UDC 632

A REVIEW OF RAPID PESTICIDE RESIDUES DETERMINATION IN VEGETABLES AND FRUITS

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With the increasing demand of production, pesticides have been widely used in fruit and vegetable yield. Pesticides are used to kill insects, fungi and other organisms that harm the growth of crops in order to ensure and promote the growth of crops. In particular, pesticides are used to control diseases and insects and regulate plant growth and weeding. From the point at this stage, the use of pesticides in agricultural production is inevitable, and the corresponding, also in rapid increase in the amount of pesticide, pesticide residue problem is along with the production and extensive use of pesticides, pesticide, especially the organic pesticide used in great quantities, cause serious problems of pesticide pollution, a serious threat to human health. That is the abuse of pesticides does harm for environment and human health, particularly in the bioaccumulation effect of pesticide residues on human body, attracting more and more attention from scientists. Therefore, it's imperative to develop high sensitivity, high selectivity, simple, rapid and low-cost methods for pesticide residues detection and analysis. The traditional methods of pesticide residue analysis mainly include gas chromatography high performance liquid chromatography, chromatography-mass spectrometry, etc. These methods have been widely used in pesticide residue detection, and a series of important achievements have been made. Although with high detection sensitivity, these methods have some problems such as complicated sample pretreatment, expensive equipment, time-consuming analysis, and the need for specialized instrument operators and so on, which cannot meet the requirements of rapid and real-time field detection of pesticide residues. Therefore, researchers in various fields have carried out and strengthened the research on rapid detection technology of pesticide residues, seeking to develop convenient, sensitive, accurate and stable new pesticide residue detection technology. In this paper, we mainly reviewed the rapid detection technologies of pesticide in fresh fruits and vegetables in recent years, including new chromatographic analysis, enzyme inhibition, fluorescence sensor, spectrophotometric and biosensor detection technology, and analyzed the development status, advantages, and disadvantages of each method, as well as the development prospect of rapid detection technology in the future.

***Key words**: pesticide residues, rapid detection techniques, cross-fusion technology, fruits and vegetables.*
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Introduction. Modern agricultural production is inseparable from the usage of pesticides to prevent and control all kinds of crop diseases, insect pests and weeds, following by severe food safety problems due to pesticide residues (Caria et al., 2021; Loganathan & Murugan, 2017). There are many kinds of pesticide, including organophosphorus pesticides, organic nitrogen pesticides (carbamate, triazine and its derivatives). Among them, organophosphorus and organic nitrogen pesticides have occupied the vast majority of the market because of their short half-life in the environment, relatively low toxicity to mammals, wide range of application and low price. As is known to all, residual pesticides are toxic, which can cause various chronic or acute poisoning, leading to physiological diseases such as rashes, asthma, chronic diseases, and neurological diseases (Calaf et al., 2021; Freire & Koifman, 2012; Li et al., 2021; Steenland et al., 1994; Upadhayay et al., 2020; Yu et al., 2021). Therefore, to ensure the quality and safety of agricultural products, efficient and rapid pesticide residue detection methods are researched (Wu et al., 2021). Traditional detection methods include chromatography, chromatography-tandem mass spectrometry and high-performance liquid chromatography and so on (Golge, 2021). They can be the preferred detection methods in the formulation of national standards for pesticide detection in many countries because of the high repeatability and stable test results. However, these methods need large detection equipment and specific operating environment, which are not suitable for the practical production requirements for the rapid field test.

In recent decades, a variety of low time-consuming, convenient and rapid detection methods developed, including but not limited to new chromatographic analysis, enzyme inhibition, fluorescence sensor, spectrophotometric and biosensor detection technology (Ninga et al., 2021; Rojas et al., 2021; Saegusa et al., 2021). What's more, these technologies have made great breakthroughs on the basis of each one, and various rapid detection technologies tend to be more and more cross-fusion, mutual penetration and advantages superposition (Hao & Wang, 2016). With the rapid development of nanomaterials, multi-cross rapid detection technologies based on nanomaterials have a great breakthrough in sensor technology improvement, and biosensor technology has a huge development advantage in rapid detection technology (He et al., 2019; Lei et al., 2018; Lu et al., 2018; Wu et al. 2017).

