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Analytical Techniques for Pesticides Detection in Food Commodities

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Abstract:

Pesticides are those substances that prevent, destroy or control a harmful organism (pest), or disease and protect the plant, crops during the production. The use of pesticides are increasing worldwide due to large population and their needs. Small quantity of the pesticides is used to kill or control the pest whereas, left pesticides remain in environment or absorbed by the food material, water is harming the health of humans, and aquatic life. Thus, the screening of these pesticides is important to ensure legally acceptable quantity 'MRLs' of pesticides that should be used in controlling the pests or disease. Different techniques such as Gas Chromatography-Mass Spectrometry (GC-MS), High Performance Liquid Chromatography (HPLC), Liquid Chromatography-Mass Spectrometry (LC-MS), Enzyme Linked Immunosorbent Assay (ELISA) and so on have been developed to detect the pesticides. These techniques have high sensitivity, high accuracy, reliability and less time consumption capability but some techniques have drawbacks i.e., HPLC has a low sensitivity for pesticides in food. This review paper studies different analytical techniques for the detection of pesticides in food.

Keywords: *Pesticides, MRLs, Chromatography*

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Introduction

Chemical substances are those substances which are used in agricultural field to increase the production with high quality by controlling the pests is known as pesticides. These pesticides include insecticides, herbicides, fungicides, disinfectants and various other substances. The mechanism of pesticide is that it attacks on the targeting system or enzymes in the pests. This mechanism is also followed in the case of humans because of the identical or very similar system or enzymes in humans. The pollution in air, soil and water is increasing by the greater use of pesticides. The health of humans are at very high risk, not only by misuse or accidental ingestion but also by the traces of chemicals left into the environment that is affecting the health. Pesticides not particularly target pests, it also affect the non-target plants and animals. According to the researchers, only 0.1% pesticides reach to the targets while left pesticides contaminate the surrounding environment (Elhag *et al.,* 2017).

Generally, all creatures, human and environment are facing the problems related to the pesticides but the high risk of exposures is seen in cases of agriculture worker and family members of pesticides applicators. There are different reason of pesticides exposure with the humans which are as follows:

- Spillage from the packaging
- Applying too much pesticides product
- Accidentally, exposure the chemicals during aerial treatment or from spray drift.
- Incorrect use of equipments.
- Failure to follow the label instructions
- Storage of pesticide in unsecured places (Darcin and Darcin, 2016)

Classification of Pesticides

Pesticides are classified on the basis of their use such as insecticides, miticides, herbicides, nematicides, fungicides, molluscicides and rodenticides due to large amount of chemicals and pesticides combination of compounds. Another classifications of pesticides were given by World Health Organization (WHO) based on their health risk. Most important and useful classification is based on the chemical structure which can be divided into four main groups.

Organochlorine: These are stable compounds that persists in the environment and have a tendency to accumulate in fatty tissues. It is used in the destruction of disease vectors such as dengue and malaria. For the preservation of grapes, lettuce, tomato, alfalfa, corn, rice, sorghum, cotton and wood Organochlorine is used during cultivation. These pesticides or their metabolites directly act on Central Nervous System (CNS) of human that alter the electrophysiological properties and enzymatic neuronal membranes due to which change in the kinetics of the flow of sodium and potassium through the membrane of the nerve cell causing symptoms such as seizures and respiratory arrest.

Organophosphates: These are the esters of phosphoric acid. The mechanism of these compounds are as they act on the Central Nervous System (CNS) and inhibits the acetyl cholinesterase enzyme due to which amount and level of neurotransmitter acetylcholine is modulated and nerve impulse is disrupted by serine phosphorylation of hydroxyl group in the active site of enzyme. This mechanism creates many symptoms such as loss of reflexes, headache, dizziness, nausea, convulsion, coma and even death. Organophosphorous compounds are used in agricultural field such as vegetable crops, fruit trees, grains, cotton, sugarcane, and others.

Carbamates: These esters are derived from the acids or dimethyl N-methyl carbamic acid. Carbamates are used in the form of insecticides, herbicides, fungicides and nematicides. They have less value in comparison to organophosphates and Organochlorine but have the same effect as the organophosphate act on acetyl cholinesterase. But the action in Carbamates is very fast, the carbamylation of the enzyme is done through the kinetics of blocking by the covalent attachment of electrophilic groups steric carbamoyl sites of the enzyme.

Pyrethroids: These pesticides is originated from the natural insecticides which are derived from pyrethrum extract from chrysanthemum flowers (pyrethrins**).** They also act on the Central Nervous System (CNS) and make changes in the nerve cell by altering the dynamics of the sodium channels that cause the increment in the opening time of nerve cell due to which sodium current flows for a long time across the membrane in both insects and vertebrates (Garcia *et al.,* 2012).

Pesticide Residues

The deposits of pesticide active ingredient, metabolites, breakdown products of pesticides are present in the components such as food grains, fruits and vegetables, soil and water are known as pesticide residues. This residue analysis gives an idea about the nature and level of any chemical contamination in the environment. Extensive efficiency, environmental and

toxicological testing of the pesticides should be registered by Governments for legal use in specified applications. Regulatory bodies set the types and amount of residues should be present legally in the food articles is called Maximum Residue Levels (MRLs). These MRLs are called as 'tolerances' in the United States (Dasika, Tangirala and Naishadham, 2011).

