

Advances in Electrochemical and Optical Methods for the Detection of Pesticide Residues: A Comprehensive Review

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Review

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Abstract: The development of pesticides has been instrumental in improving agricultural productivity by effectively controlling pests and diseases. However, the widespread use of pesticides has also raised significant environmental and health concerns. To address these issues, various electronic and optical analytical detectors have been developed to monitor pesticide residues in the environment. In the realm of electronic detectors, advancements in impedance sensors and Field-Effect Transistor (FET) sensors have demonstrated remarkable stability, sensitivity, and specificity for detecting specific pesticides. Additionally, traditional electronic sensors have seen improvements in electrode materials, surface modifications, and integration with microfluidic systems. For optical detection, fluorescence spectroscopy and ultraviolet/visible (UV/Vis) spectroscopy remain widely utilized due to their versatility and accessibility. Meanwhile, emerging techniques such as surface plasmon resonance (SPR) and surface-enhanced Raman scattering (SERS) offer detailed molecular composition analysis and high sensitivity, making them promising tools for pesticide detection. This review provides a comprehensive overview of advancements in analytical detection of pesticide residues over the past decade, highlighting the strengths and limitations of various detection technologies.

Keywords: pesticide residues; analytical detection; electronic sensors; optical sensors

1. Introduction

The development of pesticides has played a crucial role in enhancing agricultural productivity by effectively controlling pests and diseases. However, their widespread use has led to significant environmental and health concerns. Recent studies have linked pesticide exposure to a range of health issues, including neurological disorders and various types of cancer, imposing a considerable socio-economic burden. The economic impact of pesticide-related healthcare and environmental remediation amounts to billions of dollars annually.

Efforts to develop efficient pesticide detection technologies date back to the early 20th century, with initial methods primarily based on basic chemical assays. Over time, the need for more sensitive, accurate and rapid detection methods to ensure food safety and environmental protection has spurred technological advancements. In recent decades, the emergence of electrochemical and optical sensors has revolutionised this field. These modern sensors provide enhanced sensitivity and specificity, capable of detecting even trace levels of pesticide residues.

This review explores the progress in analytical technologies for pesticide residue detection, with a particular focus on recent advancements in electrochemical and optical sensors.

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2. Overview: Rapid Detection Technology Based on Electro-chemistry

Since 2014, electrochemical sensors have revolutionised the analytical detection of pesticide residues, meeting the demand for rapid, sensitive and cost-effective methods. Significant advancements in electrode materials, surface modifications and integration with microfluidic systems have enhanced the sensitivity and real-time detection capabilities of traditional electrochemical sensors. These sensors operate by measuring current or voltage changes induced by analyte interactions, offering reliable data for effective pesticide monitoring.

Impedance sensors have emerged as a key technology, leveraging changes in electrical impedance caused by pesticide interactions on the sensor surface. This approach provides high sensitivity and selectivity, essential for real-time monitoring. Advances in nanomaterials and sensor miniaturisation have further optimised these sensors, making them well-suited for portable, field-based applications.

Another notable innovation is the field-effect transistor (FET) sensor, which detects alterations in electrical properties resulting from pesticide interactions with semiconductor materials like graphene and carbon nanotubes. FET sensors are highly sensitive, offer rapid response times and can be miniaturised, positioning them as an ideal choice for onsite testing scenarios.

Recent research has explored the application of cutting-edge techniques in electrochemical sensors. For instance, non-enzymatic inhibition methods using electro-active probes such as pralidoxime (PAM) have shown considerable potential for detecting organophosphate pesticides (OPPs). Additionally, metal-organic frameworks (MOFs) and their composites with metals, metal oxides and carbon nanotubes have been extensively utilised to improve the sensitivity and specificity of impedance sensors. Similarly, advancements in FET sensors have capitalised on novel nanomaterials, resulting in lower detection limits and faster response times.

These developments in electrochemical sensor technology underscore the ongoing effort to create efficient, reliable and economical solutions for pesticide residue detection. By addressing challenges in sensitivity, specificity, and portability, these sensors play a vital role in ensuring food safety and environmental protection. As technology continues to evolve, electrochemical sensors are poised to become indispensable tools in analytical chemistry.

2.1. Traditional Electrochemical Sensors

2.1.1. Introduction

Traditional electrochemical sensors have long been a cornerstone in the analytical detection of pesticide residues due to their simplicity, sensitivity and cost-effectiveness. These sensors operate by measuring current or voltage changes resulting from analyte interactions with an electrode. Specifically, receptor-analyte interactions induce redox reactions at the electrode surface, causing potential changes. By monitoring the variations in current or voltage during the oxidation or reduction of the analyte, these sensors enable sensitive and rapid detection of pesticide residues.

Advancements in electrode materials, surface modifications and integration with microfluidic systems over the past decade have significantly enhanced the performance and application scope of traditional electrochemical sensors. These improvements have made such sensors indispensable for detecting a wide variety of pesticides, offering rapid and accurate measurements. This section explores key developments and applications of traditional electrochemical sensors in pesticide residue detection from 2014 to 2024.

