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Review article

# A review of emerging techniques for pyrethroid residue detection in agricultural commodities

Dirong Goh [a], Ahmad Faizal Abdull Razis [a,b,c,*], Nor Azah Yusof [d], Norida Mazlan [e], Noordiana Nordin [b], Choo Yee Yu [f]

[a] Natural Medicines and Products Research Laboratory, Institute of Bioscience, Universiti Putra Malaysia, 43400, UPM Serdang, Selangor, Malaysia
[b] Laboratory of Food Safety and Food Integrity, Institute of Tropical Agriculture and Food Security, Universiti Putra Malaysia, 43400, UPM Serdang, Selangor, Malaysia
[c] Department of Food Science, Faculty of Food Science and Technology, Universiti Putra Malaysia, 43400, UPM Serdang, Selangor, Malaysia
[d] Department of Chemistry, Faculty of Science, Universiti Putra Malaysia, 43400, UPM Serdang, Selangor, Malaysia
[e] Department of Agriculture Technology, Faculty of Agriculture, Universiti Putra Malaysia, 43400, UPM Serdang, Selangor, Malaysia
[f] Laboratory of Vaccine and Biomolecules, Institute of Bioscience, Universiti Putra Malaysia, 43400, UPM Serdang, Selangor, Malaysia

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ABSTRACT

Pyrethroid pesticides are essential for modern agriculture, helping to control pests and protect crops. However, due to growing concerns about their potential impact on human health and the environment, reliable detection methods are essential to ensure food safety. In this literature review, we explore the techniques used over the past decade to detect pyrethroid residues in agricultural products. Until now, various methods have been developed for detecting pyrethroid pesticides, ranging from conventional analytical approaches to innovative approaches. The conventional analytical approaches include gas, liquid, and supercritical fluid chromatography, micellar electrokinetic capillary chromatography, and enzyme-linked immunosorbent assay. Whereas innovative approaches refer to various optical-based and electrochemical-based sensors. For each method, we evaluate its strengths, limitations, and practical applications. Recent innovations are highlighted, focusing on sensitivity, selectivity, and practical applicability. By summarizing the current state of research, this review serves as a valuable resource for researchers and practitioners, providing insights into the evolving technology and strategy for detecting pyrethroid residue.

## 1. Introduction

Pyrethroid residues in agricultural commodities are a subject of global concern due to their potential impact on ecosystems, non-target organisms, and human health. The widespread application of pyrethroids in agriculture has led to their presence in sediment and water bodies, impacting benthic invertebrates and aquatic organisms [1,2]. Pyrethroids have proven effective against a broad spectrum of insect pests threatening fruit and vegetable crops, including Lepidopteran insects, aphids, whiteflies, leaf miners, and fruit borers [3]. As the demand for effective pest management grows, it is essential to carefully balance the benefits and risks of pyrethroid

* Corresponding author. Natural Medicines and Products Research Laboratory, Institute of Bioscience, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia.
*E-mail address:* madfaizal@upm.edu.my (A.F. Abdull Razis).

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use, which requires a sophisticated approach to residue detection.

When it comes to physicochemical properties, pyrethroids possess distinct characteristics, including a highly non-polar nature, low water solubility, and a strong affinity for soil and sediment particulate matter [4]. Pyrethroids are engineered to be more photostable, making them suitable for use in agricultural settings [5,6]. While they are not persistent and can be metabolized by mammals, pyrethroids have been identified to bioaccumulate in marine mammals and humans [7,8]. Pyrethroids undergo metabolic processes involving esterase and oxidase activities in mammals, insects, other organisms, and microsomal esterase and oxidase systems [9]. Typically, the metabolism of pyrethroids through esterase and oxidase actions tends to restrict their toxicity to mammals more than to insects, resulting in valuable selective toxicity properties [10–13]. Notably, pyrethroids undergo rapid degradation in soil, suggesting potential environmental safety [14–16].

Accurate detection methods for monitoring and regulating pyrethroid pesticide levels in agricultural commodities are crucial for ensuring food safety and environmental protection. With the emergence of pesticide-resistant pests, formulations containing one or more pyrethroids have been developed and applied. This has resulted in the presence of multiple pyrethroids in agricultural products, emphasizing the necessity for accurate detection methods [17]. While conventional large-scale instrument detection methods are accurate, they are not suitable for real-time and rapid field detection [18]. Advanced detection techniques, such as electrochemical and optical sensors, offer promising alternatives [19]. When combined with effective extraction techniques, these methods can reliably quantify pesticide residues in various food samples [20].

This review focuses on the imperative of detecting pyrethroid residues in agricultural products, balancing the necessity for food security with the need to mitigate ecological and health risks associated with pyrethroid residues. We comprehensively explore conventional analytical approaches and innovative approaches as indicated in Fig. 1, providing an overview for researchers and

