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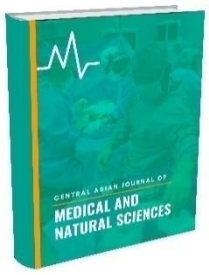

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"Soil Purification" Analysis of Pesticide Residues in the Soil

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Abstract: Pesticides are chemicals that prevent insects, weeds, and fungi from damaging crops and are used by farmers to increase the number of crops they can produce. Governments test and regulate pesticides and product testing ensures that levels are low enough to not pose a threat to human health. However, many people worry about pesticides in their foods, so they resort to expensive organic foods.

In this research, we will learn about the types of pesticides. And whether its residues affect human health. And how can we clean the soil from it.

Key words: Soil Purification.

Introduction

Pesticides: Chemicals used to kill or control pests.

Although some pesticides, especially organic chlorine pesticides that are more stable in the environment, have been banned or restricted in use, some countries still use them to control pests in agricultural production and diseases transmitted by insect vectors.

According to the World Food and Agriculture Organization and the World Health Organization, it has been recognized that the causes of contamination of various food commodities, drinking water, and land water are caused by various types of pesticides all over the world.

Soil: with a complex composition of various elements:

1. Abiotic: such as solids, liquids, and gases.
2. Biotic: such as invertebrate soil animals, algae, and microorganisms with a building (bacteria - fungi) that support the life system of roots, plant growth, flora, and other fauna.

The dynamic and symbiotic nature between biotic and abiotic components is responsible for soil fertility. Soil quality is used in soil management and can affect human and animal health.

The presence of bioavailability of pesticides in the soil negatively affects human and animal health, plants, and beneficial soil organisms. The soil in the ecosystem acts to some extent as a storehouse for pesticides.

Pesticide particles stick to the organic matter in the soil through a process called adsorption (absorption), and some pesticide particles dissolve between soil particles and water called (soil water).

When an excessive amount of water enters the soil through rainwater and irrigation water, the specialized pesticide particles become separated from the soil particles through a process called (consolidation or adsorption).

The solubility of pesticides and their absorption on the soil is inversely related to the increase in dissolution and lack of absorption. Pesticide residues are toxic as they destroy vital components, organic matter, and thus soil fertility.

The importance of the soil lies in the fact that it is a storehouse for several pesticides, and to ensure safe and healthy food for humanity and good soil quality.

Several methodologies and methods have been described for the analysis of residues of a pesticide or several pesticides in the soil, such as/ Gas or liquid chromatographic analysis is quite similar to the analysis of samples of food, vegetables, and beverages. These techniques have been mentioned in other chapters of this book.

In this chapter, all the steps for sampling and preparing samples for analyzing pesticide residues in the soil and purifying the soil from them have been explained.

* General aspects of pesticide residues in the soil:

Pesticides are applied to aerial vegetation (above the soil surface) and roots in the soil (below the soil surface) to protect subsoil plant production such as potato tubers from common insects such as termites.

By applying pesticides on agricultural lands, a group of attics occurs, including:

1. It is either absorbed by plants.

Or digested by animals, insects, worms, or micro-organisms in the soil and metabolized at a later time.

2. They move to land in the soil or suck on soil particles.
3. It evaporates and enters the biosphere through a series of chemical and microbial pathways and reactions.
4. It is filtered out from the root zone or washed from the soil surface due to irrigation water and rain.

Evaporation of water from the surface of the soil leads to the rise of pesticide well.

- The dissolution of pesticides by chemical methods is done partially, several pesticides are broken down or broken down by soil organisms into (carbon dioxide, water, and other organics as well as sunlight helps by breaking down or dividing pesticides to varying degrees).
- Soils differ in quality in terms of the characteristics of the organic matter, where microbiological activity plays a role in the degradation of pesticides.
- Decreasing the process of degradation of pesticides, as more pesticides seep beyond the root zone, where microbial activity decreases and microbial communities become less.
- However, some pesticides continue to decompose after a series of chemical reactions and leave the root zone.
- Anchormen of pesticides: a term that denotes the ability of the soil to fix pesticides in place and not allow them to be transported.
- Adsorption: It is the basic process that preserves pesticides in the soil and accumulates them on the surface of soil particles. The adsorption of pesticides in the soil depends on:

1. Chemical properties of pesticides: (solubility in water, polarity...).

2. Soil properties: (organic matter - clay content - acidity - surface properties change, permeability).

The most important organic matter in controlling the movement of pesticide residues in the soil.

Laboratory experiments were conducted on soil, worms, and atrazine, and biodegradation of atrazine was observed by worms and bacteria.

