Review Article

Detection of pesticides in environmental and food matrix: A review

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ABSTRACT

The Pesticides are widely used as the key to increase the agricultural yield in the world. Pesticides are used incautiously and in uncontrolled manner to compensate shortage of food due to ever increase in population and rapid urbanization. Because of this, concentration of pesticides in environment and food matrix is increased which causes difficulties in maintaining healthy lifestyle and becomes a root cause of multiple diseases. Hence, strict rules and regulations are made which are followed by government and are bound to regularly monitor these compounds. With time, numerous traditional to conventional methods are developed for extraction and detection of these compounds from environmental and food matrix. The present study explains overview of all-inclusive traditional to advanced methods for pre-treatment and detection of pesticides with its residues from environmental and food matrix. Also discussed about comparisons between these methods with applicability, advantages and disadvantages over one another.

1. Introduction

Food and water are essential to maintain life and it cannot be overemphasized. To maintain a healthy nutritional diet, food and water would not be contaminated from pollution.

Food and water contamination are undesirable event at the land and aquatic topographies as it roots cause of ill health and ultimately could lead to death of the affected organisms. Foremost cause of contamination is sewage from factories, fertilizer and pesticides. Of all the pollutants, utmost contaminants are pesticides.[1] Pesticides are chemicals that protect crop from unwanted insects, weeds, fungi, or other objectionable organisms that might harm the crop.[2]

The globally 2 million tons per year pesticides are used out of which 45 percent is used by Europe alone, quartile portion is used by the United State of America, and quartile portion is used by the rest of the world. India's annual pesticide consumption is just 3.75 percent. The share of uses of pesticides in Korea is 6.6kg ha^{-1} and Japan is 12.0 kg ha^{-1}, while in India, it is only 0.5 kg ha^{-1}. Worldwide, the pesticides covered 1/4 of the farming land.[3]

Leaching is type of environmental pollution which is caused by extreme use of pesticides on farming land. By this, possibilities of contamination of surface water are creates when irrigation water that has omitted pesticide-treated plants and drain into the surface waters.[4] Storms might result in spontaneous flow of polluted water into surface water.[5] Another source of pollution is drift that happens if mistakenly pesticide spray misses its targets having ricocheted by the wind or resulting from the error of missing the target, thereby applying on a non-targeted land. When the level of the pesticide contamination reaches a critical level in food and water bodies, it becomes matter of illness or death in the organisms.

Cereals, fruits and vegetables are among the most commonly grown foods in many parts of the world. In the India, major harvesting crops are Wheat (Triticum

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aestivum), Corn (sweet corn- Zea Mays), Rice (Oryza sativa), Tomato (Solanum lycopersicum), Cabbage (Brassica oleracea), Cucumber (cucumis sativus) and Honeydew (cucumis melo). Wheat, corn and rice are cereal crops mainly consume in India. In year 2013, wheat (713 million tons) is the third largest harvest crop worldwide followed by rice (745 million tons) is the second and corn (1,016 million tons) is the first.[6]

Wheat contains about 8-15% protein and therefore serves as a good source of vegetable protein.[7] It is the main ingredient in many bakeries and fast food menus world-wide. Rice is rich in dietary fiber and some vitamin complexes like nicotinic acid (niacin), riboflavin and thiamine.[8] Corn is a good source of fibrous proteins.[7]

Table 1: Annual consumption of pesticide in Asian countries[8]

S.No.	Country	Tonnes Pesticide
1	China	1,80,700
2	India	56,120
3	Malaysia	49,199
4	Pakistan	27,885
5	Thailand	21,800
6	Vietnam	19,154
7	South Korea	19,788
8	Bangladesh	15,833
9	Myanmar	5,583
10	Nepal	454
11	Bhutan	12