2.1.2. Application Example

Organophosphate pesticides (OPPs), widely used for pest control, are highly toxic and pose severe risks to the human nervous system. Their excessive application often leads to environmental bioaccumulation, causing prolonged harm to terrestrial and

aquatic ecosystems. Despite stringent regulations, OPP usage remains widespread in developing countries such as India, Pakistan, Bangladesh and Algeria, with Pakistan reporting an annual increase of over 25% in pesticide consumption. To address food safety and environmental concerns, effective and economical monitoring protocols are essential.

Conventional detection methods, such as gas chromatography (GC), high-performance liquid chromatography (HPLC), fluorimetry and competitive immunoassays, though accurate, are limited by high costs and complex procedures. In contrast, electrochemical approaches are gaining popularity for their simplicity, rapidity, cost-effectiveness and sensitivity. For instance, graphene quantum dots (GQDs) have demonstrated exceptional efficacy in quantifying fenthion pesticides by enhancing signal sensitivity. However, modified electrodes often face challenges related to signal fluctuations due to uneven nanostructure coverage, which compromises reproducibility.

Copper oxide (CuO), known for its excellent catalytic and electronic properties, has emerged as a promising alternative electrode material. CuO nanomaterials are easily synthesised at low temperatures and can be functionalised with specific chemical moieties, improving the electrode-solution interface chemistry. The morphology of nanostructures plays a pivotal role in determining sensor response, making high structural reproducibility essential. To address this, templates as growth control agents ensure consistent signals and enable the development of unique nanostructures with superior electrochemical characteristics.

A notable example is the development of a novel sensor utilising in-situ grown CuO nanostructures on indium tin oxide (ITO) substrates. This sensor exhibited outstanding sensitivity and reproducibility for detecting pesticides such as chlorpyrifos, fenthion and methyl parathion. The ITO-based electrode demonstrated strong resistance to interference from co-existing chemicals, highlighting its robustness. Additionally, the effective recovery of pesticides from real-world samples, such as cabbage and spinach, further underscores its practical applicability [37].

2.1.3. Summary of Recent Advances:

Chlorpyrifos, fenthion and methyl parathion detection have seen notable advancements through the development of nano-scale electrodes by Tunesi et al., which demonstrated excellent inhibition signal sensitivity and repeatability [37]. Additionally, Prabhu et al. introduced electro-sensing for 2,4-dichlorophenol (2,4-DCP), showcasing remarkable electro-catalytic behaviour and significantly elevated peak currents [38]. For atrazine detection, Zacco et al. developed an electrochemical magnetoimmunosensing approach, providing a rapid, simple, cost-effective and portable method for on-site analysis [39]. Additionally, Ren et al. combined microchip electrophoresis with amperometry for the detection of m-cresol and α -naphthol, achieving a broad linear range and high reliability [40]. Costa et al. introduced a voltammetric sensor for carbamate pesticides, offering superior sensitivity compared to conventional methods [41].

For carbaryl, Wang et al. developed a micro-machined thin-film electro-acoustic biosensor, characterised by its simplicity and portability, making it suitable for on-site food safety testing [42]. Cioffi et al. utilised office paper-based electrochemical strips for organophosphate detection, emphasising the practicality and accessibility of common substrates [43]. Wu et al. employed nanocomposites of reduced graphene oxide and Nafion for high-precision analysis of real samples [44]. Both Khairy et al. and Huixiang et al. focused on non-enzymatic electrochemical detection methods for organophosphates, achieving excellent reproducibility, stability and sensitivity without compromising performance [45, 46].

Facure et al. designed an innovative electronic tongue using graphene hybrid nanocomposites, offering a swift, simple, and cost-effective solution for organophosphate detection [47]. Li et al. developed a capacitive detection method for methyl-parathion using electropolymerized molecularly imprinted polymers, enabling fast, sensitive and realtime analysis [48]. Chen et al. created an electro-acoustic enzyme biosensor for organophosphorus compounds, notable for its small size, ease of operation and compatibility with integrated circuits [49].

These advancements illustrate the diverse and innovative strategies being applied to traditional electrochemical sensors. The significant improvements in sensitivity, cost-effectiveness and real-time detection capabilities demonstrate the critical role of electrochemical technologies in advancing pesticide residue analysis.

2.2. Impedance Sensors

2.2.1. Introduction

Impedance sensors have gained increasing significance in the detection of pesticide residues due to their ability to measure changes in electrical impedance caused by analyte interactions on the sensor surface. These sensors operate by detecting target molecules through variations in electrical impedance when specific receptors bind to the target analytes. Such interactions induce changes in potential difference, which can be monitored to determine both the presence and concentration of the analytes. By leveraging advanced electrode materials and receptor modifications, impedance sensors achieve accurate and efficient detection for a range of applications.