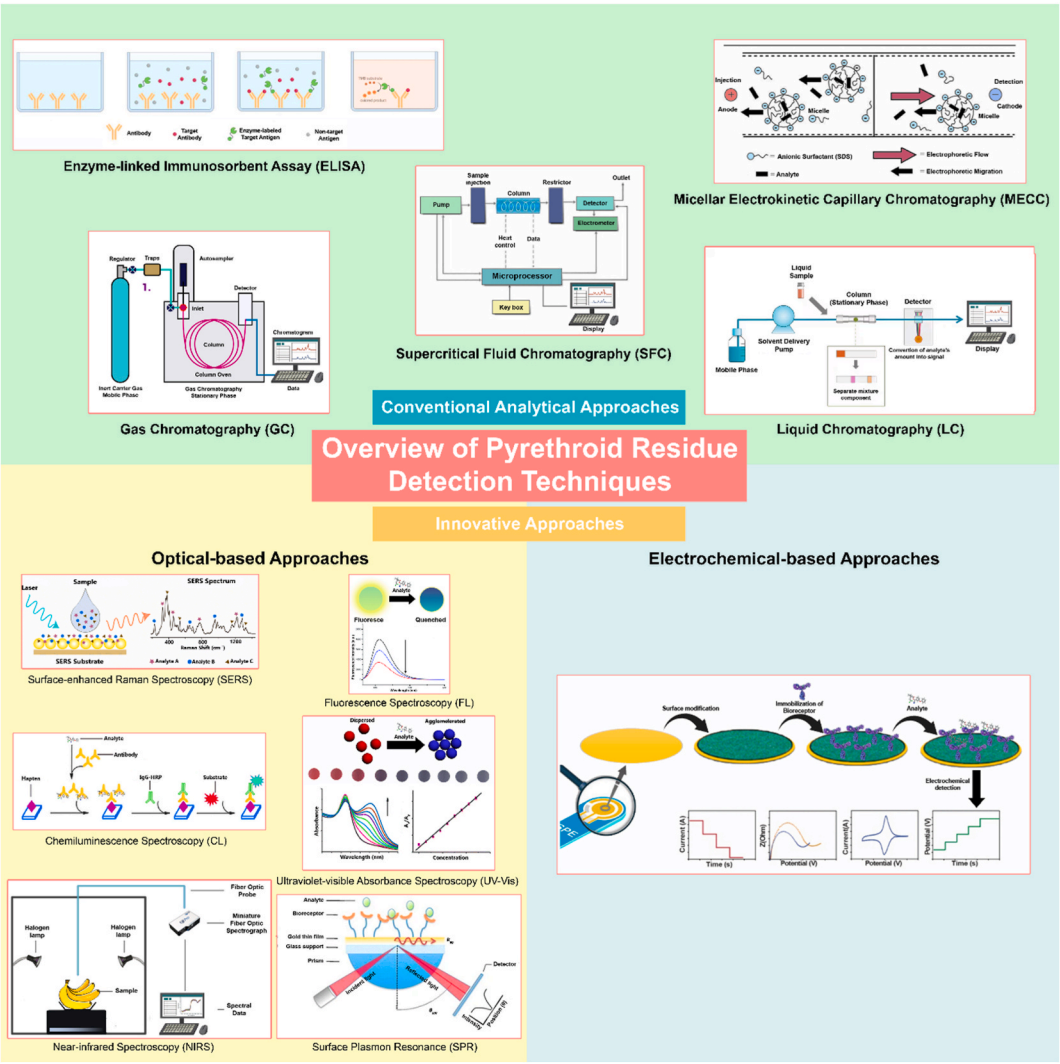


**Fig. 1.** An infographic overview outlining the detection techniques for pyrethroid pesticide residues in agricultural commodities.

practitioners to select suitable methods for different situations. We emphasize the dynamic relationship between emerging technologies and evolving pyrethroid usage, stressing the importance of adaptable and sensitive detection methods aligned with changing agricultural practices.

## 2. Conventional analytical approaches for pyrethroids detection

### 2.1. Gas chromatography (GC)

Gas chromatography (GC) is a powerful analytical technique widely employed in the field of pesticide residue analysis. GC is especially effective for the analysis of volatile and semi-volatile pesticides, such as organophosphorus, pyrethroid, and organochlorine compounds, due to their stable thermal properties, low polarity, and volatility [21]. While liquid chromatography also has its advantages and can be suitable for certain applications, gas chromatography is often preferred for the detection of pyrethroid pesticides due to its superior performance in terms of volatility, sensitivity, specificity, and speed. Liquid chromatography is generally less sensitive than gas chromatography for the analysis of pyrethroid pesticides, making gas chromatography the preferred method for their detection [22,23].

Detectors commonly employed in the detection of pyrethroid pesticides in GC include the electron capture detector (ECD), flame ionization detector (FID), mass spectrometry (MS), and tandem mass spectrometry (MS/MS), as outlined in Table 1. Given that many pyrethroid pesticides contain halogens such as fluorine, chlorine, or bromine, they exhibit a propensity for detection through ECD due to their sensitivity to halogen-containing compounds. Conversely, pyrethroids do not efficiently undergo ionization in a flame, thereby limiting the utility of FID for their detection, an observation underscored in the literature [24,25]. MS, renowned for its high sensitivity and ability to discern compounds based on their mass-to-charge ratio, serves as a suitable technique for pyrethroid detection. However, MS/MS surpasses single MS in sensitivity and specificity, making it ideal for targeted analysis of pyrethroids in complex sample matrices.

In GC analysis, sample preparation plays a crucial role encompassing several key steps. These steps involve solvent selection, homogenization, extraction, clean-up, pre-concentration, and optionally, derivatization. Solvent selection is critical for optimizing extraction efficiency, while homogenization ensures sample uniformity, thereby enhancing reproducibility. Extraction isolates analytes from complex matrices, while clean-up removes interfering substances. Pre-concentration enhances detection sensitivity by concentrating analytes. Optionally, derivatization modifies analytes to improve their chromatographic behavior. Notably, pyrethroid pesticides stand out as an exception to this, as their inherent chemical properties, such as favorable volatility, thermal stability, and adequate polarity, render derivatization unnecessary for GC analysis [26], making them amenable to direct analysis by GC without the need for derivatization.

### 2.2. Liquid chromatography (LC)

Liquid chromatography (LC) methods for detecting pyrethroids in agricultural products operate based on the principle of separating compounds according to their affinity for a stationary phase and a mobile phase. Specifically, reverse-phase liquid chromatography (RP-LC) is often preferred for pyrethroid pesticide analysis. In RP-LC, a non-polar stationary phase, such as $C_{18}$ or $C_8$, interacts with the pyrethroids, which are typically non-polar or weakly polar. The mobile phase for pyrethroid detection, usually consisting of buffered water and an organic solvent (acetonitrile or methanol), is more polar than the stationary phase. Pyrethroid compounds are eluted from the column at different rates depending on their chemical properties, such as polarity and molecular weight. This polarity gradient allows for the efficient separation of pyrethroid pesticides based on their hydrophobicity. The use of various detection modes (Table 2.), such as ultraviolet detection (UV), fluorescence detection (FLD), diode array detection (DAD), and tandem mass spectrometry (MS/MS), allows for sensitive and selective detection of pyrethroids within complex matrices.

As mentioned before, the physicochemical properties of pyrethroid pesticides made them less suitable to be detected using LC. Generally, the reverse phase of liquid chromatography coupled with electrospray ionization (ESI) sources in mass spectrometry has commonly yielded unsatisfactory results for pyrethroid detection [62]. Besides, employing high-performance liquid chromatography (HPLC) with UV or fluorescence detection for analyzing pyrethroid residues in fruits and vegetables faces challenges due to the potential occurrence of multiple analytes with similar retention times. This similarity in retention times can impede the unequivocal identification of pyrethroids [22].

In addition to direct LC analysis, derivatization of type II pyrethroids to a common chemical product, 3-phenoxybenzoic acid (3PBA), before analysis has been found to enhance sensitivity compared to detecting the parent compound [63]. Moreover, this approach simplifies the detection of individual pyrethroids rather than an entire class. Notably, this strategy enables semiquantitative analysis of the total amount of type II pyrethroids without the need for an extraction step. While acknowledging that endogenous 3-PBA presents a limitation, potentially inflating pyrethroid contamination values in samples, this can be mitigated through pre- and post-oxidation procedure analysis. Despite inherent variability in pyrethroid conversion, the advantages in analysis time and sensitivity may outweigh this drawback.