The exposure of pesticides in humans and animals primarily occurs through water and food. Pesticide exposure is ubiquitous[9] and therefore, humans are at high risk of exposure from these chemicals, and even higher if you visit frequently areas that use more-than-normal levels of pesticides like large-scale agricultural operations. Human exposure to pesticides is persistent and can occur through different routes, not only from occupational exposure dealing with production, transport, delivery and application of pesticides, but also from the wide-ranging use of these products in households and flow and accumulation of pesticides in the food chain.[10,11] Various reports suggest the risk behind the intake of different pesticides with different modes of action, continuous exposure to pesticides causes depression and neurological deficits, diabetes, respiratory diseases such as rhinitis and in extreme cases, it causes cancer, fatal death, spontaneous abortion and genetic diseases.[12]

Shows the 15 targeted pesticides which are added as persistent organic pollutants (POPs) by Stockholm Convention, 2019.[13] These are the only priority pesticides and beside these, many pesticides and their metabolites are persisting which are toxic and bio-accumulative and are detected in different environmental matrix including food (animal origin and plant origin) and water.

Table 2: Stockholm convention POP's list

| S. No.	Annexure A	Annexure B
1	Aldrin	DDT
2	Alpha hexachlorocyclohexane (Alpha-HCH)	
3	Beta hexachlorocyclohexane (Beta-HCH)	
4	Chlordane	
5	Chlordecone	
6	Dieldrin	
7	Endrin	
8	Heptachlor	
9	Lindane	
10	Mirex	
11	Pentachlorophenol and its salts and esters (PCP)	
12	Endosulfan and its related isomers	
13	Toxaphene	
14	Dicofol	

The Environmental Forensic is the combination of multiple branches of science primarily including environmental science, law and analytical chemistry. The estimation of persistent organic pollutants (POPs) for the purpose of environmental forensic analysis requires selected methods that covers long range of targeted analytes which can be analysed qualitatively and quantitatively in the environment.[14] Forensic investigation involves the identification and establishment of the source of pollutant, which will occur from an immediate chemical spill, fugitive emissions of chemicals from manufacturing plants, transportation and storage of consumer products and leaking or leaching from final products. A post processes of release of POPs i.e. volatilization and dispersion of POPs, biodegradation of materials like polymers, uptake in environment matrix, biotransformation into different unknown compounds and elimination from main matrix may create difficulties in identification of source.[15] This causes difficulties in analysis of contamination of environmental matrix for POPs. Therefore, the accurate analysis of sample should be required for which sample selection and collection, sample preparation, and selection of method of analysis is the significant part of analysis.

The aim of this study is to provides an overview on analysis of pesticides from different environmental and food matrix using different conventional and traditional sample preparation (extraction) and detection methods and form comparison between these methods with defining advantages and disadvantages of each on one another.

2. Sample Preparation

Preparation of sample is the primary phase of any analysis, including collection of samples, and its pre-treatment. Here

in case of pesticide analysis from environmental and food matrix, pre-treatment involves the extraction of pesticide with its analytes from core matrix.
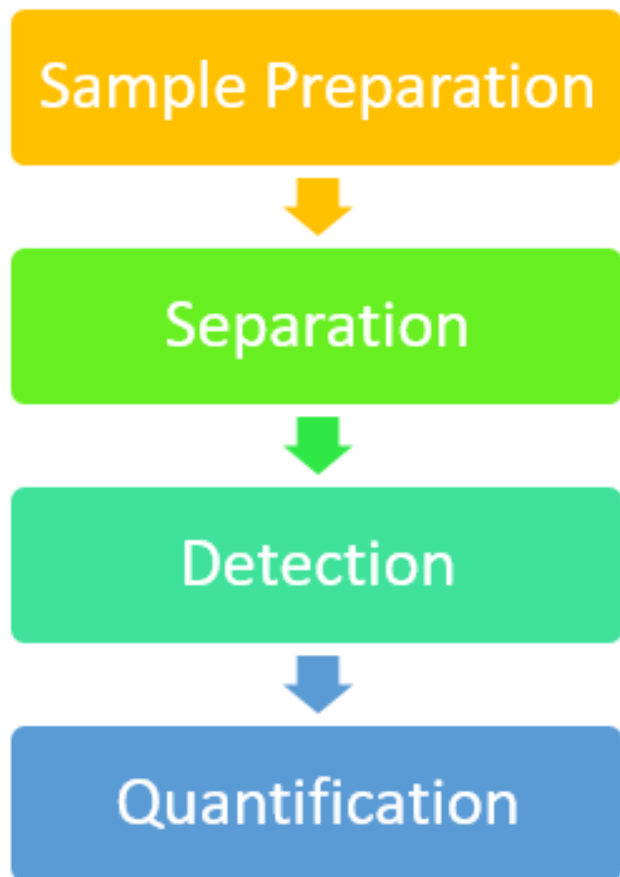
Fig. 1: Systematic approach to analyse environmental and food samples