Renowned for their high sensitivity and selectivity, impedance sensors are particularly well-suited for real-time pesticide monitoring. They can detect subtle changes in resistance or capacitance, providing precise data regarding the presence and concentration of pesticide residues. Over the past decade, significant advancements in nanomaterials, sensor miniaturisation, and functionalisation techniques have greatly expanded the performance and application potential of impedance sensors. These innovations have enabled the development of highly sensitive, portable and robust systems tailored for fieldbased and laboratory analyses.

This section delves into the major advancements and applications of impedance sensors in the analytical detection of pesticide residues since 2014, highlighting their critical role in ensuring food safety and environmental protection.

2.2.2. Example Application of Impedance Sensors in Pesticide Residue Detection:

Organophosphate pesticides (OPPs), widely employed in agriculture, pose substantial risks to humans, animals, plants and soil due to their high toxicity and environmental persistence. Common OPPs, such as malathion, glyphosate, monocrotophos, methyl parathion, chlorpyrifos and diazinon, are often applied directly to crops, leading to potential bioaccumulation. These pesticides inhibit human acetylcholinesterase (AChE) activity, highlighting the critical need for real-time monitoring to mitigate exposure and health risks.

In this study, a solvothermal method was employed to synthesise a Zr-MOF/ZrO2/MWCNT ternary composite for use in impedance sensors. These sensors detect changes in electrical impedance at the electrode surface when receptors bind to target analytes, leading to variations in potential difference. By monitoring these changes, the sensors can effectively identify and quantify the presence of pesticide residues.

Within the composite, ZrO₂ enhanced the number of active sites and increased the binding capacity of the sensor, enabling more effective interactions with target analytes. Multi-walled carbon nanotubes (MWCNTs) played a vital role in improving the sensor's conductivity, stability and surface area, significantly boosting the overall performance of the electrode. This integration of materials allowed for the development of a highly efficient sensor capable of detecting OPPs through impedance analysis.

The sensor demonstrated exceptional selectivity, stability and reproducibility in detecting OPPs. When target analytes interacted with receptors on the electrode surface, the resulting impedance changes were directly proportional to their concentration, enabling

precise quantification of pesticide residues. This high sensitivity to impedance variations underscored the sensor's effectiveness in pesticide detection.

To validate its practical applicability, the sensor system was tested on real samples, including apples, cabbage and soil. These analyses confirmed the sensor's reliability and efficiency, showcasing its potential for real-world applications in environmental monitoring and food safety. The successful detection of OPPs across diverse sample types highlighted the robustness and versatility of the system, making it a valuable tool for ensuring the safety and quality of agricultural products [26].

2.2.3. Summary of Recent Advances

The field of impedance sensors for pesticide residue detection has made significant strides over recent years, showcasing advancements in sensitivity, selectivity and practicality.

Hromadová et al. investigated the detection of atrazine and terbutylazine using double-layer capacitance measurements, identifying the critical temperature for surface film transitions [21]. Ding et al. demonstrated that their impedance-based method for detecting chlorpyrifos correlated well with traditional analytical approaches, indicating consistency and reliability [22].

Madianos et al. developed highly sensitive and selective impedance sensors targeting acetamiprid and atrazine, enhancing detection capabilities for these pesticides [23]. Org et al. introduced a microfluidic impedance immunosensor for chlorpyrifos, offering an extended detection range, improved reproducibility, enhanced stability and lower detection limits [24]. Yang et al. innovated a paper-based microfluidic chip impedance sensor for organophosphorus pesticides, employing time-sequence spectral data to build classification models for enzyme inhibition analysis [25].

Gokila et al. focused on non-enzymatic electrochemical impedance sensors for pesticides such as malathion, chlorpyrifos, dimethoate, monocrotophos and glyphosate. These sensors exhibited excellent chemical stability over 100 cycles, good repeatability and an extended shelf life [26]. Similarly, Ruankham et al.'s impedance sensor for chlorpyrifos showcased outstanding selectivity and reproducibility [27].

Malvano et al. developed a label-free impedimetric affinity sensor for carbaryl and dichlorvos, notable for its rapid response times [28]. Zhao et al. introduced a cost-effective, high-sensitivity microelectrode impedance immunosensor for carbofuran, enabling swift detection [29]. Wei et al. designed surface acoustic wave impedance sensors for dichlorvos and dimethoate, correlating enzyme activity inhibition with pesticide concentrations to provide reliable measurements [31].

Elshafey and Radi presented an electrochemical impedance sensor for alachlor, optimised for in-field applications with a simple detection platform [32]. López Rodriguez et al. created a Streptomyces spore-based impedimetric biosensor for lindane, which was cost-effective and capable of detecting pesticide residues within two days without requiring sample pretreatment. The reusable electrode surfaces further highlighted its practicality [33].

Fan et al. developed an impedance spectroscopy-based aptasensor for acetamiprid, characterised by its high selectivity [34]. Ferreira et al. pushed the boundaries of sensitivity with nanoarchitecture-based impedance sensors for fenitrothion, showcasing the continuous evolution and enhancement of impedance sensors in pesticide residue detection [36].