Recently, Yuan et al. [64] conducted a comparative analysis of multi-residue analysis methods for 32 pyrethroids in fruit and vegetable samples. They utilized gas chromatography coupled to tandem mass spectrometry (GC-MS/MS) and ultra-high performance liquid chromatography coupled to tandem mass spectrometry (UHPLC-MS/MS) for their investigation. The comparison focused on parameters such as recovery, LOQ, linearity, and matrix effects. UHPLC-MS/MS demonstrated suitability for a greater number of pesticides compared to GC–MS/MS, with lower LOQs observed for most selected pyrethroids. Specifically, all selected pyrethroids

**Table 1**
Summary of gas chromatography-based extraction and detection techniques for pyrethroids in the agricultural field over the past 10 years.

| Extraction | Detector | Pyrethroids | Sample matrix | Limit of detections (LODs) | References |
|---|---|---|---|---|---|
| LVSE | MS/MS | Phenothrin, bifenthrin, tetramethrin, cis&trans-permethrin, cyfluthrin I&II, cypermethrin, etofenprox, fenvalerate, deltamethrin, | Hemp seed oil | LOQ: 0.01–0.5 mg/kg | [27] |
| SPME | MS | *S*-bioallethrin, bifenthrin, fenpropathrin, permethrin, *1*-cyhalothrin, cypermethrin, fenvalerate, deltamethrin | Apple juice, peach juice, grape juice, orange juice, watermelon juice, Tongguanteng oral liquid, Shuanghuanglian oral liquid, three herbal extract granules, Banlangen granule, Honeysuckle granule, and Ganmaoling granule | 0.4–2.0 ng/mL | [28] |
| NLPNE | MS | Allethrin, bifenthrin, tetramethrin (isomers A and B), fenpropathrin, cyhalothrin (isomers A and B), fenvalerate (isomers A and B), deltamethrin | Tea | 0.56–13.37 ng/g | [29] |
| MA-HLLME | MS | Deltamethrin, bifenthrin, permethrin, cyhalothrin, cypermethrin | Grape, apple, apricot, peach, sour cherry, potato, onion and tomato | 4.3–9.4 ng/kg | [30] |
| QuEChERS | ECD | Bifenthrin, fenpropathrin, cyhalothrin, Deltamethrin, cyfluthrin, permethrin, cypermethrin, flucythrinate, fenvalerate, flumethrin | Cucumber and greengrocery | 0.0001–0.007 mg/kg | [26] |
| MSPE | FID | Bifenthrin, fenpropathrin, cypermethrin, permethrin, fenvalerate | Tomato, pear, cabbage, pakchoi cabbage, and honey | 0.34–0.84 μg/L | [25] |
| EVA–DLLME | MS | Permethrin, phenothrin, tetramethrin, cypermethrin, cyhalothrin, bifenthrin | Grape, pomegranate, orange, apple, and sour cherry | 9–21 ng/L | [31] |
| QuEChERS | ECD | Cypermethrin | Mango, guava | 0.004 mg/kg | [23] |
| MDSPE | MS | Bifenthrin, phenothrin, tetramethrin, cyhalothrin, permethrin, cypermethrin | Pomegranate, apple, grape, sour cherry, orange, and apricot juices | 4.0–12 ng/L | [32] |
| QuEChERS, DLLME-SFO | MS | Bifenthrin, cyhalothrin, permethrin, fenvalerate, deltamethrin | lettuce, long bean, broccoli, tomato, Carrot, pumpkin, siew pak choy, sweet choy sum, sweet pak choy, celery, amaranth, spinach, cabbage, mushroom, cucumber | 0.3–0.6 μg/kg | [33] |
| MSPE | ECD | Fenpropathrin, λ-cyhalothrin, cyfluthrin, fenvalerate, deltamethrin | Orange juice, peach juice, grape juice, pear juice and lemon juice | 0.007–0.015 μg/L | [34] |
| BSME, QuEChERS | MS | γ-cyhalothrin | Pineapple | 1.6 μg/L | [35] |
| QuEChERS | MS/MS | Bifenthrin, fenpropathrin, cyhalothrin, permethrin, cyfluthrin, cypermethrin, flucythrinate τ-fluvalinate, fenvalerate, deltamethrin | Oyster mushroom, shiitake mushroom, eryngii mushroom, crimini mushroom, enoki mushroom, and bunashimeji mushroom | 0.015–1.67 μg/kg | [36] |
| QuEChERS | MS/MS | Bifenthrin, permethrin, flucythrinate, τ-fluvalinate, fenvalerate, phenothrin, bioallethrin, cypermethrin, tefluthrin, tetramethrin, λ-cyhalothrin, cyfluthrin, etofenprox, deltamethrin | Apples, mangos, strawberries, cucumbers and tomatoes | 1.34–5.53 μg/kg | [37] |
| QuEChERS | MS/MS | Bifenthrin, fenpropathrin, cyhalothrin, permethrin, cyfluthrin, cypermethrin, fenvalerate | Tomatoes | 0.1–6.0 μg/kg | [38] |
| QuEChERS | ECD | Bifenthrin, λ-cyhalothrin, fenvalerate, deltamethrin, fenpropathrin, α-cypermethrin, fluvalinate | Cauliflower | LOQ: 0.05 mg/kg | [39] |
| HLLME | MS | Bifenthrin, phenothrin, tetramethrin, fenpropathrin, cyhalothrin, permethrin, cypermethrin, cyfluthrin, flucythrinate, deltamethrin | Grape, sour cherry, mango, apricot, peach, and orange juices | 0.006–0.038 ng/mL | [40] |
| QuEChERS | MS/MS | Bifenthrin, fenpropathrin, cyhalothrin, permethrin, cyfluthrin, cypermethrin, flucythrinate, fenvalerate, τ-fluvalinate, deltamethrin | Pear, waxberry, tomato, cucumber, cowpea | 0.3–4.9 μg/kg | [41] |
| SPME | ECD | Bifenthrin, fenpropathrin, cyhalothrin, permethrin, cyfluthrin, flucythrinate, fenvalerate, deltamethrin | Peach, cucumber, cabbage | 0.1–0.5 ng/g | [42] |
| SPE | MS | Bifenthrin, fenpropathrin, cyhalothrin, cyfluthrin, cypermethrin, flucythrinate, fluvalinate, fenvalerate, deltamethrin | Shallot, ginger, garlic, onion, leek, celery | 0.01–0.03 mg/L | [43] |
| HS-SPME | ECD | Bifenthrin, λ-cyhalothrin, β-cyfluthrin, flucythrinate-I&II | Apple, cucumber | 0.11–0.23 μg/kg | [44] |
| QuEChERS | ECD | Bifenthrin, fenpropathrin, cyhalothrin, permethrin, cyfluthrin, cypermethrin, fenvalerate, deltamethrin | Green pepper, red pepper, dehydrated red peppers. | 0.0012–0.012 mg/kg | [45] |