3. Collection

A well-planned sampling program is essential for any environmental forensic investigation. There are many factors that can possibly influence data and lead to inaccurate results and these need to be understood if they are to be defended in a courtroom. Murphy and Morrison (2014) explained in detail about the collection and processing of environmental material for forensic analysis[16]. For the investigation of any contaminant it is essential to have knowledge about the environmental chemistry so that the selection of evidence or matrix should be carried out properly. The analysis of evidence or matrix should be based on nature of matrix and the queries asked by assigning authority. The commonly asked queries are; what is the source of contaminant, what is the range of contamination, is any wildlife is harmed from contaminant and is it under the threshold limit or not? For answering each question, the different type of evidence or matrix is collected and

analyses using different techniques. Figure 1 told us about the systematic approach to analyse environmental and food matrix.

4. Extraction

Extraction is very typical procedure in which the required material or compound is saperated from whole matrix. For the analysis of targeted pesticides from various food matrisex requires different extraction procedures and the selection of this procedure depends on the type and nature of analytes as well as matrix. In previous literature, no any ideal method is defined for the saperation of pesticide, but analytical laboratories follows their own modified system which depends on the properties and nature of matrix, i.e. whether the food belongs to animal origin or plant origin and how much amount of fat is present in food.

Besides this, extraction procedure follows a typical pathway involving the discharge of desired analyte from the matrisex, followed by refinement process which encompasses step or series of steps for analytical procedure during which mainstream of undesired co-extracts are removed by chemical and physical treatments. Prior to apply or follow any extraction method, the sample needs pre-treatment by which sub-samples are prepared. Pre-treatment includes maceration and homogenization of original sample which is done by chopping the sample using any powerful chopping device, and mixing the homogenized sample with suitable solvent for advancement of recovery. The most common solvents used for extraction of pesticides are MeOH, MeCN, Acetone, Ethyl acetate, Benzene, Hexane etc. and some times the mixtue or combination of this solvents are also used. Addition to this, some salts are also used which is used to neutralize the prepared sub-sample. The most common salts used for the extraction are NaCl, $MgSO_4$ etc.[17–19]

In intitial days, the liquid-liqid extraction and solid-liquid extraction methods was used because of limited resources but from last 10 years with the development of resources many other methods are introduced in which each of them having specific advantages and disadvantages on other methods. The most common extraction procedures previously followed are described below.

4.1. Liquid -liquid extraction

Liquid -liquid extraction (LLE) is also called solvent extraction or partitioning which applies to liquid matrix. The separation of analyte and matrix is based on their relative solubilities in comparatively immiscible liquids. LLE comes in the category of most adaptable and reliable techniques of extraction of pesticides which shows compatibility with most of the instruments. Different non-polar extraction solvents such as n-hexane, benzene and ethyl acetate and water-miscible solvents such as dichloromethane, methanol,

MeCN and/or acetone and water solvents are employed for the extraction of pesticides or analyte from its matrix.[20] Mondal et al. (2018) employed the conventional LLE technique for pesticide extraction from river water sample and found optimal extraction with Ethyl acetate and DCM (8:2, v/v).[21]

4.2. Solid phase extraction

Solid phase extraction (SPE) is a simple and rapid method developed as an alternative to LLE for the separation, purification, concentration and /or solvent exchange of solutes for solutions and also having the capability to treat a large volume of samples with high recovery.[22] Different solvents like acetonitrile, methanol, ethyl acetate, dichloromethane, acetone, acetic acid, hexane, toluene, petroleum ether, cyclohexane, diethyl ether is employed in this technique for the efficient separation and extraction of pesticides which is decided based on molecular characteristics of the pesticides and its polarity. Extraction or sample preparation and analysis of residues of pesticide from fruits and vegetables is done by wide range of SPE cartridges.[23–25] The Solid phase extraction method is well known to be the fastest and most effective method for the extraction of pesticides.