Current rapid detection technology of pesticides

1. Chromatographic detection techniques

Chromatography, which is highly sensitive and mature, mainly include gas chromatography, gas chromatography-mass spectrometry, high performance liquid chromatography and other technologies (Hao et al, 2010; Tong et al., 2014; Wu et al., 2009). Although their disadvantage like expensive equipment requirement, high technical personnel, complex pretreatment and testing time, cross-fusion of rapid detection technologies make these methods showing great potential in the market for rapid detection recently. Khan (Khan et al., 2018) proposed a pressurized liquid extraction by ethyl acetate based method for simultaneous analysis of different pesticide residues in tuber crops, and then selectively identified and quantified the residuals by GC-MS selected reaction monitoring. They got the limits of quantification with 0.1–10 ng/g, and recovery rate from 70 % to 120 %.

Chromatographic detection technique is mainly used in laboratory precision detection. This technology shows high selectivity for organophosphorus pesticides, but its scope of action is relatively limited. The current research directions are mostly focused on improving pretreatment technology, enrichment methods and extraction methods. In other aspects, the method for rapid detection in the market needs to be further improved.

2. Enzyme inhibition detection techniques

Enzyme inhibition rapid detection method is based on the inhibitory effect of pesticide residues in food on enzyme. This technology has the advantages of simple and quick operation and simple pretreatment, and a variety of simple instruments have been developed for rapid detection in the market currently. However, this method has great limitations, and with poor stability due to many factors to be controlled (Gumpu et al., 2017; Li et al., 2019). So, there are a large room for improvement in sensitivity and accuracy. Through the effective combination with the biosensor technology, the sensitivity and accuracy of the enzyme inhibition technology have been greatly improved. After the fusion, the enzyme inhibition method with the biosensor technology is more suitable for rapid detection (Badawy, 2021; Singh et al., 2020). The rapid detection principle of enzyme inhibition method is relatively simple. By organophosphorus pesticides inhibiting the activity of acetylcholinesterase, the catalytic process can produce less H_2O_2, and the oxidation ability can be reduced, resulting in visible discoloration reaction of the substrate (Albendin et al., 2021; Lin et al., 2021). The intensity of colorimetric signal is an important factor in the research of enzyme inhibition method to realize the real visual detection. Yang (Yang et al., 2019) proposed an enzyme inhibition method to detect the pesticide residues of the milk. He established a system to study the inhibitory reactions of organic phosphorus and aminoformate residues in milk. The analysis of color reactions of milk showed a good correlation between color intensity and content of tolclofos-methyl, methamidophos and isoprocarb 1-naphthalenyl methyl carbamate, and the detection range of four kinds of pesticides is 0.5 ~ 1.0 mg/kg.

By combining with the biological sensing technology, the application scope of the enzyme inhibition method is expanded, and the enzyme sensitivity is enhanced. Enzyme inhibition sensor is one of the most widely used rapid detection technique in the current rapid detection market, but there are still many problems with its own (Wu et al., 2019). Recently, many researchers study the selective purification of enzyme, effective oxidation pretreatment, colorimetric signal enhancement and false positives elimination. The sensitivity of enzyme inhibition method is influenced by the purity of the enzyme, the concentration of substrate and environmental factors, etc., and the stability and sensitivity of the enzyme suppression method are need to be higher (Arduini et al., 2019; Pundir et al., 2019; Sgobbi & Machado, 2018).