After adding the pesticide, the bacteria in the soil begin their task, and by measuring the carbon dioxide (CO2) released, the decomposition of atrazine is known, and the effect of worms appears when the release of carbon dioxide increases

*** Analysis of pesticide residues in the soil:**

The procedures for analyzing pesticide residues in the soil are subject to great development in the current situation as a result of the increase in the number of pesticides and the multiplicity of their compounds, as the procedures for sampling and taking special, extreme, or extreme care in them are among the most important things when analyzing pesticide residues in the soil.

Basic rules must be followed when taking samples to represent reality and in anticipation of an error in entering these samples for analysis.

The complexities of analyzing pesticide residues in the soil are due to the following reasons:

1. The change and increase in the content of the components included in the organic matter, such as folic acid and humic acid.
2. Adsorption of various pesticide residues on soil particles strongly leads to a difference in the percentage of these residues and their estimates on the ground.
3. Most of the common methods for separating pesticide residues from the soil are done by interaction with organic solvents, followed by cleaning procedures to remove interferences before analysis (to isolate pesticides from the soil, they are usually mixed with organic solvents), where the soil is filtered and filtered from organic solvents to remove particles of materials using anhydrous sodium sulfate as a dry substance.

- When examining dried soil samples, the Sox let separation technique is used by thermal dissociation of the analyzed materials. When examining dried soil.
- This technology is simple and economical, in addition to:

A. Additional time is needed for sample cleaning procedures, which gives opportunities for the loss of the analyzed substance (analyzer).

B. The extraction process uses organic solvents of high purity, which are considered a burden on the environment, and this makes them equal to the benefits of the process of analyzing pesticide residues (which makes the Sorel method not widespread).

To analyze the identification of pesticides, several models of chemical and biological techniques are used, including:

1. Vital tests: enzyme test - immunoassay - radiation test.
2. Chemical tests:

- ✓ Thin layer chromatography (TLC).
- ✓ Column chromatography and microelectrophoresis (CE).

Gas chromatography (GC).

Liquid chromatography (LC).

The most widely used of these techniques are gas chromatography (GC) and liquid chromatography (LC). Currently, in addition to column fine separation (GC), sensitive and selective separators such as mass spectrometry (MS) are used.

Some pesticides that cannot be analyzed using gas chromatography are analyzed:

1. Liquid chromatography (LC).
2. Ultraviolet devices.
3. Fluorescence.
4. Photodiodes array.
5. Mass Spectrometry (MS).

Sample preparation:

The preparation of samples for pesticide analysis requires a specific mechanism for preparing samples, where pesticides are extracted either individually or in groups in several organic solvents.

Extraction transfers and converts several soil pesticides into a liquid organic state.

Traditional extraction procedures include:

Mixing the sample with polar or non-polar organic solvents, then fractionating it using (liquid-liquid) (LLP) technology with other organic solvents (determining its ability to retain water).

- ✓ Choose the extraction method (extraction).
- ✓ The type of pesticide used depends on:
- ✓ The nature of the sample.
- ✓ Chemical properties of pesticide residues.

Since the soil as an adsorbent retains pesticides on the surface.

Where it is removed from the surface using the appropriate pesticide and study its polar properties.

When extracting, in addition to the components required to be extracted, various impurities are obtained that may enter into the analysis, which requires a cleaning process for the samples before using liquid (LC) or gaseous (GC) chromatography for separation and detection (some methods neglect the cleaning steps).

Extraction procedures:

The steps of the extraction process are as follows:

Aerobic loosening of soil samples. Especially in the case of samples with high humidity such as samples of fruits, fodder, and vegetables, without applying low temperature to wet soil samples.

Wet soil samples are subjected to freezing temperatures to avoid the enzymatic dissolution of pesticide residues using dry ice.

- ➢ In the case of analyzing multiple pesticide residues, we rely on the method of ultrasonic-assisted extraction ( ) of moist soil samples without the need to dry them.

The size of the samples is /50g/ in most cases, and sometimes /20g/ and /10g/ are taken according to the case.

- ➢ Acetone is the preferred organic solvent for isolating pesticides from soil samples. Acetone can also be mixed with other solvents such as hexane, benzene, dichloromethane (DCM), and

petroleum ether (PE). Other solvents such as/ hexane, and acetate (E) are used. +Ac), dichloromethane (DCM) methanol.

Various mixtures are also used in a particular (specific) extraction process:

1. Acetonitrile (Men) with water (H2o).
2. Acetonitrile (Men) with an aqueous solution of ammonium carbonate.
3. Methanol with water.
4. Methanol with buffer.
5. Ethyl acetate Enact with an aqueous solution of ascorbic acid.

After the completion of the first step in the extraction, which is the selection of the appropriate organic solvent, we choose the method of extraction

(Liquid - Liquid) (LLE) or division to get rid of impurities, as it is considered less used/ and new techniques have been developed to clean samples from impurities.