4.3. Solid phase microextraction

Solid phase microextraction (SPME) is a rapid and simple sample preaparation method having the outstanding characteristics like solvent free, fast, handy and poratble method established by Arthur and Pawliszyn (1990) in 1990.[26] This extraction method is working on the principle of the partitioning of analytes between the matrix and the immobilized solid phase microextraction fibre. The extraction and concentration of pesticides or analyte is done in a single step. SPME involves two different steps: an extraction (retention of the analytes on the stationary phase) and a desorption step. Extraction step is influenced by some factors like fibre type, extraction time, ionic strength, sample pH, extraction temperature and sample agitation and the desorption step is influenced by some variables like temperature, desorption time, focusing oven temperature, and solvent employed and its volume. SPME for food samples, especially in fruit and fruit juice involves an important factor, called matrix effect. The reduction of negative matrix effect can be done by diluting the samples 50 to 100 fold with distilled water. Zhang et al. (2019) performed SPME coupled with GC to analyze Diazinon and Chlorpyrifos from apple matrix with LOQ and LOD of 0.60, 0.18 and 0.67, 0.20 respectively.[27] Sanganalmath et al. (2019) also used SPME for extraction of Quinalphos from post-mortem blood sample, they found the maximum 88.04% extraction on using Diethyle ether as solvent.[28]

4.4. Matrix solid phase dispersion

Barker, Long, and Short (1989) primarily discused this method for the extraction of analytes from solid and semi-solid samples.[29] The procedure is made easy by integtaing extraction and cleanup into a single step. It is a quick method with less loss of sample and solvent consumption. This technique allows for extraction of pesticides from homogenised food matrix from solid support such as the synthetic magnesium silicate (Florisil) or silica (C8 or C18). It causes good recovery and reproducibility of samples after extraction. The method is less time and solvent consuming. Multi-residual methods based on matrix solid phase dispersion using alumina, silica and Florisil were for analyses of pesticide residues in vegetables is described in previous literature. Recoveries using all sorbents were found similar in literature, while extracts from Florisil were the cleanest. Amongst the three eluting systems used in MSPD, dichloromethane is the best.[30]

4.5. Quick, easy, cheap, effective, rugged, and safe method (QuEChERS)

This latest and advanced technique is based on extraction by acetonitrile solvent proceeded by a clean-up of analyte using dispersive-solid phase extraction method (d-SPE). The use of acetonitrile in this extraction procedure is possible due to its high recovery. It has been widely employed due to its microscale extraction procedure which is responsible for the simplicity of this technique.[31] The technique requires less amount of solvent and time than all the previous methods that is why it is also environment friendly technique.[32,33]

4.6. Gel permeation chromatography

Gel Permeation Chromatography is a separation technique for macromolecules such as lipids, proteins, polymers or dispersed highmolecular–weight compounds from the sample. The technique GPC is based on principle of size-exclusion of analyte with pores of gel. For separation pre-sample solution is introduce into the Gel permeation chromatographic system equipped with column preceded by a guard column. Chromatography was performed with suitable mobile phase. It uses organic solvents or buffers and porous gels for separation. The packing of gel is decided by a given exclusion range i.e. size of analyte we have to separate from matrix and pore size, which must be larger than the targeted analyte. This technique is most preferable for sample cleaning-up purposes. Gel permeation chromatography cab be used for cleansing or selection of extracts from complex matrix containing a wide range of analytes could be polar and/or non-polar.[34]

