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Research Article

# Estimation of pesticide residues in fruits, vegetables and water

Pragti*, H.N. Sharma, Yogesh Singh, L.P. Srivastava[1], Nisha Srivastava, Sonam, Pawan Kumar

*Department of Environmental Toxicology, School of Life Science, Dr. B.R. Ambedkar University, Agra-282002, U.P., India.*
*[1]Pesticide Toxicology Laboratory, CSIR-Indian Institute of Toxicology Research, Lucknow- 226001, U.P., India.*

**Abstract**: Nowadays, pesticide use is on increase day by day in every field of life like agriculture, household and in industry. Most of the pesticides are not biodegradable and those which are degradable cannot degrade completely. Hence, they reach to our fruits, vegetables, grains and water bodies. Even after washing the pesticide residues left in our foodstuffs. Since last decade, it has been observed that analysed pesticide residues were either BDL or < MRL in the majority of samples. In this study vegetable, fruits and grains are an important food item and proper care should be taken to use the very safe pesticide fort avoiding potential risks to human. It is, therefore, suggested that the food item collected from in and around Lucknow City, India is comparatively safe from pesticide residues. A periodical monitoring of pesticide residues in other food commodities is the recent need for the consumers as well as authorities of food quality control.

**Keywords:** Pesticides, QuEChERS method, Liquid liquid extraction, Gas chromatography.

## 1. Introduction

Pesticides are substances or mixture of substances intended for preventing, destroying, repelling or mitigating any pest. Pesticides are a special kind of products for crop protection. Crop protection products, in general, protect plants from damaging influences such as weeds, diseases or insects. A pesticide is generally a chemical or biological agent (such as a virus, bacterium, antimicrobial or disinfectant) that through its effect deters, incapacitates, kills or otherwise discourages pests. Target pests can include insects, plant pathogens, weeds, mollusks, birds, mammals, fish, nematodes (roundworms), and microbes that destroy property, cause nuisance, spread disease or are vectors for disease. Although there are human benefits to the use of pesticides, some also have drawbacks, such as potential toxicity to humans and other animals.

Pesticides are not only applied in agriculture field to protect crops from pests but also one public property, indoor control of insect, within swimming pools and for other essential purpose. Pesticide applicators should understand the hazards and risks associated with the pesticides they use pesticides a very greatly in toxicity. Toxicity depends on the chemical and physical properties of a substance and may be defined as the quality of being poisonous or harmful to animals or plants. Poisons have many different modes of action, but in general cause biochemical changes which interfere with normal body functions.

Pesticides are categorized into four main substituent chemicals: herbicides, fungicides, insecticides and bactericides any substance or mixture of substances intended for preventing, destroying or controlling any pest, including vectors of human or animal disease, unwanted species of plants or animals causing harm during or otherwise interfering with the production, processing, storage, transport or marketing of food, agricultural commodities, wood and wood products or animal feedstuffs, or substances which may be administered to animals for the control of insects, arachnids or other pests in or on their bodies. The term includes substances intended for use as a plant growth regulator, defoliant, desiccant or agent for thinning fruit or preventing the premature fall of fruit. Also used as substances applied to crops either before or after

*Corresponding author:
E-mail: harendra_n_sharma@yahoo.com.

harvest to protect the commodity from deterioration during storage and transport.

The toxicity of any compound is related to the dose. A highly toxic substance causes severe symptoms of poisoning with small doses. A substance with a low toxicity generally requires large doses to produce mild symptoms. Toxicity can be either acute or chronic. Acute toxicity is the ability of a substance to cause harmful effects which develop rapidly following absorption, i.e. a few hours or a day. Chronic toxicity is the ability of a substance to cause adverse health effects resulting from long-term exposure to a substance.

## 2. Materials and Methods

### 2.1 Analyzed Pesticides

Total sixteen pesticide, including α, β, γ, δ HCH, p,p'-DDD, p,p'-DDE, p,p'-DDT, o,p´-DDD, α-Endosulfan, β-Endosulfan, Cypermethrin-I, Cypermethrin-II, Fenvalerate-I, Fenvalerate-II, Malathion and Chlorpyrifos were taken for the analysis of pesticide residues in food commodities.