2.3. FET Sensor (Field-Effect Transistor Sensor) in Pesticide Residue Detection

2.3.1. Introduction

Field-effect transistor (FET) sensors are advanced biosensors that detect changes in electrical properties resulting from interactions between target analytes and the sensor surface. These sensors operate by leveraging receptor-analyte interactions at the electrode surface, which induce changes in the electric field across the sensor's layered structure. The typical FET sensor structure includes a gate electrode, an insulating layer, and a semiconductor channel. When target molecules bind to receptors on the gate electrode, the resulting changes in the electric field modulate the potential difference across the channel. This modulation alters the current flow through the transistor, enabling highly sensitive detection of the analytes.

The sensitivity of FET sensors is significantly enhanced by the use of semiconductor materials like graphene and carbon nanotubes, which offer large surface areas and excellent electronic properties. Functionalising these materials with specific receptors - such as enzymes, antibodies or aptamers - allows for selective binding to target molecules, including pesticide residues. This specific interaction directly impacts the current flowing through the transistor, enabling precise and quantitative detection.

FET sensors are particularly valuable for real-time and on-site testing due to their high sensitivity, rapid response times and potential for miniaturisation. These features make them an ideal tool for use in food safety and environmental monitoring, where timely and accurate detection of pesticide residues is crucial.

2.3.2. Application Example

In 2024, Wang et al. developed an innovative field-effect transistor (FET)-based biosensor specifically designed to detect organophosphorus pesticides (OPs) in vegetables [6]. OPs are extensively used in agriculture for their cost-effectiveness and pest control efficiency but pose significant health risks by inhibiting acetylcholinesterase, potentially leading to severe symptoms and even fatality. Traditional detection methods, such as thinlayer chromatography (TLC), high-performance liquid chromatography (HPLC) and mass spectrometry (MS), while accurate, are expensive and time-consuming. These limitations have driven the demand for faster, more cost-effective detection technologies.

Wang et al. addressed this need by designing a bridge-type aptamer-functionalised Fe-Co/NPC-FET biosensor. This innovative sensor employs a two-dimensional nanoporous carbon material (Fe-Co/NPC) synthesised using an Fe-based zeolitic imidazolate framework (ZIF-67) template. The Fe-Co/NPC material, characterised by its high surface area, magnetic properties and excellent biocompatibility, significantly enhances the biosensor's performance.

The biosensor leverages the principles of FET sensors, utilising a multi-layer structure consisting of a gate electrode, an insulating layer and a semiconductor channel. When target analytes (e.g., OP residues) bind to receptors on the electrode surface they alter the electric field at the gate. These changes modulate the potential difference across the channel, leading to variations in current flow through the transistor. This mechanism enables highly sensitive and selective detection of OP residues.

Beyond its sensitivity and specificity, the biosensor demonstrates remarkable reusability, making it a cost-effective solution for continuous monitoring. The integration of advanced nanomaterials and the multi-layer structure significantly enhances its durability and performance. By employing aptamer-based receptors, the sensor achieves exceptional selectivity for OP residues, further improving accuracy and reliability.

Wang et al.'s research underscores the transformative potential of nanomaterials in advancing FET-based sensor technology. Their work highlights the role of these materials in developing next-generation biosensors that efficiently detect agricultural contaminants and monitor environmental pollutants. The successful application of this biosensor in real-world scenarios, such as ensuring vegetable safety, demonstrates its capability to revolutionise biosensing technologies. By addressing critical challenges in food safety and environmental science, this innovation offers a promising solution to pressing global issues [6].

2.3.3. Summary of Recent Advances

Over the past decade, significant progress has been made in the development of FET sensors for detecting pesticide residues. Sasipongpana et al. and Simonian et al. developed low-cost, field-applicable sensors and pH-sensitive FETs for OP detection using organophosphate hydrolase enzyme [1, 2]. Meanwhile, Islam et al. and Kumar et al. introduced highly stable, sensitive microfluidic-based FETs and Ag–ZnO–SWCNT-based FETs for selective detection [3, 4]. Researchers like Kumar et al. and Schöning et al. focused on creating CFO/s-SWCNT-based systems and early FET sensors for broad pesticide application [5, 7]. Work by Wang et al. and Ishii et al. highlighted reusable Fe-Co/NPC-FET sensors and carbon nanotube FETs with recombinant acetylcholinesterase [6, 8]. Innovations by Yu et al. and Zhang et al. leveraged photoelectrochemical-based extended-gate FETs and highly sensitive β-cyclodextrin modified perfluorinated copper phthalocyanine FETs [14, 15].

Additionally, Zhu and Cao developed graphene-based FETs for detecting various organophosphates and atrazine [16, 17]. Finally, Wang et al. and Bhatt et al. made advances with photoelectrochemical solution-gated graphene FETs and flexible, low-cost electrolyte-gated carbon nanotube FETs. These studies reflect significant advancements in material science, sensor design and application specificity, showing the evolution and diversification of FET sensors in pesticide residue detection [18, 19].

