane and subjected for clean up by column packed with florisil and activated charcoal (5:1 w/w). The extract was eluted with 50 ml mixture of ethyl acetate:n-hexane (3:7 v/v). Eluted extracted was dried up to dryness and redissolved in 5ml n-hexane and then injected to GC-ECD for analysis.

## RESULTS AND DISCUSSION

First by running chromatogram of standards (Fig.1), we have determined retention time of the peaks for standards and their peak areas corresponding to 5 ppm concentration. In the chromatogram of standards, the peaks of different isomers of benzene hexachloride (BHC) were found at Rt values 5.210, 5.834, 6.003 and 6.676 which correspond to α-BHC, β-BHC, γ-BHC and δ-BHC respectively. The peaks of heptachlor and heptachlor epoxide were found at Rt value 8.285 and 11.637 respectively. The peak at Rt value 9.731 was found for aldrin. The peak of γ-chlordane was found at Rt value 12.934. The peak at Rt value 13.825 was found for endosulfan I +α-chlordane. Rt value of the peak of dieldrin+ p, p'-DDE was found at 15.241. The peaks at Rt values 16.704, 18.787 and 24.278 correspond to endrin, endrin aldehyde and endrin ketone respectively. The peaks of endosulfan II and endosulfan sulphate were found at Rt values 17.385 and 20.584. The peaks of p,p'-DDD and p,p'-DDT were found at Rt values 18.173 and 21.026. The peak at Rt value 26.181 was found for methoxychlor. Rt value of the peak of decachlorobiphenyl (DCBP) was found at 42.525.

The chromatogram of mango ( Fig.2) exhibited five peaks at Rt values 8.359, 18.849, 20.349, 24.219 and 26.127 which were very near to the Rt values of heptachlor, endrin aldehyde, endosulfan sulphate, endrin ketone and methoxychlor respectively. In the chromatogram of papaya (Fig.3) five peaks at Rt values 8.429, 18.828, 20.303, 24.217 and 26.695 have been noticed which were very near to the Rt values of heptachlor, endrin aldehyde, endosulfan sulphate, endrin ketone and methoxychlor respectively. The chromatogram of bottle gourd (FIG.4) exhibited six peaks at Rt values 5.828, 6.224, 8.645, 15.770, 21.008 and 24.584 which were very close to the Rt

values of β-BHC, δ-BHC, heptachlor, dieldrin+p,p'-DDE, p,p'-DDT and endrin ketone respectively. Detected pesticides and their concentrations have been reported in Table.1.

**Table (1): Concentration of pesticides evaluated through Gas-Liquid Chromatography.**

| S. No. | Name of the Sample | Name of Pesticide Found | Concentration of Pesticides (ppm) |
|---|---|---|---|
| **1** | **Mango** | Heptachlor | 0.164 |
| | | Endrin aldehyde | 0.096 |
| | | Endosulfan sulphate | 0.098 |
| | | Endrin ketone | 0.119 |
| | | Methoxychlor | 0.551 |
| | | | |
| **2** | **Papaya** | Heptachlor | 0.213 |
| | | Endrin aldehyde | 0.043 |
| | | Endosulfan sulphate | 0.043 |
| | | Endrin ketone | 0.053 |
| | | Methoxychlor | 1.656 |
| | | | |
| **3** | **Bottle gourd** | β-BHC | 3.747 |
| | | δ-BHC | 1.036 |
| | | Heptachlor | 0.134 |
| | | Dieldrin+p,p'-DDE | 0.141 |
| | | p,p'-DDT | 0.587 |
| | | Endrin ketone | 0.691 |