Sample cleaning method: After homogenizing the samples with the appropriate organic solvent ((Salting out principal))

It is used to facilitate the separation of lysates so that different organic solvents with limited water-holding capacity can be extracted by adding sodium chloride.

In the traditional practice of sample preparation, complete drying of the extract is required and the presence of traces of moisture in the sample impedes the preparation of the organic solvent well, which calls for the use of sulfate. Anhydrous magnesium sulfate is a highly efficient desiccant.

Recently, new methods have been used to prepare samples by making them:

1. Can be automated 2- Reducing extraction time 3- Using the least amount of organic solvent.

To take advantage of these new technologies, it was necessary to include them as:

1. It reduces environmental risks.
2. Save time.
3. Availability of work.
4. Reducing the exposure of laboratory workers to toxic chemicals.
5. Reducing preparation costs.

Several extraction techniques began with the preparation of a model to facilitate the analysis of pesticide residues in the soil, which includes:

- ✓ Ultra-critical Fluid Extraction (SFS).
- ✓ Ultrasound extraction.
- ✓ Microwave-assisted extraction (MAE).
- ✓ Microwave and particle-assisted extraction (MAME).
- ✓ Method of segmentation (fractionation) and microwave extraction (MAE)

Surgical fluid extraction (SFE) is the only alternative technique for extracting pesticide residues from non-fat samples (instead of extraction with organic solvents).

Pesticide residue extraction protocols have been described for a limited class of pesticides (such as cleaning times for polar samples).

Rapid Solvent Extraction (ASE) Trade Name (Dionne), also known as Pressure Liquid Extraction (PLE): Small volumes of organic solvent are used to extract pesticides from a variety of samples.

The direct sample submitted (act) includes: placing a small amount of the sample material or the available extraction liquid in fine ampoules to be analyzed using gaseous chromatography (GC), which reduces the time allocated for preparing samples (by keeping them in small ampoules)

Sample cleaning:

Despite the improvements in the sensitivity of the devices, the separation of pesticide treatment from a series of samples with a complex composition, as it requires good treatment of the sample because it contains pesticide elements in addition to several pollutants in the form of accompanying extracts. The extract should be pure (purity) when conducting the detection and separation of pesticide residues in Arabia.

- Sample cleaning procedures in the case of several different samples:

(Steam distillation, oxidation, saponification, fractionation, collection over distillation, adsorption chromatography, ……..etc.).

Liquid fractionation, solid phase extraction (SPE), and gel chromatography are also used

(GPC) … etc. of the means provided to facilitate sample heating.

Several forms of absorption chromatography are used to clean samples, namely:

Column chromatography, solid phase extraction (SOE), and solid phase micro extraction (SPME).

Conventional column chromatography for sample cleaning uses:/ aluminum, silica gel, and fluorescent tube diffusion for the determination of different pesticides.

To purify different samples, apply and collect a closed box cartridge of (SPE) solid phase extraction, as they have several models:

- amino propyl - Fluorosis - ENVI-Carb

To separate pesticides from the soil, mixtures of classic extracts with organic comet soil are applied with solid phase micro extraction (SPME).

*** Detection and separation:**

Pesticide residues can be determined with the help of any technique such as microbiological, enzymatic, dusty chloride (spectral chromatography), electrophoresis, electrophoresis, etc.

Organ phosphorus pesticides (OPP) are analyzed by enzyme inhibition tests and are currently replaced by (chloride chromatography spectroscopy) methods.

Nowadays these methods are not feasible as thin-layer chromatography (TLC) methods cannot be used frequently.

Some techniques showed success in analyzing pesticides and had a high analytical capacity, including:

- ✓ Gas chromatography (GC).
- ✓ High-performance liquid chromatography (VPLC).
- ✓ High-performance liquid chromatography (CE).
- ✓ Micro capillary electrophoresis (CE).

* When linking the spectrophotometer with gas chromatography (GC) or high-performance liquid chromatography (HPLC), the resulting analyzes of pesticide residues were accurate.

-- To determine organic chlorine pesticides (OC), neutron activation analysis (NAA) was used.

Pesticide analysis is also done:

1. Using low-pressure gas chromatography (LP-GC) with sequential mass spectrometry (MS-MS).
2. Capillary gas chromatography with atomic radiation detection by a different mass spectrometer (MS) detector.

The main benefits of UPC compared to HPLC are that it has faster analysis, narrow peaks (gives increased signal…..), and emphasis on pesticides destined for analysis.

Direct surface analyses are carried out using static secondary ion mass (SIMS), which is satisfactorily used to identify various pesticides such as:

Alachlor, atrazine, cabin, carbofuran, chlorpyrifos, chlorsulfuron, chloral-dimethyl, D2.4, durned, glyphosate, marathon, methyl, acid, mean, oxbow and refine, Parquet, Thai Milk ,and Tri-Flora line.