2.4. Fluorescence Spectroscopy and Ultraviolet/Visible Spectroscopy (UV/Vis)

2.4.1. Introduction

Fluorescence spectroscopy and ultraviolet/visible spectroscopy (UV/Vis) are critical techniques in the analytical detection of pesticide residues. These methods utilise the interaction of light with substances to provide comprehensive data about the chemical properties and concentrations of analytes. In recent years, significant advancements in these spectroscopic methods have enhanced their precision, sensitivity and application scope, particularly in food safety and environmental monitoring. Fluorescence Spectroscopy measures the emission of light by a substance that has absorbed light, while UV/Vis spectroscopy examines the absorption and transmission of ultraviolet and visible light through a sample. Both techniques are known for their high sensitivity and ability to provide realtime analysis. This section will explore the key developments and applications of fluorescence and UV/Vis spectroscopy in detecting pesticide residues.

2.4.2. Example Application of Fluorescence Spectroscopy in Pesticide Residue Detection

Pesticides, including insecticides, herbicides, fungicides, and other pest control agents, are tightly regulated due to their potential risks. The 2005 FDA Glossary lists 1,045 entries, each featuring a variety of heteroatoms and functional groups. As such, developing analytical methods capable of detecting multiple pesticide classes simultaneously in a single analysis is crucial. Traditional techniques, such as liquid chromatography and gas chromatography coupled with tandem mass spectrometry (LC–MS/MS and GC–MS/MS), are commonly used due to their speed and sensitivity. However, these methods can struggle with challenges like complex sample matrices, isobaric interferences, and the rapid degradation of analytes during ionization.

Fluorescence spectroscopy presents a potential solution to some of these challenges. Recent advancements have focused on enhancing the sensitivity and specificity of fluorescence-based techniques. For instance, vacuum ultraviolet (VUV) detectors enable rapid measurement of absorption spectra in the 115 to 240 nm range, which covers almost all chemical species. This broad spectrum allows for the differentiation of isomers with identical mass spectra but distinct absorption profiles. Unlike mass spectrometry, VUV spectroscopy does not require ionization, making it well-suited for unstable compounds that degrade quickly under MS conditions. Additionally, its high data acquisition rate (up to 100 Hz) makes VUV ideal for fast GC applications.

An initial study using the GC–VUV method demonstrated a detection limit of 186 pg on-column for captan, a fungicide that is typically difficult to detect using GC–MS. The study aimed to highlight VUV's potential as a universal detector for identifying multiple pesticide classes. Absorption spectra were collected for various pesticides, including organochlorines, organophosphates, carbamates, and pyrethroids, with particular attention to the detector's ability to distinguish signals from co-eluting compounds, enabling sensitive quantitative analysis.

As fluorescence spectroscopy continues to advance, it offers a highly effective and sensitive approach for detecting pesticide residues. Its ability to handle complex sample matrices and differentiate a wide range of pesticide classes makes it a valuable tool for ensuring food safety and environmental protection.

2.4.3. Example Application of Ultraviolet/Visible Spectroscopy (UV/Vis) in Pesticide Residue Detection

Paclobutrazol is a widely used plant growth regulator that delays growth, inhibits stem elongation, increases stress resistance and boosts yield in crops such as rice, wheat and peanuts. Due to various pesticide residue incidents raising public concern, detecting paclobutrazol residues has become crucial. Traditional methods like gas chromatography, high-performance liquid chromatography (HPLC), ultra-high-performance liquid chromatography-tandem mass spectrometry (UPLC-MS/MS), and immunoassays are effective but have limitations, including complex sample processing and high reagent consumption.

Fluorescence spectroscopy provides a solution with its high selectivity, sensitivity, and non-destructive detection capabilities. Derivative fluorescence spectra, obtained from the first derivative of the fluorescence spectrum, enhance detailed spectral characteristics, offering narrow bands, high sensitivity and comprehensive information. This method is widely used to analyse pesticide residues.

A fluorescence spectrum measurement system was employed to detect pesticide residues in paclobutrazol solutions and apple juice mixed with paclobutrazol. The system predicted pesticide residue quantitatively and evaluated performance based on the model correlation coefficient, recovery rate and relative standard deviation. The study demonstrated the capabilities of fluorescence spectroscopy in providing sensitive and accurate pesticide residue analysis, emphasising its utility in food safety and environmental protection [79].

2.4.4. Summary of Recent Advances

Recent years have seen significant progress in fluorescence spectroscopy for pesticide residue detection. Zhao et al. demonstrated laser-induced fluorescence with excellent discrimination potential for carbendazim, diazine, fenvalerate and pentachloronitrobenzene at 10 ppb concentrations [51]. Yu et al. achieved high recovery rates for paclobutrazol using fluorescence techniques [52]. Wu highlighted fluorescence's excellent application potential for detecting starane at 4.2 × 10−6 M [53]. Zhang developed a rapid fluorescence method for 2,4-D, providing an effective platform for detecting trace food pollutants in complex matrices at 90 nM [54].