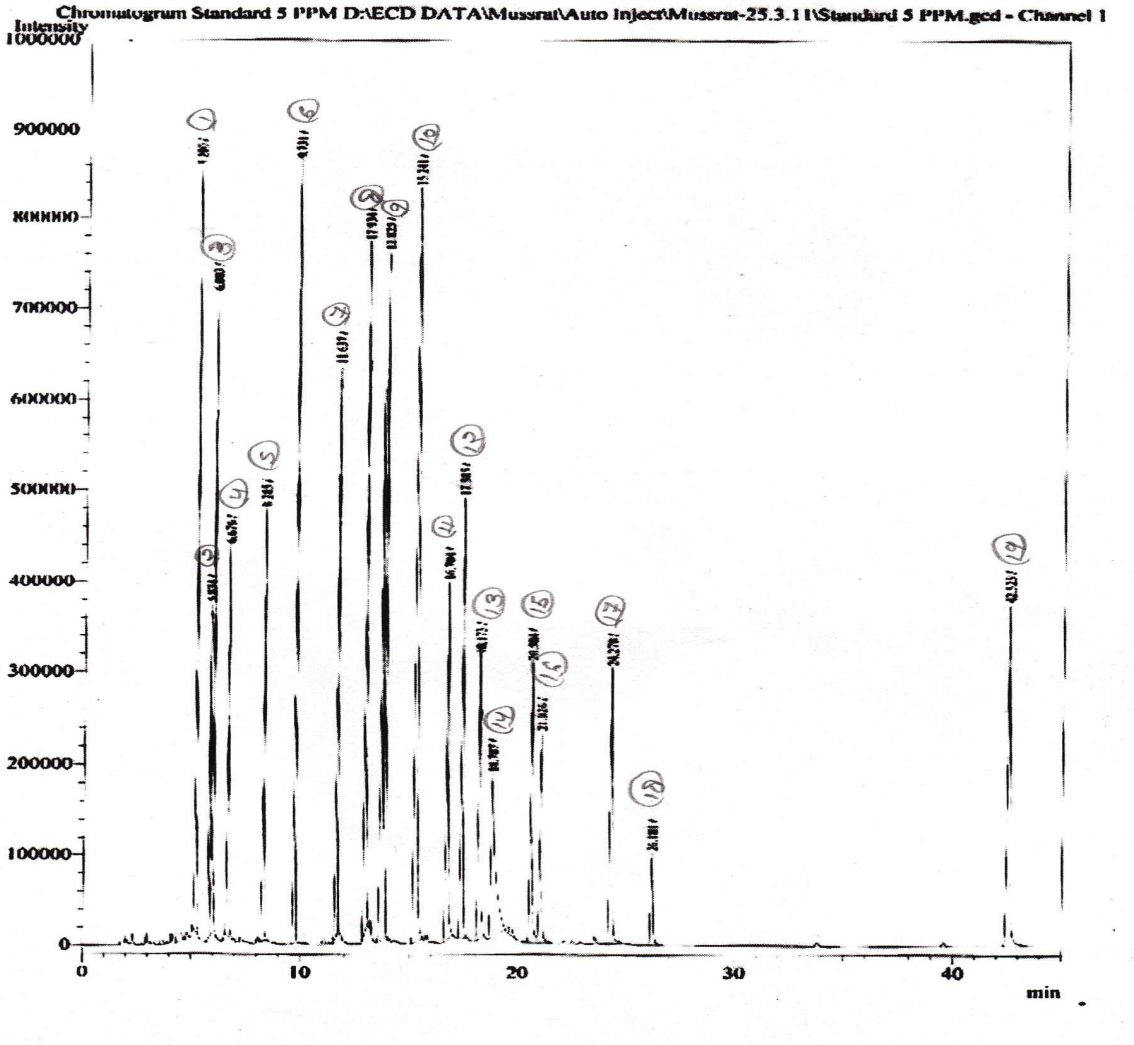


**Fig. 1: Gas chromatogram of standard**

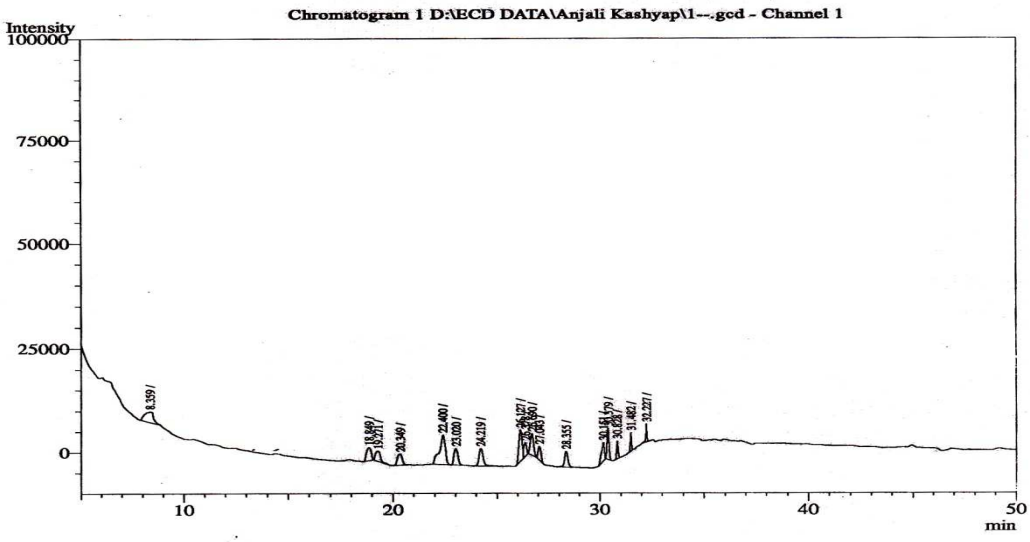


**Fig. 2: Gas chromatogram of mango**

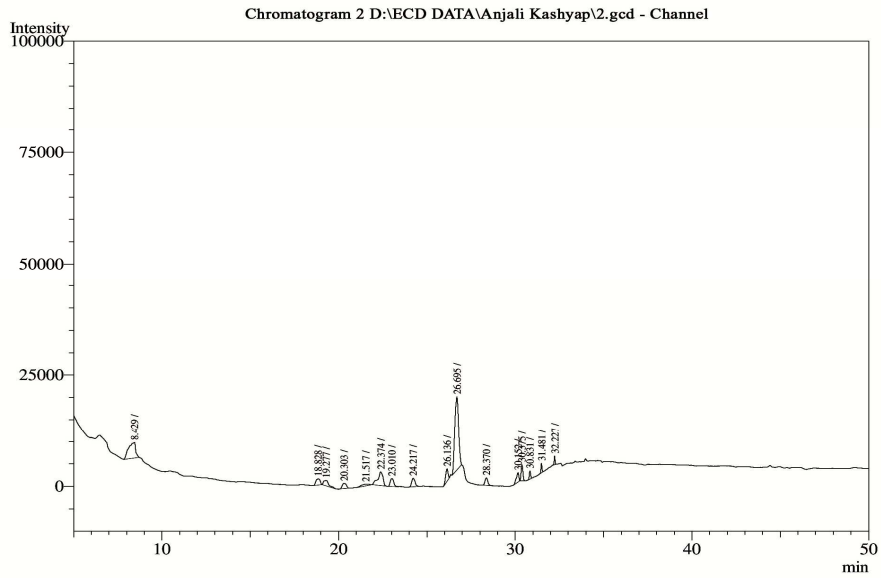


**Fig. 3 Gas chromatogram of papaya**

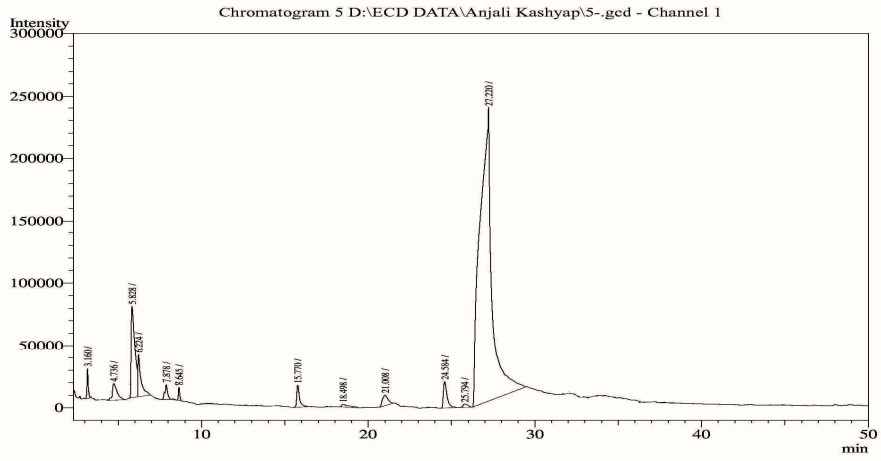


**Fig. 4 Gas chromatogram of bottle gourd**

## CONCLUSION

In order to minimize health risk as well as for enforcement activities, monitoring of pesticide residues is increasingly important and essential. A simple, effective and quick method using GC-ECD has been developed for monitoring the different pesticides. The obtained results indicated that both fruits- Mango and Papaya contain methoxychlor in maximum concentration and vegetable- Bottle gourd is contaminated with β-BHC in maximum concentration.

**Acknowledgments**
We thank to Advanced Instrumentation Research Facility (AIRF), New Delhi for providing GC analysis graph and data of samples.