4.7. Dispersive liquid-liquid micro-extraction

Dispersive liquid-liquid micro-extraction (DLLME) was developed for the analysis of polyaromatic hydrocarbon and residues of organophosphorus pesticides from water samples.[35,36] This technique uses small amount of a mixture of extraction and dispersive solvents with high miscibility, thus avoiding the dislodgement of the organic solvent drop inherent. A whitish solution is formed when an suitable combination of high-density water-immiscible extraction solvent and dispersive solvents are injected rapidly into an aqueous solution of the sample matrix[35,37,38] containing the analytes. The only drawback is persisting with this technique is it is a manual procedure that requires centrifugation, which is time-consuming. Automation based on a sequential injection system has been used to overcome the drawback.[39] The concentration of analytes is then enhanced into the extraction solvents, which are isolated into the bulk aqueous solution when the mixture is centrifuged, thus the technique dispersive liquid-liquid micro-extraction (DLLME) is also known as, two-step micro-extraction technique. After centrifuging, a sedimented phase of the extraction solvent accumulates at the bottom of the extraction vessel and can be injected into analytical instruments[39,40] with or without further treatment (clean-up). The selection of the type and volume of dispersive solvent is as important as that of the extraction solvent, because it helps the extraction solvent to form fine droplets in the sample matrix and ensures a high enrichment factor.[41]

4.8. Microwave assisted extraction

This method involves the extraction of food samples by suspending them into some traditional solvents like n-hexane, methanol-water and treating it in a kitchen-type microwave oven for 30 seconds (at frequency of 2450 Hz), without allowing the sample to boil. Repitition of irradiation step should be done several times to produce maximum yield of extracted compound. Then, centrifugate the samples and supernatant should be removed for chromatography.[42] The Microwave assisted extaction is more efficient than conventional methods as the yield is also good. This method is appropriate for extractions of wide range of sample due to less consuption of time and labour.[43]

4.9. Soxhlet extraction

Soxhlet extraction was firstly implemented by Mutua et al, (2015) and further it was optimized for extraction of different analytes. The optimization is done on the basis of analyte and sample matrix, which is selection of extraction solvent and sample wetting.[44] Suitable amount of sample is place in a cap which will then load into a chamber of Soxhlet extractor and further place into a flask containing 100 mL of methanol. Soxhlet was fitted with the condenser and refluxed at 85°C for 24 hours. Thereafter, the extract was reduced to 1 mL using a roto-evaporator. It was then transferred into a 100 mL volumetric flask then top up with distilled water. SPE was then applied under optimum conditions for analytes clean up.[45]

5. Instrumental Techniques for Detection

Due to the versatility in physical and chemical properties of matrix, it is very difficult to develop ideal method for detection of pesticide. In last two decades, GC and LC techniques are used greatest for detection and quantification of pesticides in fruits, vegetables and cerals due to their sensitivity, identification and separation capacities. Apart from these, other methods like enzyme-linked immunosorbent assay (ELISA) and capillary electrophoresis (CE)[46] has also been used for detection of pesticide residue.Figure 2 gives us information about techniques to analyze environmental and food samples for detection of pesticides and its residues.
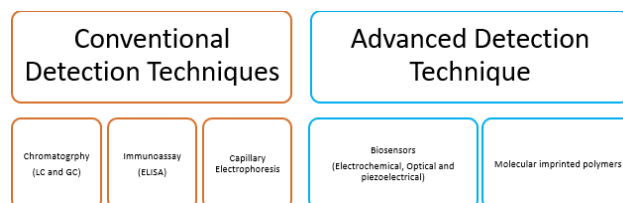
Fig. 2: Techniques to analyze environmental and food samples

5.1. Gas chromatography (GC)

Previous literature defines the state of detection of pesticides which is carried out by Gas chromatopraphy coupled with several detectors. Due to their sensitivity, detectors like electron flame photometric detector (FPD),[47] capturing detector (ECD),[48] mass selective detector (MSD), and nitrogen phosphorus detector (NPD) are used.[48]