(*continued on next page*)

**Table 1** (*continued*)

| Extraction | Detector | Pyrethroids | Sample matrix | Limit of detections (LODs) | References |
|---|---|---|---|---|---|
| QuEChERS | MS/MS | Bifenthrin, fenpropathrin, λ-cyhalothrin, permethrin I&II, cyfluthrin, cypermethrin, α-cypermethrin, fenvalerate, fluvalinate I&II, deltamethrin | Tomato | 0.005 mg/kg | [46] |
| SPE | ECD | Bifenthrin, tetramethrin, fenpropathrin, permethrin, cypermethrin, fenvalerate, deltamethrin | Yam rhizomes, *Radix Puerariae Lobatae, Radix Ginseng, Ternate Pinellia, Chinese Thorowax* Root and *Pilose Asiabell* Root | 0.18–1.82 μg/kg | [47] |
| UA-DLLME-SFO | ECD | Bifenthrin, fenpropathrin, cyhalothrin, permethrin, cyfluthrin, cypermethrin, fenvalerate, deltamethrin | Green tea, black tea, jasmine tea, Púer tea, Tieguanyin tea | 0.08–0.5 μg/kg | [48] |
| MSPD | ECD | Cypermethrin, deltamethrin | Bovine milk | 0.002–0.007 μg/g | [49] |
| DLLME, QuEChERS | ECD | Tetramethrin, bifenthrin, λ-cyhalothrin, permethrin, cyfluthrin, cypermethrin, flucythrinate, fenvalerate, τ-fluvalinate, deltamethrin | Apple, pear, grape, peach, orange, lemon, kiwi and mango juices | 0.2–2 μg/L | [50] |
| DLLME | FID | Fenpropathrin, sumithrin, cyhalothrin, cis-permethrin, trans-permethrin, deltamethrin | Sunflower oil, corn oil, colza oil, olive oil | 0.02–0.17 mg/kg | [24] |
| SDME | MS | Bifenthrin, permethrin | Coconut water | 0.1–0.36 μg/L | [51] |
| SPE | MS/MS | Phenothrin, permethrin, cyfluthrin, cypermethrin, deltamethrin, fenvalerate | Chicken eggs | 1.0–7.0 μg/kg | [52] |
| QuEChERS | MS/MS | Acrinathrin, bifenthrin, cypermethrin, deltamethrin, esfenvalerate, λ-cyhalothrin, permethrin, resmethrin, τ-fluvalinate | Honey | 0.07–0.2 ng/g | [53] |
| SPE | MS/MS | Bifenthrin, λ-cyhalothrin, cypermethrin, fenvalerate | Green tea, dark tea, scented tea, black tea, and oolong tea | 0.2–5.0 μg/kg | [54] |
| QuEChERS | ECD | λ-cyhalothrin, cyfluthrin, cypermethrin | White and Black pepper | 0.002 mg/kg | [55] |
| QuEChERS | ECD | Cypermethrin, fenvalerate, fluvalinate, deltamethrin | tomato and brinjal | 0.01 mg/kg | [56] |
| SPE | ECD | Bifenthrin, cypermethrin, deltamethrin | Tea | 0.0056–0.071 mg/kg | [57] |
| SPE | ECD | tetramethrin, Fenpropathrin, cypermethrin, fenvalerate, deltamethrin | Bee pollens | 1.662–19.125 μg/kg | [58] |
| HF-LPME | MS | Fenpropathrin, cyhalothrin, deltamethrin | Vegetable juice, apple juice, peach juice, orange juice, kiwi juice | 0.02–0.07 ng/mL | [59] |
| MSPE | MS | bifenthrin, λ-cyhalothrin, cyfluthrin, cypermethrin, fenvalerate, deltamethrin | Orange and lettuce | 0.01–0.02 ng/g | [60] |
| QuEChERS | MS/MS | Transfluthrin, allethrin, bifenthrin, λ-cyhalothrin, permethrin, cyfluthrin, cypermethrin, ethofenprox, fenvalerate, τ-flivalinate, deltamethrin | Rice grains | 0.01–0.05 mg/kg | [61] |

LVSE, limited-volume solvent extraction; NLPNE, nanoconfined liquid phase nanoextraction; QuEChERS, quick, easy, cheap, rugged, and safe method; HF-LPME, Two-phase hollow fiber liquid phase microextraction; MSPE, magnetic solid-phase extraction; UA-DLLME-SFO, ultrasound-assisted dispersive liquid-liquid microextraction based on solidification of floating organic droplet method; MSPD, matrix solid-phase dispersion; DLLME, dispersive liquid-liquid microextraction; SDME, single-drop microextraction; HS-SPME, headspace solid-phase microextraction; LOQ, limit of quantification; HLLME, homogeneous liquid-liquid microextraction; DLLME-SFO, dispersive liquid-liquid microextraction based on solidification of floating organic droplet; BSME, binary solvent microextraction; MA-HLLME, microwave-assisted homogenous liquid-liquid microextraction; MDSPE, magnetic dispersive solid phase extraction; EVA–DLLME, evaporation–assisted dispersive liquid-liquid microextraction.

**Table 2**
Summary of liquid chromatography-based extraction and detection techniques for pyrethroids in the agricultural field over the past 10 years.