3. Fluorescence detection techniques

Fluorescence detection method is based on the different material molecules, the different absorption and reaction of light wavelength. This technology has high sensitivity, but it is limited to the luminous pesticide, and the non-luminous pesticide still needs to be added with fluorescent agent, and is susceptible to the interference of external factors, with poor adaptability (Ouyang et al., 2021; Wang et al., 2021). In recent years, through the fusion of biosensors, this detection technology has also made great progress (Chen et al, 2021; Han et al., 2021; Liang et al., 2021; Lin et al., 2021). The fluorescence sensor has the advantages of simple operation, quick response, high sensitivity and good reproducibility. The fluorescence sensor consists of two parts: the fluorescence signal element and the recognition element. Enzymes, antibodies, aptamers and molecularly imprinted polymers (MIP) are combined with nanomaterials to further enrich the types of fluorescence sensors (Zhou et al., 2018). Carbon Quantum Dots (CQDs) have been proposed as the photo-sensitizer for this purpose, however the optical properties of pure CQDs restrict the detection limit of such an approach. Doping is an effective strategy to introduce novel electronic structure into the CQDs to solve this problem. using ionic liquids as a single source, H. Li (Li et al., 2016) proposed a novel N and S co-doped CQDs by a simple ultrasonic method. The doping in the structure introduces localized states which can trap photo-excited electrons and enhance their PL lifetime. These quantum dots are successfully used as the basis of a simple, efficient sensor for ultrasensitive pesticide detection (Limit of Detection = 5 ppb). J. Hou (Hou et al., 2015) used tyrosinase to catalyze the oxidation of tyrosine methyl ester on the surface of carbon dots to corresponding quinone products, which can quench the fluorescence of carbon dots, and the enzyme inhibition rate is proportional to the logarithm of the methyl parathion concentration in the range 1.0×10−10–1.0×10−4 M with the detection limit (S/N = 3) of 4.8×10−11 M.

The combination of fluorescence detection method and biosensor method has greatly promoted the rapid detection of pesticide residues (Hou et al., 2015; Long et al, 2015; Meng et al., 2013; Upadhyayula, 2012). Q. Luo (Luo et al., 2018) proposed a simple method for the preparation of highly selective and sensitive fluorescent probes based on Rhodamine B (RB) modified silver/gold bimetal nanoparticles (RB-Ag/Au NPs). Because that the coordination ability of Ag/Au NPs and organophosphorus pesticides (Ops) is stronger than that of Ag/Au NPs and RB, RB will be displaced from the Ag/Au NPs surface, accompanied by the fluorescence recovery of RB. It can be applied to the determination of OPs in real fruit and water samples with the limit of detection (LOD) as low as 0.0018 ng/mL.

4. Spectrophotometric colorimetry techniques

Colorimetry determines the content of components to be measured by measuring the color depth of colored substance solution (Kostelnik & Pohanka, 2018; Liu et al., 2012). This method has high sensitivity and selectivity, and the reaction product is stable. In recent years, more and more researchers have combined spectrophotometric colorimetry with new sensors, and the newly emerging sensing materials have greatly improved the detection sensitivity of spectrophotometric technology. A. Kodir (Kodir et al., 2016) developed a novel pesticide colorimetric sensor based on L-cysteine-modified silver nanoparticles (L-cys-AgNPs). By reducing the silver nitrate solution in the presence of Diospyros blancoi leaf infusion, and then mixing with the l-cysteine solution, the colorimetric sensor was prepared. In the presence of cypermethrin, the color of l-cys-agnps was obvious, and the peak absorbance decreased from 1.15 to 0.17.

The optical colorimetric sensor synthesized from gold nanoparticles has high sensitivity (Li, et al., 2018). Moreover, the gold nanoparticles are stable, and the reaction with pesticides can make the gold nanoparticles aggregate and produce visible color changes (Bettazzi et al., 2021; Hua et al., 2021; Ma et al., 2021; Vilian et al., 2021; Wang et al., 2021). Using this principle, Bala (Bala et al., 2016) built a colorimetric apparatus based on gold nanoparticles to measure the phosphorous in a mixture. The results showed that the linear relationship was good within the concentration range of the uv-vis wavelength from 0.01 nm to 1.3 mm, and the detection limit was 1.3 mm, indicating a high sensitivity. Recently, biosensors based on nanomaterials have developed rapidly in pesticide detection, and more and more new nanomaterials have been used to prepare electrochemical biosensors. By Introduction of nanomaterials It greatly promoted the development of the biosensor technology, and with the progress of material science, all kinds of polymer and nano materials combine to form nanocomposites are also solved the traditional biological sensing technology stability and sensitivity is not high question, nanometer materials to make biological sensing technology has entered a new period of development.