In addition, mass spectrometry are also used to improve sensitivity of detection which are equipped with analyzers such as Quadrupole,[49] ion trap (IT),[50] time of flight mass analyzer (TOF),[51] triple quadrupole (QqQ).[52] Further, to decrease interference of matrix or effect of matrix on analysis selective ion monitoring (SIM)[53] or multiple reaction monitoring (MRM)[54] are used, and mass to charge ratio (m/e) of analyte are focused to achieve a lower limit of detection and quantification with less interference. Húšková et al. (2009) was reported method for the analysis of residues of OP, OC and carbamate pesticides by use of gas chromatography coupled with negative chemical ionization mass spectrometry. Most of the GC separations is carried out with fused silica 30 mm x 0.2 mm i.d., 0.25μm using helium or nitrogen gas as a carrier.[55]

The use of GC methods for pesticides has been decreased since from last decade due to envolvement of highly toxic and less persist polar pesticides which are found inapt for the GC detection methods due to their volatile nature and thermaly unstability.

5.2. Liquid chromatography (LC)

From the literature of last decade, numerous liquid chromatography technique are defined for detection of pesticide and their residues, most of them are coupled with different detectors like photodiode array (PDA), ultraviolet (UV), mass (MS) detectors and diode array detector (DAD). The octadecyl (C18) column is the most commonly used stationary phase for the liquid chromatographic separation. It has been used for multi-residue analysis and decreases the runtime in gradient mode. Wang et al. (2012) was performed multi-residual analysis for seven different neonicotinoid insecticides by employing high-performance liquid chromatography (HPLC) coupled with DAD and separation was achieved by aglient TC-C18 column.[56] Also, AlRahman, Almaz, and Osama (2012) was developed method to estimate rate of degradation of acaricide and fenpyroximate pesticides in apple, grape and citrus by analyzing with HPLC-DAD technique.[57] Apart from the above methods, Wang et al. (2014) has also reported a method for the use of molecular imprinted solid-phase extraction for the detection of trichlorfon, monocrotophos by HPLC.[58]

Even though liquid chromatography coupled with detectors like UV, PDA and DAD systems have been used, it becomes difficult to provide structural information for the identification of residual content of pesticides from food matrix. The detection of mass of analytes has been useful for these structural intercessions and also to provide structural information from fragmentation pattern and molecular masses by tandem mass spectrometry (MS/MS).

Numerous studies are done by liquid chromatography coupled with mass detection techniques For this different reverse phase columns have been used as the stationary phase like C-8, C-12 and C-18 (differ in pore size) with different organic mobile phases (eg. acetonitrile and methanol) and buffers (eg. formic acid, ammonium acetate, ammonium formate, acetic acid). Mixture of solvent are also used as mobile phase (eg. water-acetonitrile and water-methanol) in gradient mode with flow rate ranging from 0.2 - 1.0 mL/min.[59]

In Mass detection, ionization source like electrospray ionization (ESI) are frequently used.[60] It has ability to ionize both polar and non-polar analytes. In addition to, mass analyzer, Q-Trap[61] and triple quadrupole (QqQ)[62] is also useful for qualitative and quantitative analysis. Apart from these analyzers Bakırcı and Hışıl (2012) was described a multi-residual method for the analysis of pesticide and their residues (total 128) using single quadrupole detector[47].

Guan et al. (2011) was also reported a new method for the estimation of organophosphate pesticides from Vegetables and fruits using LC-MS/MS method equipped with ESI and QqQ-MS.[63] Further, Tian et al. (2016) was reported a different method for simultaneous determination of penflufen pesticide and its one metabolite in cereals and vegetables by employing a modified QuEChERS method with addition of LC-MS/MS.[64] In reference to the use of LC-MS and MS/MS, a new method has been reported in the past few years with the use of ultra-performance liquid chromatography (UPLC) due to its chromatographic efficiency and sensitivity to analyze pesticides in food stuffs. Carneiro et al. (2013) and Liu et al. (2010) also developed a method for the determination of pesticides in vegetables and fruits by QqQ-MS using ESI.[65,66] Mastovska et al. (2009) was described method for multi-residue analysis of pesticides from cereal grain using the QuEChERS method combined with the automated direct sample introduction in UPLC-MS/MS.[67] Grimalt et al. (2010) was defined method for the quantification and confirmation capabilities of UPLC coupled with triple quadrupole and hybrid quadrupole time of flight mass spectrometry in pesticide residue analysis[68]. Apart from fruits and vegetable, Rong et al. (2017) was performed simultanious estimation of three pesticides and their metabolites in unprocessed foods using UPLC-MS/MS.[62]