| Extraction | Detector | Pyrethroids | Sample matrix | Limit of detections (LODs) | References |
|---|---|---|---|---|---|
| bio-SUPRAS-LPME | UV | Fenvalerate, fenpropathrin, bifenthrin, etofenprox | Honey and whole milk | 5–10 μg/L | [65] |
| MIPs | MWD | λ-cyhalothrin | Apple and cucumber | 0.5 ng/g | [66] |
| MSPE | UV | Permethrin, beta-cyfluthrin, λ-cyhalothrin, bifenthrin | Cabbage | 0.0011–0.0058 μg/mL | [67] |
| MEA-SHS-DLLME | DAD | Tetramethrin, λ-cyhalothrin, fenvalerate | *Lentinus edodes, Flammulina velutiper, Pleurotus ostreatus, Auricularia auricular, and Hypsizygus marmoreus* (edible fungi) | 0.0067–0.0329 mg/kg | [68] |
| SBSE | UV | Fenpropathrin, s-cypermethrin, fenvalerate, permethrin, ethenothrin, bifenthrin | Tobacco leaves | 0.20–0.66 μg/L | [69] |
| MSPE | UV | Cyfluthrin, esfenvalerate, permethrin, bifenthrin | Cucumber, celery, and grapes | 3.98–6.21 ng/mL | [70] |
| QuEChERS | MS/MS | λ-cyhalothrin, bifenthrin, β-cyfluthrin, deltamethrin, permethrin, fenpropathrin, etofenprox | Tea, cucumber, and tomato | 0.007–1.875 μg/kg | [71] |
| CD-assisted DLLME-SFOD | UV | λ-cyhalothrin, deltamethrin, bifenthrin | Rice, wheat, maize, and millet | 3.5–9.5 μg/kg | [72] |
| QuEChERS | MS/MS | Acrinathrin, allethrin, bifenthrin, cyfluthrin, cyhalothrin, cypermethrin, cyphenothrin, deltamethrin, etofenprox, fenpropathrin, fenvalerate, flucythrinate, fluvalinate, permethrin, phenothrin, resmethrin. Silafluofen, tetramethrin, tralomethrin | Tea and orange | 0.07–0.29 μg/kg | [73] |
| SADLLME | DAD | Bifenthrin | Apple, pear, melon, and tomato | 2.3 μg/kg | [74] |
| UETC-IL-DLLME | DAD | Fenpropathrin, λ-cyhalothrin, fenvalerate, permethrin, bifenthrin | Herbal tea | 1.25–1.35 μg/L | [75] |
| UAATPE, VADLLME | DAD | Deltamethrin, permethrin, fenpropathrin, bifenthrin | Longan fruit | 0.005576–0.007738 μg/mL | [76] |
| SPE | UV | Tetramethrin, α-cypermethrin | Grapefruit, orange, spinach, celery, oats and *lyceum barbarum* | LOQ: 0.02–0.05 mg/kg | [77] |
| MSPE | UV | Deltamethrin, cypermethrin | Apple juice, cucumber juice, orange juice | 0.05–0.1 μg/L | [78] |
| DLLME | DAD | Transfluthrin, fenpropathrin, fenvalerate, ethofenprox, bifenthrin | black tea, green tea, oolong tea, apple juice, red grape juice and purple grape juice | 0.06–0.17 ng/mL | [79] |
| MAE, UADLLME | UV | Fenpropathrin, deltamethrin, fenvalerate, permethrin, etofenprox, bifenthrin | Litchi fruit | 1.15–2.46 μg/L | [80] |
| LSPE, MSPE | UV | β-cyfluthrin, bifenthrin, fenvalerate, permethrin, decamethrin | Cabbage, pakchoi, Chinese kale, rape, Chinese chive, lettuce, amaranth, broccoli, cauliflower, Chinese cabbage | 0.0200–0.0392 ng/g | [81] |
| QuEChERS | MS/MS | Permethrin, cypermethrin, deltamethrin, esfenvalerate, bifenthrin, cyfluthrin, cyhalothrin | Tomatoes, oranges, grapes, apples, bananas, onions, lettuce, green peppers, carrots and broccoli. | 1–141 ng/kg | [82] |
| SPE | UV | β-cyfluthrin, cyhalothrin, cyphenothrin, permethrin | Rhubarb, *Herba lysimachiae*, *Ardisia japonica,* and the fruit of *Camptotheca acuminata* | 0.0083–0.0108 μg/g | [83] |
| MSPE, SFE-MSPE | UV | Fenpropathrin, cyhalothrin, fenvalerate | Apple, peach, cucumber, and tomato | MSPE: 1 μg/L; SFE-MSPE: 0.1 mg/kg | [84] |
| SPE | FLD | Etofenprox | bell pepper, cucumber, eggplant, Japanese mustard spinach, spinach, and tomato | 0.62–1.28 ng/g | [85] |
| DLME, D-μ-SPE | UV | Tetramethrin, fenpropathrin, deltamethrin, permethrin | Cucumber and cabbage | 0.2–2.0 ng/g | [86] |
| LSE, MSPE | UV | λ-cyhalothrin, cypermethrin, deltamethrin, esfenvalerate, permethrin, bifenthrin | Chinese cabbage and celery | 0.63–1.2 ng/g | [87] |

bio-SUPRAS-LPME, bio-supramolecular solvent-based liquid phase microextraction; MIPs, molecularly imprinted polymers; MWD, multi wavelength detector; MSPE, magnetic solid-phase extraction; SBSE, stir bar sorptive extraction; CD-assisted DLLME-SFOD, cyclodextrin-assisted dispersive liquid-liquid microextraction based on solidification of floating organic droplets; MEA-SHS-DLLME, magnetic effervescence-assisted switchable solvent dispersive liquid-liquid microextraction; SADLLME, salting-out assisted extraction with the dispersive liquid-liquid microextraction; UETC-IL-DLLME, ultrasound enhanced temperature-controlled ionic liquid dispersive liquid-liquid microextraction; UAATPE, ultrasonic-assisted aqueous two-phase extraction; VADLLME, vortex-assisted dispersive liquid-liquid microextraction; MAE, microwave-assisted extraction; UADLLME, ultrasonic-assisted dispersive liquid-liquid microextraction; LSPE, liquid-solid phase extraction; SFE-MSPE, supercritical fluid extraction coupled with magnetic solid-phase extraction; DLME, dispersive liquid microextraction; D-μ-SPE, dispersive μ-solid phase extraction.

were found to be suitable for UHPLC-MS/MS, while only 29 pyrethroids were suitable for GC–MS/MS. Analysis of real samples using both techniques yielded similar results, suggesting UHPLC-MS/MS as a viable alternative to GC–MS/MS for routine analysis of pyrethroids in fruits and vegetables. This study contributes valuable insights into the efficacy of UHPLC-MS/MS as an alternative method to GC–MS/MS for multi-residue detection of pyrethroids in agricultural produce.

### 2.3. Supercritical fluid chromatography (SFC)

Supercritical fluid chromatography (SFC) stands at the forefront of modern analytical techniques, offering a powerful and versatile approach to separation and analysis. SFC utilizes supercritical fluids as the mobile phase, offering several advantages over traditional chromatographic methods. Unlike traditional chromatographic methods such as GC and LC, SFC operates in a supercritical state, where the fluid exhibits properties of both liquids and gases. This hybrid nature imparts distinct advantages, including enhanced kinetic performance, lower consumption of organic solvents, highly efficient chiral separation, and improved productivity at the preparative scale [88].

When coupled with the ion mobility quadrupole time-of-flight mass spectrometry (IM-Q-TOF/MS), this method can detect 20 pesticides including pyrethroids (fenpropathrin, etofenprox, bifenthrin, fenvalerate, cyhalothrin) in potato and yam with low limits of detection ranged from 0.7 to 6.0 ng/mL [89]. Another study by [90] compared the uses of supercritical fluid extraction (SFE) and microwave-assisted extraction (MAE) techniques to extract seven pyrethroids and their common metabolites, phenoxybenzyl alcohol from vegetable samples. The primary benefits of SFE stem from the characteristics of supercritical fluids, which are economical, free from contaminants, and more cost-effective to dispose of compared to organic solvents. MAE offers advantages in terms of shortened extraction time and reduced solvent usage, making it an environmentally friendly technique. The finding indicates both extraction methods have comparable extraction recoveries and suitable for rapid and efficient extraction of pyrethroids from vegetable samples. The use of supercritical fluid chromatography coupled with ultraviolet detection (SFC-UVD) for the separation and detection of pyrethroids in El-Saeid's study enables the detection of these compounds with limit of detections (LODs) ranging from 0.31 to 0.54 mg/L. This approach offers benefits such as heightened sensitivity, improved resolution, and reduced consumption of organic solvents.