## REFERENCES

[1] K Beena; TS Kathpal, *J. Environ. Monit. Assess.*, **2009**, 151(1-4), 19-26.
[2] B Kumari; R Singh; VK Madan; R Kumar; TS Kathpal, *Bull. Environ. Contam. Toxicol.*, **1996**, 57(5), 787-793.
[3] B Kumari; VK Madan; R Kumar; TS Kathpal, *Environ. Monit. Assess.*, **2002**, 74(3), 263-270.
[4] B Kumari; R Singh; VK Madan; R Kumar; TS Kathpal; J Singh, *Environ. Monit. Assess.*, **2003**, 87(3), 311-318.
[5] DG Hela; DA Lambropoulou; IK Konstantinou; TA Albains, *Environ. Toxicol. Chem.*, **2005**, 24(6), 1548-1556.
[6] S Bondarenko; J Gan; DL Haver; JN Kabashima, *Environ. Toxicol. Chem.*, **2004**, 23(11), 2649-2654.
[7] KY Abdel-Halim; AK Salama; EN El-Khateeb; NM Bakry, *Chemosphere*, **2006**, 63(9), 1491-1498.
[8] PC Wilson; JF Foos, *Environ. Toxicol. Chem.*, **2006**, 25(11), 2847-2852.
[9] TA Arcury; SA Quandt; BG Mellen, *J. Agric. Safety and Health*, **2003**, 9(3), 221-232.
[10] MA O'Malley, *Occup. Med*., **1997**, 12, 327-345.