### 2.2 Glassware's

Conical flask (250ml), separatory funnel (1000ml), measuring cylinder (250ml and 500ml), funnel (100ml). Whatman filter paper No - 41, separatory funnels stand, tissue paper, filter paper, cotton, Aluminum foil, centrifuge tube (50ml), Micro-centrifuge tube (Eppendorf), parafilm, marker.

### 2.3 Instrumentation

Following Instruments used in the analysis:

**a. Gas Chromatograph (GC)**

A gas chromatograph (GC) is an analytical instrument that measures the content of various components in a sample (Fig. 1). The analysis performed by a gas chromatograph is called gas chromatography. The sample solution injected into the instrument enters a gas stream which transports the sample into a separation tube known as the "column." (Helium or nitrogen is used as the so-called carrier gas.) The various components are separated in the column. The detector measures the quantity of the components that exit the column. To measure a sample with an unknown concentration, a standard sample with known concentration is injected into the instrument. The standard sample peak retention time (appearance time) and area compare to the test sample to calculate the concentration.

**b. Electron Capture Detector (ECD)**

A number of detectors are used in gas chromatography. In this study, electron capture detector (ECD) was used. The ECD or electron capture detector measures electron-capturing compounds (usually halogenated) by creating an electrical field in which molecules exiting a GC column can be detected by the drop in current in the field. It finds its greatest application in analysis of halogenated compounds.

**c. R-23 Centrifuge**
**d. DeltekTC Microcentrifuge**
**e. Rotospin**
**f. Weighing balance**
**g. Rotavopour**
**h. pH-meter**

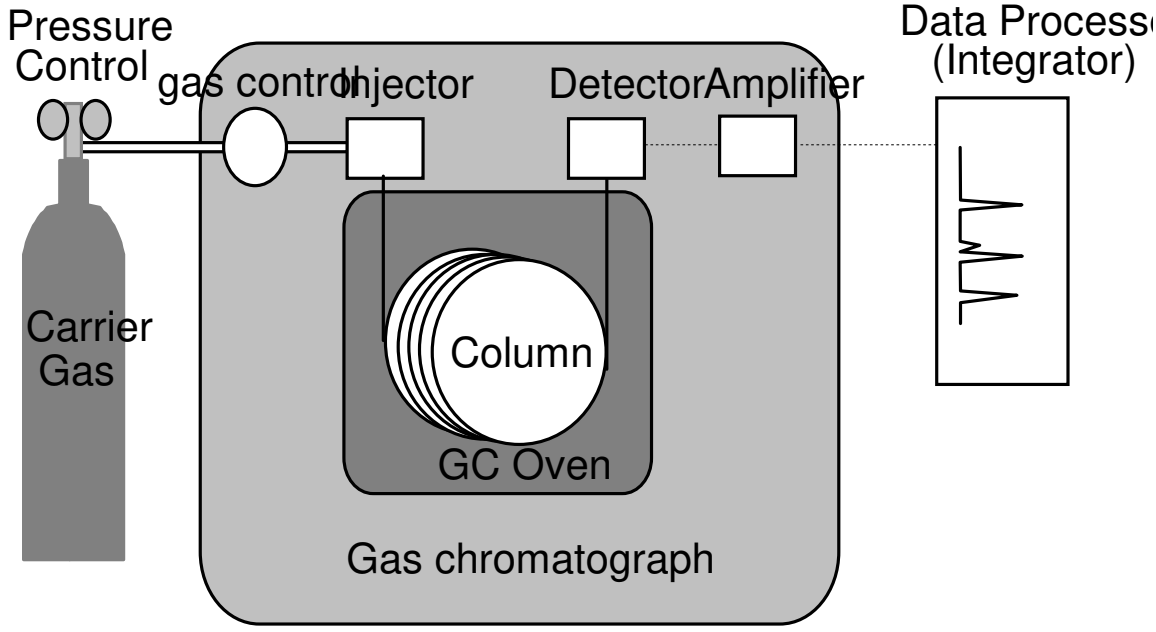


**Fig. 1. Basic Components in a Gas Chromatograph.**

### 2.4 Principle of Gas-Chromatography (GC)

The basic principle of GLC has derived from the partitioning a gas (mobile phase) and liquid or solid (stationary phase). The liquid coats of finely divided material are placed in a column. When a gas comes in contact with the liquid and phenomenon takes place according to Henry's laws of partition, i.e.-

$$x/m = Kc$$

Where x = mass of gas dissolved in liquid;
m = mass of liquid on which amount of gas dissolved;
c = gas of concentration dissolved in the gas phase;
K = constant.