In addition, the employment of polysaccharide-based columns in supercritical fluid chromatography coupled with mass spectrometry (SFC-ESI-MS/MS) has been reported to successfully identify λ-cyhalothrin, metalaxyl, and their enantiomers at a concentration level of 5 μg/kg [91]. Similarly, W. hua Zhang et al. [92] utilized supercritical fluid chromatography with photodiode array detection (SFC-PDA) to separate and identify fenpropathrin enantiomer residues in fruit and vegetable puree. By employing a mobile phase consisting of methanol and supercritical carbon dioxide, fenpropathrin was identified using a UV detector at a wavelength of 230 nm and quantified through the external standard method. This established method is capable of quantifying two fenpropathrin enantiomers at a LOQ of 0.2 mg/kg, with recoveries ranging from 80.6 to 105 %, and relative standard deviations (RSD) reaching 2.6–7.7 %. Shi et al. [89] employed supercritical fluid chromatography coupled with ion mobility quadrupole time-of-flight mass spectrometry (SFC-IM-Q-TOF/MS) for detecting multiple pesticide residues, including pyrethroids, in Chinese yam and potato. IM-Q-TOF/MS enhances detection accuracy by providing unique collision cross-section values, facilitating the differentiation of isomers and complex molecules, enabling the detection of fenpropathrin, etofenprox, bifenthrin, fenvalerate, and cyhalothrin at a LOD ranging from 0.7 to 6.0 ng/mL. In summary, SFC offers enhanced separation efficiency, including chiral pyrethroid enantiomers, rapid analysis, reduced solvent consumption, and environmental friendliness, making it a promising and versatile approach for the analysis of pesticide residues.

Murcia-Morales et al. [62] conducted a comprehensive comparative study evaluating the performance of SFC-MS/MS and GC-MS/MS for the analysis of pyrethroids in fruits and vegetable matrices. Fourteen pyrethroids were intentionally introduced into 17 real fruit and vegetable extracts, and both methods successfully detected these compounds. The LOQs for SFC ranged from 2 to 100 μg/L, which is on par with the range observed for GC-MS/MS. This study offers important insights, showing that SFC could be a strong alternative to GC for analyzing pyrethroids and even suggests that SFC might be expanded for broader use in detecting multiple residues at once.

### 2.4. Micellar electrokinetic capillary chromatography (MECC)

Micellar electrokinetic capillary chromatography (MECC) is a powerful analytical technique that has seen significant methodological and instrumental developments over the years [93]. It is a type of capillary electrophoresis (CE) that utilizes micellar solutions of ionic surfactants to separate analytes based on differential partitioning and migration [94,95]. The technique is known for its ability to separate neutral as well as charged analytes, making it a versatile tool in analytical chemistry [95]. MECC has also been used for the enantio-separation of compounds using modified micellar electrokinetic chromatography [96]. In MECC, a surfactant, typically a micelle-forming agent, is added to the separation buffer to form micelles. These micelles solubilize hydrophobic compounds, allowing for their separation in a background electrolyte. The analyte is separated by its distinct distribution between two-phase systems: the mobile aqueous phase and the micellar pseudo-stationary phase [97].

Capillary electrophoresis (CE) has emerged as an appealing method for pesticide residue analysis, thanks to its high resolving power, minimal solvent consumption, and straightforward sample pretreatment. Despite these advantages, the utilization of CE is often underreported in comparison to other analytical separation techniques, primarily due to its perceived lower sensitivity. To overcome the shortcoming of CE, Yue et al. [98] developed a method that can extract pyrethroid pesticides from a capillary filled with background electrolyte (BGE) of reverse-flow micellar electrokinetic capillary chromatography (RF-MECC) in the headspace in-tube microextraction (HS-ITME) above the sample solution into the acceptor phase in the capillary. The extracted three pyrethroids

**Table 3**
Summary of the fluorescence-based detection method for pyrethroids in the agricultural field in the past 10 years.

| Fluorescence probe | Recognition element | Pyrethroids | Sample matrix | Limit of detections (LODs) | References |
|---|---|---|---|---|---|
| NAP | Hydrazine group | Deltamethrin | Celery | 2.23 μM | [106] |
| SiQDs@$Eu^{3+}$ | SiQDs | Deltamethrin | Lettuce | 0.68 μM | [107] |
| RDB, R6G, C6 | Cyclodextrin | Deltamethrin, fenvalerate, cyfluthrin, fenpropathrin | Apples, pears, and tomatoes | Deltamethrin: 0.024 mg/L; fenvalerate: 0.025 mg/L; cyfluthrin: 0.009 mg/L; fenpropathrin: 0.016 mg/L | [128] |
| Cu NCs | Biotemplate | Cypermethrin, λ -cyhalothrin | Tomato and bottle gourd | Cypermethrin: 27.06 nM; λ -cyhalothrin: 23.28 nM | [129] |
| PBC | PBC@ALB | Permethrin | Tomato, cucumber, and lettuce | 0.029 mg/L | [127] |
| PNMOF | – | λ-cyhalothrin | Apple, cabbage, and pear | 0.34 μg/L | [111] |
| FON | FON-Trp | Bifenthrin | Tea | 9.34 μg/kg | [130] |
| MO-CDs | – | Deltamethrin and fenvalerate | Cabbage, corn, and rice | Deltamethrin: 0.04 μM; fenvalerate: 0.26 μM | [126] |
| Ln-MOF | MIPs | Fenpropathrin, cypermethrin, bifenthrin, β-cyfluthrin, cyfluthrin, cyhalothrin, τ-fluvalinate, permethrin | Apple and spinach | 8 ng/mL | [115] |
| CDs | MIPs | λ-cyhalothrin | Tea, cucumber, apple | 0.61 μg/L | [109] |
| UCNPs | Antibodies | Fenpropathrin, cypermethrin, fenvalerate | Apple, pear, Chinese cabbage and cucumber | Fenpropathrin: 0.01 μg/L; cypermethrin: 0.015 μg/L; fenvalerate: 0.011 μg/L | [119] |
| $Eu^{3+}$ | MIPs | Fenvalerate | Brussels sprouts, cucumbers, and eggplants | 51.29 μg/mL | [131] |
| Cyclo-WW + Zn(II) QDs | Cyclo-WW + Zn(II) | λ-cyhalothrin | Tea | 2.9 μg/L | [132] |
| TGA@Mn–ZnS–QDs | – | Cypermethrin | Tomato, okra, pea, and spinach | 0.132 μg/mL | [125] |
| NRF | – | Fenpropathrin | *Myrica rubra*, grape, apple, mango, pear juices | 1.5 μg/L | [120] |
| RCDs | MIPs | λ-cyhalothrin | Tea, apple, grape | 0.89 μg/L | [110] |
| CCFs | MIPs | λ-cyhalothrin | Cabbage, cucumber, tea, sweet potato, and apple | 0.368 μg/L | [108] |
| UCNPs | MIPs | Deltamethrin | Grape, cabbage | 0.749 μg/L | [133] |
| Mn and Cu co-doped $ZnIn_2S_4$ QDs | MIPs | λ-cyhalothrin | Broccoli, potatoes, carrots, spinach, and tea | 0.246 μg/g | [134] |
| UCNPs | MIPs | α-cypermethrin | Apple, pear, and cabbage | 0.03 mg/L | [135] |
| POA | MIPs | Bifenthrin, cypermethrin, cyhalothrin, cyfluthrin, permethrin, deltamethrin, fenpropathrin, phenvalerate, phenothrin, tetramethrin | Mutton and beef | 7.5–17 ng/mL | [136] |
| S-doped CDs | MIPs | λ-cyhalothrin | Green tea, spinach | 0.5 μg/kg | [137] |
| FeSe-QDs | MIPs | Cyfluthrin | Fish | 1.0–1.3 μg/kg | [138] |
| QDs | MIPs | Cypermethrin | Fish | 1.2 μg/kg | [139] |
| Allyl fluorescein | MIPs | Cyhalothrin | Honey | 0.004 nM | [140] |
| $YVO_4$:$Eu^{3+}$ | MIPs | λ-Cyhalothrin | Chrysanthemum | 1.76 μM | [141] |
| $Fe_3O_4$/$SiO_2$-MPS | MIPs | λ-Cyhalothrin | Honey | 2.3 ng/mL | [142] |
| CdTe QDs | MIPs | Deltamethrin | Cabbage, leek, spinach, apple | 0.16 μg/mL | [143] |