[11] JL Daniels; AF Olshan; DA Savitz, *Environ. Health Perspect*., **1997**, 105(10), 1068-1077.
[12] F Kamel; AS Rowland; LP Park; WK Anger; DD Baird; BC Gladen; T Moreno; L Stallone; DP Sandler, *Environ. Health Perspect*., **2003**, 111(14), 1765-1772.
[13] JA Firestone; T Smith-Weller; G Franklin; P Swanson; WT Longstreth; H Checkoway, *Arch. Neurol*., **2005**, 62, 91-95.
[14] LS Engel; ES O'Meara; SM Schwartz, *Scand. J. Work, Environ. Health*, **2000**, 26(3), 193-198.
[15] D Kumar; P Chauhan; Neelam, *Res. J. Agric. Sci*., **2011**, 2(3), 528-531.
[16] D Kumar; RC Sharma; P Chauhan, *Res. J. Agric. Sci.*, **2011**, 2(1), 40-43.
[17] J Harald; W Franz; T Horst, *Anal. Bioanal. Chem.*, **2008**, 391(1), 97-116.
[18] I Mukherjee; S Singh; PK Sharma; M Jaya; M Gopal; G Kulshrsestha, *Bull. Environ. Contam. Toxicol*., **2007**, 78, 380-383.
[19] M Bhanti; A Taneja, *Chemosphere*, **2007**, 69(1), 63-68.