Li et al. focused on micro-/mesoporous fluorescent sensors for imidacloprid, achieving excellent reusability and sensitive fluorescence responses at 30 ppb [55]. Guo et al. introduced a multienzyme-targeted fluorescent sensor for dichlorvos, offering sensitivity at 1.14 pg/L and in situ visualization [56]. Dong et al. simplified organophosphorus detection using fluorescence, achieving sensitivity at 15.03 pg/mL [57]. Vadia et al. developed a "Turn OFF-ON" fluorescence sensor for Fe3+ ions and propiconazole in pharmaceutical and vegetable samples, with detection limits of 0.18 μ M and 0.054 μ M, respectively [58].

In UV/Vis spectroscopy, Wan et al. combined GC-VUV for powerful multiclass pesticide screening of 38 pesticides [78]. Fan et al. integrated QuEChERS technology with UV spectroscopy to explore rapid screening approaches for bensulfuron-methyl, propanil and cypermethrin in rice, focusing on overcoming challenges of low content and high matrix interference [79]. Sahu et al. developed simple, selective and rapid methods using UV-Visible spectrophotometry and FTIR for flonicamid detection [80].

These advancements in fluorescence and UV/Vis spectroscopy highlight significant improvements in sensitivity, selectivity, and real-time capabilities for pesticide residue detection, ensuring more efficient and reliable methods for food safety and environmental protection.

2.5. Surface Plasmon Resonance (SPR)

2.5.1. Introduction

Surface plasmon resonance (SPR) is an advanced optical technique that measures changes in the refractive index near a sensor surface, allowing for the real-time monitoring of molecular interactions without the need for labels. SPR sensors work by utilising polarised light to detect changes at the sensor surface, where specific receptors bind to target molecules. In an SPR sensor, polarised light is directed at a metal film through a prism. When the light reaches the metal surface at a specific angle, it induces surface plasmons, which are electron oscillations at the interface between the metal and the dielectric (typically the sample).

The binding of target molecules (analytes) to the receptors on the sensor surface changes the local refractive index, causing a shift in the resonance angle of the polarised light. This shift is measurable and can be related to the concentration of the target molecules. The prism is essential in achieving the precise angle needed to excite the surface plasmons.

By monitoring these changes in the resonance angle, SPR sensors provide real-time, label-free detection of various biological and chemical substances, making them highly sensitive and selective. This makes SPR sensors an invaluable tool in applications such as medical diagnostics, environmental monitoring, and food safety.

This method has been adapted for detecting pesticide residues, offering rapid, highly sensitive, and specific analysis. SPR sensors are especially useful in food safety and environmental monitoring due to their ability to detect multiple analytes simultaneously and provide continuous, real-time data. Recent advancements in SPR technology have focused on improving sensor performance, enhancing detection limits, and expanding the range of detectable pesticides.

2.5.2. Example Application of SPR in Pesticide Residue Detection

Introduced in 2002, boscalid is a carboxamide fungicide, while clothianidin and nitenpyram are neonicotinoid insecticides introduced in 2002 and 1995, respectively. Clothianidin, a derivative of nitenpyram, contains a chlorothiazol ring, whereas nitenpyram includes a chloropyridine ring. These pesticides are frequently applied simultaneously in agriculture to prevent fungal diseases and insect pests, especially in warm and humid conditions. In Japan, the maximum residue limits (MRLs) are 1–40 mg/kg for boscalid, 0.2–40 mg/kg for clothianidin and 0.5–5 mg/kg for nitenpyram.

This research introduces a novel simultaneous SPR immunosensor designed to detect boscalid, clothianidin and nitenpyram without cross-reactions among these chemicals. Surface plasmon resonance (SPR) sensors operate by utilising polarised light to detect changes at the sensor surface. In an SPR sensor, polarised light is directed towards a metal film through a prism, which induces surface plasmons - oscillations of electrons at the metal-dielectric interface.

The innovative design of this immunosensor employs hapten derivatives specific to each pesticide, allowing for the selective capture and quantification of each target analyte in a single assay. When the target pesticides bind to the receptors on the sensor surface, they alter the local refractive index, causing a shift in the resonance angle of the polarised light. This shift is measured and correlated to the concentration of each pesticide. The prism plays a crucial role in achieving the precise angle needed to excite the surface plasmons, ensuring accurate detection.

This advanced method allows for the simultaneous monitoring of multiple pesticide residues in agricultural products, providing a practical and efficient solution for food safety. The SPR immunosensor's ability to detect several pesticides concurrently without cross-reaction demonstrates its high selectivity and reliability. Additionally, the real-time monitoring potential of the SPR sensor ensures rapid detection, which is critical for timely intervention in food safety measures.