NAP, 1,8-naphthalimide; SiQDs@$Eu^{3+}$, silicon quantum dots europium (III) ions complex; Cu NCs, copper nanoclusters; PBC@ALB, 3-hydroxy-2-(4-(piperidin-1-yl)phenyl)-4H-benzo[g]chromen-4-one-albumin complex; PNMOF, peptide nanodots-bridged metal–organic framework; FON-Trp, Fluorescent organic nanoparticles-L-tryptophan; MO-CDs, carbon dots derived from *Moringa oleifera*; cyclo-WW + Zn(II), cyclo-ditryptophan-Zinc (II) complex; TGA@Mn–ZnS–QDs, thioglycolic acid-caped Mn-doped ZnS quantum dots; NRF, non-covalent ratiometric fluorophore; CdTe QDs, Cadmium telluride quantum dots; $Fe_3O_4$/$SiO_2$-MPS, Iron oxide silica 3-(methacryloxyl) propyl trimethoxysilane; $YVO_4$:$Eu^{3+}$, Yttrium orthovanadate europium (III) nanoparticles; FeSe-QDs, Iron selenide quantum dots; S-doped CDs, sulfur-doped carbon dots; POA, 4-phenoxyaniline; UCNPs, upconversion nanoparticles; CCFs, Carbazole-conjugated frameworks; RCDs, red-emission carbon dots; TGA@Mn–ZnS–QDs, thioglycolic acid-caped Manganese-doped Zinc sulfide quantum dots; Mn and Cu co-doped $ZnIn_2S_4$ QDs, Manganese and copper co-doped zinc indium sulfide quantum dots; $Eu^{3+}$, europium (III) ions; Ln-MOF, lanthanide luminescent metal-organic framework; FON, fluorescent organic nanoparticle; RDB, rhodamine B; R6G, rhodamine 6G; C6, coumarin 6.

(cyfluthrin, fenpropathrin, tetramethrin) were then separated by CE. Under the optimized conditions, the enrichment factors for the three pyrethroids were 309, 133, and 288. The established method achieved good linearity, low limits of detection below 1.00 ng/mL, and good extraction repeatability with RSD below 7.83 %. The proposed method successfully analyzed the pyrethroids in fruit samples with acceptable recoveries and precisions.

### 2.5. Enzyme-linked immunosorbent assay (ELISA)

Enzyme-linked immunosorbent assay (ELISA) has emerged as a valuable tool for the detection and quantification of pesticides in various environmental and agricultural samples. ELISA offers high sensitivity and accuracy in determining pesticide residues, making it a preferred method for pesticide analysis [99]. The working principle of ELISA in pesticide detection involves the specific binding of pesticides (antigens) to antibodies immobilized on a solid surface, followed by the detection and quantification of this interaction through an enzymatic reaction.

Huang et al. [100] generated monoclonal antibodies (mAbs) against cypermethrin through mice immunization and cell fusion. The anti-cypermethrin mAbs were used to establish an indirect competitive immunosorbent assay (ic-ELISA) for detecting cypermethrin in vegetables. The ic-ELISA demonstrated an $IC_{50}$ of 2.49 ng/mL and an $IC_{10}$ of 0.40 ng/mL, exhibiting good selectivity against various pyrethroids. Furthermore, these mAbs were used to develop a colloidal gold lateral flow immunoassay (LFIA) with a cut-off value of 0.6 μg/mL and visual LOD of 0.3 μg/mL. Similarly, Xu et al. [101] synthesized a unique immunizing hapten to generate a highly selective mAb against fenpropathrin. Validated through ic-ELISA, these mAbs exhibited a strong affinity and selectivity, with a half-maximal inhibitory concentration of 31.05 μg/L and minimal cross-reactivity (<4.8 %) with pyrethroid analogs. This mAb was used to develop a fluorescence immunochromatographic assay (FICA) for detecting fenpropathrin in fruits and vegetables, demonstrating a LOD of 0.012 mg/kg, below the established maximum residue limit. Wei et al. [102] developed mAbs against fenvalerate and established an ic-ELISA for monitoring fenvalerate in six dark teas, achieving $IC_{50}$ of 29.12 ng/mL and cross-reaction rates with pyrethroid structural analogs below 0.6 %. Additionally, a latex microsphere immunochromatographic test strip was developed, with a LOD of 10.0 ng/mL, allowing for rapid and specific on-site fenvalerate detection. However, all these immunochromatographic assays are based on an indirect competitive detection principle, which may pose challenges in result interpretation.