Analytical Techniques

In determination of pesticides from analytical techniques, sample preparation is the main step in which extraction of analytes from matrix bulk is performed and then the clean-up the contents from any co-extractives presence in the matrix. There are different methods for the extraction of Pesticides form the matrix such as Liquid-liquid extraction (LLE), Solid-phase extraction (SPE), Matrix solid-phase dispersion (MSPD), quick easy cheap effective rugged and safe (QuEChERS), Solid-phase microextraction (SPME) and so on. After the extraction and clean-up of the sample from the matrix, next step is the detection of targeted pesticides. For the modern multi-residue target pesticide analysis, the most common techniques are Gas Chromatography, Liquid Chromatography coupled to Mass Spectrometry (GC-MS, LC-MS) and Tandem Mass Spectrometry (GC-MS/MS, LC-MS/MS) with triple quadrupole mass analyzers and High Performance Liquid Chromatography (HPLC). Other advanced techniques are electrochemical sensors, optical sensor, and immunosensors and enzymatic biosensor for the analysis of pesticides (Samsidar, Siddiquee and Shaarani, 2018).

Review of Literature

Dasika, Tangirala and Naishadham (2012) proposed a combination of Liquid Chromatography Tandem Mass Spectrometry (LCMS/MS) in their paper for the analysis of pesticides in fruits and vegetables and gave a conclusion that fast and easy qualitative screening of target pesticides within 45 min of LCMS/MS.

Bresin *et al.* (2015) discussed about the analytical technique 'Gas chromatography-mass spectrometry' with the extraction method QuEChERS for caffeine removal from the extract. GC-MS have a high sensitivity and less time consuming for the detection of pesticides. Same benefits are with the liquid chromatography tandem mass spectrometry (LC-MS/MS).

Rani and Dhiraj (2015) studied on water to detect the pesticide 'organ phosphorous' by High Performance Liquid Chromatography. This method showed accuracy, linearity, precision and robustness. And this method can be used without sample preparation (separation of pesticides from the aqueous phase).

Stachniuk and Fornal (2016) concluded that the combination of liquid chromatography with selective mass spectrometry is a change in the analytical field. From this technique, detection of many compound with trace amount has been easy. He also discussed about the high performance liquid chromatography coupled with QQQ tandem mass spectrometer that have the short duration of analysis by determination of number of compounds in a single analytical cycle. But this HPLC/MS has a limitation of sensitivity to the accompanying matrix components.

Tette *et al.* (2016), In their paper find out the quantity of pesticides in honey using modified QuEChERS for sample preparation and the Ultra high performance liquid chromatography mass spectrometry (UHPLC-MS/MS) for the detection and concluded that this method is fast efficient and reliable.

According to Rimawi (2016), High performance liquid chromatography is a simple, accurate, precise and selective method for the determination of pesticides in water. In this study, the detection and quantification of pesticides in water was possible at low concentration. This method is suitable for real water samples such as groundwater, surface water, and waste water**.**

Chang, Hsieh and Chiu (2016), after the analysis of pesticides in environmental sample by Capillary Electrophoresis Mass Spectrometry, they concluded that Capillary Electrophoresis has high versatility, high efficiency and selectivity due to which it has become more powerful technique for the screening of pesticides.

Djue Tea, Sabarudin and Sulistyarti (2017) analyzed the pesticides Diazinon and Chlorantranilipole in soil sample from High performance liquid chromatography with ultra violet detector in their study and concluded that both pesticides were detected in real sample above 85%. And in the suspected sample there was not diazinon as well as chlorantranilipole.

Elhag *et al.* (2017) in their paper, they detected the Organophosphorous pesticides in the vegetables by Gas chromatography-mass spectrometry and gave a conclusion that It is a powerful technique to determine the pesticides at low levels. And gave a suggestion of periodic monitoring programs to reduce exposure, accumulation and toxicity of pesticides.

Nazir, Rafique and Ahad (2017) analyzed a pesticides in honey sample by microextraction

technique with Gas chromatography-Electron coupled plasma. GC-µECD is a best technique for the pesticides analysis. GC and LC with mass spectrometry are also suitable technique for the detection of accaricides and neonicotinoid pesticides in honey.

Samsidar, Siddiquee and Shaarani (2018) discussed several analytical techniques such as GC and HPLC with selective detectors NPD, ECD, MS DAD AND fluoresces detectors for the detection of pesticides in the matrix because of the high sensitivity. But these techniques are time consuming and expensive. To overcome from this limitation, new advanced techniques enzymatic biosensor has been developed to detect the pesticide residues. This device is ecofriendly and have low cost compare to other analytical devices.

Conclusion

Pesticides that are used to kill or control the pests, also have an adverse effect on the humans, aquatic life and plants. These pesticides produce the different types of diseases such as cancer, endocrine disease, asthma, learning disability etc. Different types of technique are used to detect the pesticides in matrix such as GC-MS, LC-MS/MS, HPLC, ELISA etc. This review concludes that the analytical technique 'Liquid Chromatography Tandem Mass Spectrometry (LC-MS/MS)' is a best technique for the detection of pesticides in matrix (grains, fruit, vegetable, water and soil) as it is reliable, have high sensitivity, and consumes less time.

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