5.3. Other detection methods

Techniques such as Capillary Electrophoresis and ELISA (Enzyme linked immunosorbent assay) are known as fast and cost effective separation and detection methods for pesticide analysis. ELISA provides highly sensitive detection of pesticides. ELISA technique is based on the interaction of antigen-antibody.[69] The only drawback of this method is un-stability of antibodies and un-sufficient blocking of immobilized antigen which creates false positive results. Yang et al. (2008) applied Enzyme linked immuno sorbent assay for detection of carbofuran pesticide and its metabolites from wide range of matrix using synthesized haptens 4-[(2,3-dihydro-2,2-dimethyl-7-benzofuranyloxy)carbonyl-amino] butanoic acid (BFNB) and 6-[(2,3-dihydro-2,2-dimethyl-7-benzofuranyloxy)-carbonylamino] hexanoic acid (BFNH) to form compex technique a conjugate-coated direct competitive ELISA method.[70] The recoveries was ranged from 104.6 %, 108.3 %, 106.3 % upto 100.1 %. Navarro et al. (2013) was employed duplex ELISA for estimation of organophosphate pesticides (chlorpyrifos and fenthion) and their residues in tangerine juice samples.[71] The developed method was employed by merging of two separate ELISAs for the respective organophosphate pesticide into one ELISA test. The method achieved a detection limit of 0.20 ± 0.04 μg L^{-1} (chlorpyrifos) and 0.50 ± 0.06 μg L^{-1} (fenthion). The recoveries obtained were of 95 % to 106 %.

Capillary electrophoresis is important technique, it requires less amount of reagent and sample and has high efficiancy of separation. Li et al. (2017) was established method using capillary electrophoresis coupled with biomimetic immunoassay (BI-CE) for estimation of trichlorfon and its residues from vegetable matrix.[72] The Limit of detection (LOD) for this is ranging from 0.16 to 0.13 μg L^{-1} with recovery rate between 78.8 to 103 % for trichlorfon from cucumber and kidney bean samples. Advantage of this technique is efficiany CE and specificity of BI. Daniel et al. (2015) was used to study CE method joint with tandem mass spectroscopy for the detection of prevalence of halosulfuron-methyl and its residue in sample of tomato and sugarcane juice.[73] The following developed method have limit of detection upto 2 ppm. The results obtained from analysis indicate that the MS detection system is the superior detection system (high sensitivity), and have the only disadvantage is high sampling cost.

5.4. Advanced detection methods

In past pesticides are analyzed using classic analytical methods like gas and liquid chromatography which has high sensitivity at low detection limits. However, these methods have limited drawbacks like being laborious, and needs costly instruments along with complications. Hence, an advanced approach for pesticide analysis are reported using sensor-based technique, which have numerous advantages including cost effectiveness, simplicity, less time consumption, high sensitivity and selectivity on-site detection.[74] Biosensors such as optical, electrochemical, piezoelectric and molecular imprinted polymer (MIP) are commonly used as detection methods in biosensors. Caetano and Machado (2008) was defined biosensing method for the detection of carbaryl pesticides from tomatoes.[75] This technique followed by the inhibition of acetylcholinesterases (AchE) activity. Limit of detection (LOD) was ranges between 2.0 x 10-6 mol/L.