5. Biosensor techniques

Biosensor techniques generally use enzymes, antigens, antibodies, cells and other active sensitive materials as recognition elements (Silva et al., 2020; Tang et al., 2020). The change in concentration will be converted into electrical signals after recognition and then displayed and recorded by amplification. Recently, biosensors based on nanomaterials have developed rapidly in pesticide detection, and more and more new nanomaterials have been used to prepare electrochemical biosensors, which greatly promoted the development of the biosensor technology (Akdag et al., 2020; Ayat et al., 2021; Chouichit et al., 2020; Jain et al., 2021; Lah et al., 2021). Although the stability and sensitivity of traditional biosensors technology is not high, all kinds of polymer and nanomaterials combine to form nanocomposites have solved these questions.

The electrochemical biosensor based on the inhibition of acetylcholinesterase is a promising method for the detection of organophosphorus. The irreversible oxidation peak of the active product thiocholine is an important marker for the detection of organophosphorus (Alex & Mukherjee, 2021; Cao et al., 2020; Caratelli et al., 2020; Davletshina et al., 2020; Silva et al., 2020; Singh et al., 2020). Different from traditional organophosphorus detection methods, this method does not need expensive experimental equipment and well-trained technicians, and the detection cost is low and efficient. To improve sensor sensitivity and reduce detection limits, the researchers used different nanomaterials in the sensor, such as Au nanoparticles (Li & He, 2021; Lipinska et al., 2021; Rashed et al., 2021; Yang et al., 2021), carbon nanotubes (Kathiresan et al., 2021; Li et al., 2021; Qian et al. 2021; Rashid et al., 2021; Siew et al. 2021), graphene (Gan et al., 2021; Rashid et al., 2021; Siew et al., 2021; Sun et al., 2021; Zhou et al., 2021) and magnetic nanomaterials (Da Silva & Brett, 2020; Lu et al., 2020; Shen et al., 2021). The large specific surface area and easy modification characteristics of nanomaterials provide more active sites on the electrode surface, which is more conducive to full contact with the reactants, thus providing detectable electrical signals. Zhao (Zhao et al., 2015) constructed an ultra-sensitive current sensor by using Au nanoparticles (AuNPs)-β-cyclodextrin (β-CD) and Prussian blue-chitosan (PB-CS) and acetylcholinesterase(AChE), and realized the high sensitivity detection of malathion and carbaryl through the synergic action of multiple components, with detection limit as low as 4.14 pg/mL and 1.15 pg/mL, respectively. By cross-linking acetylcholinesterase onto the IL-GR/Co3O4 / CHI electrode constructed from ionic liquid modified graphene (IL-GR) and Co3O4 nanoparticles, Y. Zheng (Zheng et al., 2016) was able to effectively reduce the loss of enzyme activity and improve the detection sensitivity. A linear relationship between the inhibition percentage (I%) and logarithm of the concentration of dimethoate was found in the range from 5.0 × 10−12 to 1.0 × 10−7 M, with a detection limit of 1.0 × 10−13 M (S/N = 3).

In order to further enhance the stability of the biosensors, a nanocomposite material which can significantly enhance the mechanical strength of each component is formed by introducing a polymer into the nanometer material (Bagheri et al., 2017; Cinti et al., 2016; Guler et al., 2017; Huang et al., 2010; Huo et al., 2014; Jeyapragasam & Saraswathi, 2014; Wei & Wang, 2015; Zheng et al., 2015). New biosensors have developed rapidly, and the stability and sensitivity of all kinds of biosensors have been greatly improved, but they are only used for single pesticide and the detection range still is very small. So, they can't be widely used for the rapid detection of a variety of organophosphorus pesticides on the market. The development of nanometer materials made great progress for biological sensor technology in sensitivity and stability, which has significantly outpaced the development of other rapid detection technologies (Jiang et al., 2020; Wang et al., 2016). Therefore, the cross-fusion detection methods combining with biosensor and other rapid detection technologies retain the development advantages, and overcome many limiting factors in the rapid detection technology, making the rapid detection technology develop rapidly and become perfect.