The simultaneous detection capability and the real-time monitoring potential of this SPR immunosensor showcase its high applicability and effectiveness in ensuring the safety of food products from pesticide contamination. By leveraging the principles of SPR and the specificity of hapten derivatives, this sensor system represents a significant advancement in the field of biosensors, offering robust and efficient solutions for agricultural and environmental applications [71].

2.5.3. Summary of Recent Advances in SPR for Pesticide Residue Detection

In recent years, Surface Plasmon Resonance (SPR) sensors have seen significant advancements in detecting pesticide residues. Rajan reported that while the sensitivity of SPR sensors decreases with increasing pesticide concentration, the detection accuracy improves[69]. Saylan demonstrated the reusability, fast response, and ease of use of SPR sensors for detecting and real-time monitoring cyanazine, simazine and atrazine [70].

Hirakawa et al. developed a rapid, accurate, and simultaneous SPR sensor for detecting boscalid, clothianidin, and nitenpyram [71]. Miyake et al. showcased the high applicability of SPR sensors in analysing pesticide residues like azoxystrobin in vegetable samples, including boscalid, chlorfenapyr, imazalil, isoxathion and nitenpyram [72]. Jiao et al. highlighted the power of SPR-based immunosensors in providing detailed binding information between antibodies and pesticide targets [73].

Tomassetti et al. focused on atrazine detection, demonstrating better selectivity toward non-triazine pesticides [74]. Dissanayake et al. emphasised the importance of SPR sensors in the practical development of devices for organophosphorus pesticide detection [75]. Yao et al. reported excellent sensitivity, selectivity, and high stability of SPR sensors for detecting chlorpyrifos [76]. Çakır and Baysal developed SPR sensors with higher selectivity, sensitivity and lower detection limits for dimethoate and carbofuran compared to LC–MS/MS [77].

These advancements highlight the versatility and enhanced performance of SPR sensors in pesticide residue detection, ensuring reliable and efficient monitoring for food safety and environmental protection.

2.6. Surface-Enhanced Raman Scattering (SERS)

Surface-enhanced Raman scattering (SERS) is a highly sensitive optical technique that amplifies the Raman scattering effect through the use of nanostructured materials. SERS sensors exploit the interaction of light with these nanostructures to detect analytes with exceptional sensitivity. Typically, a layer of nanostructured gold particles is employed to enhance the Raman scattering signals of target molecules.

The sensor surface is functionalised with specific receptors that bind selectively to the analytes. When light is directed onto the sensor, it interacts with the nano gold particle layer, generating an enhanced electromagnetic field. This field amplifies the Raman scattering signals of the molecules bound to the receptors, allowing for the detection of even trace amounts of analytes. The resulting Raman spectra provide unique molecular fingerprints, offering detailed information about the composition and concentration of the target substances.

The use of nano gold particles is pivotal, as their presence significantly enhances the sensor's sensitivity, enabling the detection of low-concentration analytes. This high sensitivity, coupled with the ability to provide detailed molecular information, makes SERS an ideal technique for detecting pesticide residues. By leveraging the distinctive properties of nanomaterials, SERS achieves high specificity and precision.

Recent advancements in SERS technology have focused on improving its sensitivity, selectivity, and reproducibility, thereby broadening its applications in food safety and environmental monitoring. Innovations in nanomaterial design and sensor fabrication have further enhanced the performance of SERS sensors, ensuring reliable and consistent detection results.

This section explores the critical developments and diverse applications of SERS in the analytical detection of pesticide residues, highlighting its potential to revolutionise monitoring practices in agriculture and environmental protection.

2.6.1. Example Application of SERS in Pesticide Residue Detection

Pesticides, particularly organic sulfur-based broad-spectrum protective bactericides, are widely used to improve the quality of fruits and vegetables by inhibiting bacterial enzymes and disrupting metabolic cycles. However, the residues of these pesticides on the surfaces of fruits and vegetables pose significant health risks, including neuronal damage and potential death of the central nervous system through bioaccumulation in the food chain. This underscores the critical need for sensitive and rapid methods to analyse pesticide residues in food.

This study presents a novel approach to fabricating surface-enhanced Raman scattering (SERS) substrates using three-dimensional gold (3D Au) nanostructures anchored on polydimethylsiloxane (PDMS) membranes (3D Au@PDMS). SERS sensors amplify Raman scattering signals by leveraging the interaction of light with nanostructured materials. In this study, tightly packed 3D Au nanostructures were created via organic-aqueous interfacial self-assembly and then transferred onto a transparent and flexible PDMS surface. These substrates exhibit unique advantages, such as flexibility, optical transparency, and the ability to stand unsupported, making them versatile and robust.

The PDMS membrane serves as a durable and adaptable platform, while the 3D Au nanostructures generate a surface capable of significantly enhancing the electromagnetic field when exposed to light. This enhancement is attributed to the formation of numerous "hot spots" - regions of concentrated electromagnetic fields - within the bilayer films of Au nanoparticles. These hot spots amplify the Raman scattering signals of molecules bound to the sensor surface, enabling highly sensitive detection. Compared to monolayer films, the bilayer films of Au nanoparticles offer substantially stronger electromagnetic enhancement, improving the efficiency of the detection process.