The direct surface analyses of the sample are useful for the analysis of traditional pesticides before the sample is treated by the extraction method, as the types of analyzes have developed. The total time for analysis using SIMS secondary ion mass spectrometry was about / 10 minutes per sample.

Nude electrophoresis (CE) is an accurate separation technique suitable for the analysis of different types of chiral and achiral pesticides. We also detect by integrating before columns selective derivatives, charts, and sensitivity of detection methods, for example:

Detection using a fluorescent laser launcher.

Where the enrichment technique (CE) is critical to determining the pesticides and their trace levels to remain satisfactory.

Soil properties are related to humic acid (HA) as it affects the transfer and … of pesticides. It is used in the control of pesticides and immobilizes them, and thus helps in the preparation of chromatographic column preparations and reforms.

Rodenticides and herbicides are analyzed in this column, where hemic acid (HA) is observed to be more polar than neutral pesticides, however, the column remains stable over a long period and is considered more specific for pesticide hazard estimation.

Contemporary methods of analysis:

The basic steps for the analysis of pesticide residues in soil are limited in this section

1: To separate and isolate Chbrpyoifos from organ phosphorous pesticides (OPP):

50 g of soil samples are taken and shaken with 100 ml of a mixture of acetone-hexane at a ratio of (9:1) on a mechanical shaker for one hour (1 hour), then the mixture is taken and filtered through anhydrous sodium sulfate (dried material), then it is exchanged for (10 g) hexane before it is

analyzed by gas chromatography.

In another way, (10g) of soil is extracted three times with (50 ml) of acetone and divided into (50 ml) of hexane three times.

2: To extract Endosulfan:

/ 50 g / soil mixed with (0.5 g) activated charcoal and fluorosis and (10 g) anhydrous sodium sulfate. The mixture was shaken with (100 ml) acetone-hexane (1:9) for one hour (1 h).

3: To separate and isolate Chbrpyoifos and Endosulfan together:

It is taken / 100 g / soil mixed with / 0.5 g / activated charcoal and / 10 g / anhydrous sodium sulfate and extracted by the socket device with a mixture of n-hexane and acetone (9:1).

4: To extract multiple residues of organic chlorine and organic phosphorous pesticides from soil samples with high humidity:

where (LLP) liquid-liquid fractionation is used, where a sample / 50 g / soil is taken from the river bed and shaken with / 150 ml / of a mixture of dichloromethane and acetone at a ratio of (1:4) for four hours on an automatic shaker, then re-extraction with (50g) dichloromethane after adding (6ml) of aqueous phosphoric acid solution (1ml of O-phosphoric acid + 5ml of water) for twenty minutes and in combination with DCM extract exchanged with (10 ml) of N-hexane before etching.

5: In the case of extending several pesticides from organic chlorine derivatives OCPS:

/ 50 g / soil mixed completely with / 150 ml/manganese cyanide mixture with water at a ratio of

(2:1) kept overnight, then the extract was filtered through Whitman filter No. (1), then filtered and dried with (600 ml) of aqueous sodium chloride solution at a percentage of

(5% in a liter of water) in the separation pool and divided twice with (100 ml) of N-hexane.

6: In the case of residues of 6 industrial parathyroid pesticides and 15 organic chlorine pesticides:

Successfully extracted from / 50 g / bottom soil or air-dried sediment by mixing with a mixture of hexane - acetone in a ratio (1:4)

- Incidence of the effect of quantities of pesticides in the soil by: liquid phase micro-extraction (LPME) in combination with gas chromatography and mass spectrometry (GC-MS).

This technique involves the use of a small volume (3 µL) of an organic comet impregnated in the membrane fiber space, which is attached to a conventional gas chromatography needle syringe.

Various features of this method were developed such as:/ the organic comet test, extraction time, piston guard pattern, concentrations of hemic acid and salt, and part of the organic comet, in soil samples.

The detection incidence of (LODS) was between (0.05) and (0.1) µg/g with gas chromatography and mass spectrometry under selective ion control (SIM).

Also, this method gives a good accuracy ranging from (6% to 13%), and the relative standard for deviations was less than (10%) for most of the targeted pesticides. This process is completed within minutes.

7: For the extraction of Carbofuran insecticide:

/ 20 g / soil extracted with / 100 ml / of a mixture of / 1:9 / of methanol and bier at / PH = 8 /. Where the pesticide is divided three times with / 50 ml/dichloromethane. After concentrating the detergent on the silica gel rod, it is carried out before desalination on the E CD-Electron Capone detection gas chromatography.

8: Residue of quiralphos = prorate is done:

It was extracted with 10% aqueous acetone and divided into three sections with / 50 ml / of dichloromethane (DCM), after concentration it was cleaned on a silica gel column before analysis on gas chromatography with a thermal ionization detector (T1D).

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