Nanomaterial are being widely used for the detection of pesticides, recently.[76] The method involves the use of graphene and gold nanoparticle for the detection of organophosphate pesticides and carbamates. The detection of pesticide is based on the immobilization of Acetylcholineeserase through adsorbtion method with LODs found of 4.14 pg mL^{-1} for organophosphates and 1.15 pg mL^{-1} for carbamates. Additionally one more method was reported, ecomposes molecular imprinted polymers (MIPs) which is based on the working of the biological receptor and it has wide application for selectively sensing material and in identifying analytes of high molecular weight.[77] Zheng et al. (2015) was developed a lab-on-paper device having MIP with chemiluminescence detection.[78] The designed MIP has successfully detected dichlorvos with excellent selectivity, and found the limit of detection (LOD) upto 0.8 ng mL^{-1}.

The detection of pesticides in food comodity have been utilising nanotechnology based methods to invent fast and straight forward techniques.

The detection of pesticides in food comodity have been utilising nanotechnology based methods to invent fast and straight forward techniques.[79] A calorimetric study was conducted by Kim et al. (2018) for the detection of pesticides in eatables by inhibition of acetylcholinesterase and indoxyl acetate.[80] Yet the usage of nanomaterials like metals and metal oxide nanoparticles in the determination of pesticides have shown major concerns due to its toxicity.[81] Other than the advantages provided by these detection methods, the major drawback associated to it is the limited number of pesticides detected, making the method vulnerable.

5.5. Comparison of conventional extraction and detection approaches

In previous literatures numerous extraction methods and clean-up procedures like Liquid-liquid extraction (LLE), Gel permeation chromatography (GPC), Solid phase extraction (SPE), Solid phase micro-extraction (SPME) and Matrix solid phase extraction (MSPD) have been developed and used for the extraction and cleanup of various analytes to produce highly sensitive methods by reducing the effect of matrix. Even though many methods produces good recovery rate, even they do have some drawbacks such as time consumption, not easy to accessible and effect of matrix. Later, the QuEChERS method was emerged that is used for the multi-residue analysis because of its significant extraction efficiency.[82,83] This technique is found to be cost effective and can couple with instruments such as GC and LC due to its simplicity. In comparison to the conventional method, due to recent advances in QuEChERS method it is found effective for the cleanup procedure by using multi-walled carbon nanotubes.[84] Consequently, the conventional method requires PSA material for its cleanup, which leads to absorb acidic analytes and to degrade base-sensitive compounds.[85] To resolve this issues new method, was developed which involves the use of buffers like 1% acetic acid are known as buffered QuEChERS method.[86]

Apart from the extraction process, detection of pesticides at lower limits plays a major part in the pesticide residue analysis. In Initial days, Liquid chromatographic methods was rarely used with UV, DAD and fluorescence detectors because of its low sensitivity and selectivity. After the introduction of Mass detectors, there has been an increase in the usage of the LC systems because of its sensitivity. Electron spray ionization was found to be the more powerful analytical tool for the detection of pesticide and its residues in food matrix. From the previous literature it was concluded that the detection of pesticide based on UHPLC coupled with tendem MS (QqQ) method has been increased due to its higher sensitivity and selectivity for the detection of

pesticide residues in food matrix.

Now a days gas chromatographic techniques was restricted for the quantitative analysis because decrease in use of volatile and thermally stable compound unlikely to liquid chromatographic technique. So that the use of liquid chromatographic technique is increased for the detection of pesticides.

6. Conclusions and Future Perspectives

The increase in importance of food safety have brought a tremendous improvement in the residue analysis of pesticides in environmental and food matrix. The development of various detection techniques and pre-treatment has reduced the time, interference of matrix and sample size during the analysis. Nevertheless, the preferred extraction and detection system continues to be the same. It is coupled with mass detectors for quantification. But these methods have proved to be expensive and time consuming. Because of these limitations needs to be fixed in the near future and eco friendly and favourable cost effective methods are need to be developed which could identify larger number of pesticides in a single step for lower limit of the maximal residual levels.That's why, biosensors, nanotechnology and molecular imprinted polymer are the suggested as advanced methods for the detection of pesticides.

7. Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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Author biography

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