This advanced sensor system enables the rapid and simultaneous detection of thiram residues on the peels of various fruits and vegetables. By utilising the unique properties of the 3D Au@PDMS SERS substrates, this study highlights a promising method for labelfree and efficient detection of pesticide residues. The innovative design demonstrates the potential of SERS technology in practical applications, providing a robust and effective solution for ensuring food safety. The ability to quickly and accurately detect pesticide residues with these substrates underscores their significance in environmental monitoring and public health [66].

2.6.2. Summary of Recent Advances in SERS for Pesticide Residue Detection

Recent years have witnessed significant advancements in the development of surface-enhanced Raman scattering (SERS) sensors for pesticide residue detection. These innovations have expanded the applicability of SERS for rapid, sensitive and reliable analysis, particularly in complex matrices.

Wang et al. combined SERS with dispersive liquid-liquid microextraction, achieving excellent sensitivity and stability for the rapid detection and quantification of organophosphorus pesticides (OPPs) such as triazophos and parathion-methyl [59, 60]. Guo et al. utilised plasmonic core-shell nanoparticles, including nanocubes and nanocuboids, to enhance SERS sensitivity for various analytes, further demonstrating the versatility of this technique [59].

Zhang et al. highlighted the capability of SERS to detect multiple pesticides, including thiram and methamidophos, directly from fruit surfaces, showcasing its applicability in real-world, complex matrices [61]. Similarly, Abbas et al. developed SERS sensors with high sensitivity, ultra-low detection limits and remarkable uniformity for the detection of chlorpyrifos [62]. Tang et al. focused on detecting chlorpyrifos and imidacloprid, demonstrating SERS as a promising tool for practical applications in food safety and environmental monitoring [63].

Kumar et al. introduced a straightforward "paste and peel off" method for using SERS to detect trace amounts of thiram directly from fruit peels, offering a simple and effective approach [64]. Ma et al. emphasised the remarkable sensitivity of SERS for detecting paraoxon [65], while Xie et al. showcased its potential for rapid, high-sensitivity, on-site detection of contaminants such as sumithion and thiram, particularly on nonplanar surfaces [66].

Ye et al. and Lee et al. explored quantitative analysis using SERS, detecting pesticides such as triazophos, thiram, fonofos and ferbam. Their work highlighted the high sensitivity, cost-effectiveness and ease of fabrication of these sensors [67, 68].

These advancements underscore the transformative potential of SERS in pesticide residue detection, offering rapid, sensitive, and practical solutions for ensuring food safety and environmental protection. The continuous evolution of this technology positions SERS as a vital tool in the ongoing efforts to safeguard public health and the environment.

3. Conclusion

The analytical detection of pesticide residues has seen remarkable advancements, particularly with the development of electrochemical and optical sensors. These technologies have significantly improved the sensitivity and accuracy of detection, playing a pivotal role in enhancing food safety and environmental monitoring. Moving forward, continuous innovation in sensor materials and technologies holds great promise for even more efficient and robust detection methods. Integrating these advanced sensors into routine monitoring practices will be essential in mitigating the adverse effects of pesticide use, ensuring a healthier and safer environment for all.

Looking ahead, several key areas present exciting opportunities for future research and development. One promising avenue is the miniaturisation of sensor technologies. By creating smaller, more portable sensors, real-time monitoring becomes feasible across diverse settings, from agricultural fields to food processing facilities. This portability could greatly enhance the convenience and accessibility of pesticide residue detection, enabling on-the-spot decision-making and more widespread adoption.

Another critical focus is the development of user-friendly, home-based detection systems. As sensors become more affordable and easier to use, they could empower consumers to regularly test their produce at home. This democratisation of technology has the potential to reduce reliance on centralised testing facilities, improve public awareness, and encourage proactive prevention of pesticide contamination.

Additionally, the integration of artificial intelligence (AI) presents a transformative opportunity for enhancing sensor performance. AI algorithms can optimise the recognition and quantification of pesticide residues by analysing complex data patterns that tra-

ditional methods might overlook. AI-driven sensors could offer more accurate and reliable results, with rapid real-time data processing and decision-support capabilities, further streamlining the detection process.

The future of pesticide residue detection lies in the convergence of these innovative approaches—miniaturisation, home-based systems and AI optimisation. By pushing the boundaries of sensor technology, we can develop sophisticated and effective methods for safeguarding food safety and protecting the environment. Through ongoing research, interdisciplinary collaboration and technological advancements, we are poised to create a healthier and safer future for all.

Target substance Testing methods LOD Reference Brief review/features

Appendix A

whololayer Molec-
ular Crystal-FET 0.60ppb [13] The devices also show excellent conformal ability on various curved surfaces with negligible performance degradation.

Monolayer Mo-Monolayer Molec-

lecular Crystal-

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