The phenomenon is completely selective and there are different values for k for different pesticides. The column, oven (injection) and detector are three important components of gas chromatography.

### 2.5 Type of chromatography

It is divided into three methods–

- Paper chromatography
- Thin layer chromatography
- Gas-liquid chromatography

The main objective of residue analysis is to determine by the shortest possible time as much pesticide residue as possible, which occur in the sample of unknown origin. It is an advantageous method, which enables the determination of pesticide residues of different chemical classes is the same extract. The main advantages of chromatographic method are:

- Rapid screening for pesticide analysis.
- Then identification (confirmation and qualification).

### 2.6 Sample collection

A total of 24 samples of different food commodities including fruits (apple, banana), vegetables (okra, cauliflower, and tomato), and grains (wheat, rice, and pulses), were collected from Lucknow city, India.

### 2.7 Chemicals

All solvents like n-hexane, acetone, Dichloromethane (DCM) and ethyl acetate (HPLC grade) were used. All Glassware is distilled before use. Acetone was refluxed over potassium permanganate for 4 hours and then distilled. Salt like Sodium chloride (NaCl), anhydrous Sodium sulfate ($Na_2SO_4$) and anhydrous Magnesium sulfate ($MgSO_4$) used and before using they were purified with acetone and baked for 4 hours at 600˚C in a muffle furnace to remove possible phthalate impurities. Primary, secondary amine (PSA) biodiesel 40µm parts 12213024 were used for sample preparation. Pesticide Standards were procured from Supelco, Sigma-Aldrich, USA, Fluke, Sigma-Aldrich Schweiz, and Rankem Pvt. Ltd., New Delhi, India.

### 2.8 Used methods

**a.** QuEChERS method
**b.** Liquid liquid extraction (LLE)

### 2.9 Extraction and cleanup for vegetables and fruits

The collected fresh vegetable/fruit sample (100g) was washed, cleaned, chopped and grind in warring blender. 10g macerated sample of each vegetable/fruits was taken for multi-pesticide residue analysis by QuEChERS method. 10g of macerated sample was mixed with 10ml ethyl acetate, 4gm of anhydrous $MgSO_4$, 1.0gm activated NaCl and shaken for 10 minutes at 5rpm on the Rotospin test tube rotator. The extract was centrifuged for 10 minutes at 8,000 rpm. 1ml aliquots of vegetable extract were cleaned with the mixture of 100mg PSA, 150mg anhydrous $MgSO_4$ and 10mg activated charcoal. The extract was again shaken for 10 min. at 50 rpm on Rotospin and centrifuged for 10 min. at 8,000 rpm. The supernatant was collected in 2ml vial and mixed with 5µm acidified ethyl acetate (ethyl acetate acidified with 5% formic acid). 1µl of clean sample inject in GC-(ECD/NPD) and registered all chromatogram.

## 3. Result and Discussion

The presence of pesticide residue in vegetable has become a global phenomenon. A level of 16 pesticides, including (Organochlorine, Organophosphorus, Herbicides and Synthetic Pyrethroids) analyzed in different food commodities lick (vegetables, fruits and grain). 70 to 96% recoveries were found in different food commodities. The analyzed pesticides were α-HCH, β-HCH, γ-HCH, δ-HCH, pp-DDD, pp-DDE, pp-DDT, op-DDD, α-endosulfan, β-endosulfan, cypermethrin-I, cypermethrin-II, Fenvalerate-I, Fenvalerate-II, Malathion and chlorpyrifos. Out of total 24 analyzed samples only apples, okra, wheat and pulses were found HCH, Endosulfan, cypermethrin and malathion sowing in Table 1. 24 water samples were analyzed and pesticide residues were not detected. Limit of detection (LOD) 0.005 to 0.010mg/$kg^{-1}$ of following pesticides varied from 0.001-0.009mg $kg^{-1}$. Similarly, the present recovery of OCs, OPs, SPs and H varied from 72 to 100% from the fortification level of 0.01mg $kg^{-1}$. The pesticide residue recorded below the detection limit was considered as nondetectable (ND). The level of pesticide residues in various food commodities was compared with their MRL fixed by prevention of food adulteration act (PFA), Government of India, 1954.