Conclusions. In recent years, with the improvement of market requirements for the rapid detection technology of pesticides, organophosphorus pesticides, as an important part of the pesticide market, whose development speed of the rapid detection technology is very rapid. There are a wide variety of traditional detection technologies for pesticide, and each of them have own pros and cons, with development difficulties (Chen et al., 2021; C. J. Li et al., 2021; J. J. Li et al., 2021; Liang et al., 2021; Lin et al., 2021; Teysseire et al., 2021). At present, the rapid detection methods in the pesticide market tend to be more and more cross-fusion with various detection technologies. With the progress of science and technology, the development of new nanomaterials also makes great contributions to the improvement of rapid detection technology. Especially for the biosensor technology, who highly require for new material, the development of nanomaterials directly promotes the progress of this technology (Burratti et al., 2021; Du et al., 2021; M. Li et al., 2021; Ren et al., 2021; X. Y. Zhou et al., 2021). As the cross-fusion of a variety of rapid detection technologies, biosensor technology shows strong combination, and is suitable for a variety of rapid detection method of combining. Through the combination of biosensor detection technology and other rapid detection technologies, many difficulties in the development of rapid detection technology have been overcome. The advantages of rapid detection technology, such as enzyme inhibition detection technology, fluorescence detection and spectrophotometric detection technology, have been amplified, therefore the rapid detection techniques become more extensive and faster (Badawy, 2021; Cao et al., 2020; Singh et al., 2020; Q. S. Wei et al., 2020; N. Yang et al., 2020).

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ДОСЛІДЖЕННЯ ПРОЦЕСУ ШВИДКОГО ВИЗНАЧЕННЯ ЗАЛИШКІВ ПЕСТИЦИДІВ В ОВОЧАХ ТА ФРУКТАХ

Зі збільшенням попиту на виробництво сільськогосподарської продукції, збільшується використання пестицидів, які на сьогоднішній день забезпечують збереження врожаю фруктів та овочів. Пестициди використовують для контролю чисельності шкідливих організмів, забезпечуючи тим самим оптимальні умови для росту та розвитку сільськогосподарських культур. Сучасне виробництво сільськогосподарської продукції неможливе без застосування пестицидів. Але зловживання під час використання пестицидів завдає шкоди навколишньому середовищу та здоров'ю людей, особливо внаслідок *біоакумуляції пестицидних речовин в організмі людини. Тому питання створення високочутливих, селективних, простих, швидких та недорогих методів виявлення та аналізу залишків пестицидів є актуальним. Традиційні методи аналізу залишків пестицидів ґрунтуються на різних видах хроматографії (газова, рідинна), мас-спектрометрії тощо. Ці методи широко застосовують для виявлення залишків пестицидів. І хоча вони мають високу точність, поряд з цим є ряд недоліків: складна попередня підготовка зразка до аналізу, висока вартість обладнання, трудомісткий аналіз та потреба у спеціалізованих операторах приладів. Тому науковці у різних областях проводять дослідження технології швидкого виявлення залишків пестицидів. У цій роботі ми розглядаємо технології виявлення пестицидів у свіжих фруктах та овочах за останні роки. Розглядаються такі методи, як хроматографічний аналіз, інгібування ферментів, флуоресцентні датчики, спектрофотометричний та біосенсорний методи. Проаналізовано стан їх розвитку, переваги та недоліки кожного методу, а також перспективи розвитку технології швидкого виявлення залишків пестицидів у майбутньому.*

Ключові слова: *залишки пестицидів; методи швидкого виявлення; технологія перехресного синтезу; фрукти та овочі.*

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