The low level of OC, OP and SP residues in fruits, vegetables and grains of the present study are an indicative change in uses pattern of pesticides, where

shift has taken place from persistent OC's to the easily degradable groups like OP's and SP's. All the water samples were safe in terms of pesticide residues. Since last decade, it has been observed that analyzed pesticide residues were either BDL or < MRL in the majority of samples. In this study vegetable, fruits, and grains are an important food item and proper care should be taken to use very safe pesticide for avoiding potential risks to human. It is, therefore, suggested that the food item collected from in and around of Lucknow City, India is comparatively safe from pesticide residues. A periodical monitoring of pesticide residues in other food commodities is the recent need for the consumers as well as authorities of food quality control.

**Table 1. Pesticide residue level in different food commodities.**

| Food commodities | Pesticides detected | Range concentration in (mg $kg^{-1}$) | PFA below MRL | PFA above MRL |
|---|---|---|---|---|
| **Apple** | Malathion | 0.009-0.010 | YES | NO |
| **Banana** | ND | ND | - | - |
| **Okra** | β-Endosulfan | 0.101-0.113 | YES | NO |
| **Cauliflower** | ND | ND | - | - |
| **Tomato** | ND | ND | - | - |
| **Wheat** | Cypermethrin-I | 0.132-0.151 | NO | YES |
| **Rice** | ND | - ND | - | - |
| **Pulses** | γ- HCH | 0.008-0.010 | YES | NO |

*ND = Not Detected; MRL = Maximum Residual Limit; PFA = Prevention of Food Adulteration Act. (PFA), Govt. of India (1954).

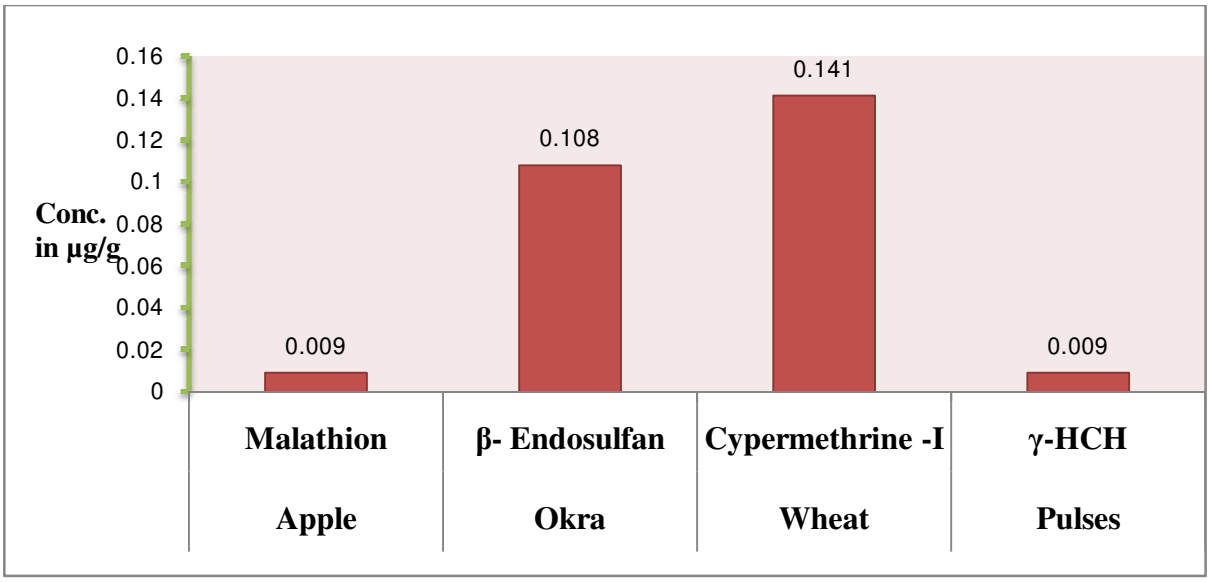


**Fig. 2. Pesticide residue level in different food commodities.**

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