Despite ELISA being regarded as one of the conventional methods for pesticide detection, it presents challenges in detecting small-molecule pesticides. One of the primary challenges is the unsuitability of ELISA for the detection of small molecules, as highlighted in the study by Liu et al. [103]. This limitation arises from the difficulty in synthesizing high-affinity antibodies for small pesticide molecules [104]. Additionally, the development of ELISA for small-molecule pesticides requires high selectivity and sensitivity, which can be challenging due to the inherent properties of these molecules. The study by Hua et al. [105] emphasizes that while ELISA is a sensitive screening method for detecting organophosphorus (OP) pesticides, it is dependent on a laboratory platform, involves a relatively long assay time, and requires several operational steps. Therefore, the conversion of the mAbs from the ELISA platform to the innovative lateral flow assay, as demonstrated by Refs. [100,101], provides an alternative for immunoassay-based detection of pyrethroids in agricultural commodities, offering additional merits such as rapidity and ease of use.

## 3. Innovative approaches for pyrethroids detection

### 3.1. Optical-based approaches

Optical-based detection methods represent a versatile array of techniques that leverage the interaction between light and matter for analytical purposes. The incorporation of optical-based techniques in detecting pyrethroid pesticide residues not only simplifies the analytical procedures but also improves the accuracy and dependability of outcomes. Diverse spectroscopic methods address specific analytical needs, including fluorescence spectroscopy (FL), ultraviolet–visible (UV–Vis) absorbance spectroscopy, chemiluminescence spectroscopy (CL), near-infrared (NIR) spectroscopy, surface-enhanced Raman spectroscopy (SERS), and surface plasmon resonance (SPR). Each of these techniques is customized to address distinct analytical needs in the comprehensive detection and quantification of pesticide residues in agricultural produce. Several optical-based techniques, including FL [106–112] and UV–Vis [113,114], have been reported to perform on-site analysis when integrated with smartphones or miniaturized devices. This integration allows for on-site and rapid analysis without the need for sophisticated instrumentation that would otherwise be required in a laboratory setting.

#### 3.1.1. Fluorescence spectroscopy (FL)

Fluorescence spectroscopy-based detection methods have emerged as a powerful tool for the analysis of pyrethroids in agricultural fields, offering considerable advantages in terms of sensitivity, selectivity, and versatility. With the development of advancing technologies, various kinds of materials have been widely employed for the fabrication of fluorescence sensing platforms, including semiconductor quantum dots, carbon dots, fluorescent dyes, metal-organic framework, fluorescent organic nanoparticles, luminescent lanthanide complex, and upconversion nanoparticles. Table 3 summarizes the fluorescence-based detection methods for pyrethroids in agricultural fields over the past 10 years and provides a comprehensive insight into recent advancements.

For fluorescence-based detection of pyrethroids, molecularly imprinted polymers (MIPs) are the most extensively studied recognition elements. Additionally, various other elements, including antibodies, cyclodextrins, fluorescent organic nanoparticles (FONs), cyclo-dipeptides-Zinc(II) complexes, and non-covalent ratiometric fluorophores (NRFs), have also been reported.

Molecularly imprinted polymers (MIPs) are synthetic materials known for their precise molecular recognition capabilities, achieved through in-situ co-polymerization of functional monomers around a template molecule. In a study by L. Bai et al. [115], a sol-gel

strategy was utilized to create an imprinting cavity on a lanthanide luminescent metal-organic framework (Ln-MOF) for detecting the pyrethroid metabolite, 3-phenoxybenzaldehyde (3-PBALD). This innovative approach, coupled with esterase hydrolysis, enabled the rapid detection of twelve pyrethroids in apple and spinach samples. By targeting the common metabolite of pyrethroids, this study demonstrates the potential of using a single type of MIPs for detecting multiple synthetic pyrethroids. Despite their broad applicability, MIPs face challenges including reproducibility issues [116], limited understanding of binding mechanisms [117], and difficulties in extracting template molecules [118].

The exceptional sensitivity and selectivity of antibodies, functioning as biological recognition elements, stem from their notably high equilibrium association constants, facilitating precise recognition of specific antigens. This capability has been leveraged through advancements in nanomaterials and nanotechnology to drive the development of sophisticated fluorescence-based immunosensors. In a recent study by L. Zhao et al. [119], a universal pyrethroid antibody was employed to construct a three-layer sandwich structure incorporating rare earth Upconversion nanomaterials (UCNPs) for detecting fenpropathrin, cypermethrin, and fenvalerate. This innovative sensor achieved impressive LODs of 0.01 μg/L, 0.015 μg/L, and 0.011 μg/L, respectively, for samples of apple, pear, Chinese cabbage, and cucumber. The sensor operates through a process involving competitive binding of the pyrethroid standard to capture probes, followed by the separation of resulting conjugates using an external magnetic field, and subsequent fluorescence detection employing upconversion nanoparticles. Validation of the method was conducted via high-performance liquid chromatography (HPLC), demonstrating good accuracy and enabling quantitative analysis of pyrethroids in food samples.

In addition to conventional recognition elements, Fang et al. [120] identified the non-covalent ratiometric fluorophore (NRF), which displays a dual-emitting phenomenon at 315 and 560 nm. This distinctive attribute enables NRF to function as a ratiometric indicator for the sensitive detection of fenvalerate, cyfluthrin, flucythrinate, cypermethrin, cyhalothrin, permethrin, and deltamethrin. Upon the introduction of these pyrethroids, the fluorescence intensities (FIs) at 315 nm experienced significant reduction, while those at 560 nm showed minor changes. Consequently, the ratios ($I_{560}/I_{315}$) of the FIs at 560 nm to those at 315 nm could be employed as an effective ratiometric indicator. Leveraging this discovery, the researchers devised a ratiometric fluorescence probe, which they applied to detect fenpropathrin in various fruit juices, achieving a LOD of 1.5 μg/L.

Quantum dots (QDs) and carbon dots (CDs) serve as cutting-edge nanomaterials in fluorescence probing, offering highly sensitive and precise detection capabilities. QDs possess intrinsic water solubility, exceptional resistance to degradation, heightened quantum efficiency, and broad absorption spectra, alongside uniform and narrow fluorescent emission, rendering them ideal for crafting fluorescence-based chemosensors [121–123]. Likewise, CDs possess unique attributes such as low toxicity, biocompatibility, high photostability, and ease of surface modification, making them a promising material for developing fluorescence sensors [124]. Utilizing the Gonzalez method, Muhammad et al. [125] synthesized thioglycolic acid-capped Mn-doped ZnS quantum dots (TGA@Mn-ZnS-QDs) for detecting cypermethrin in tomato, okra, pea, and spinach, achieving a LOD of 0.132 μg/mL. Similarly, Yusuf Vadia et al. [126] developed green-fluorescent carbon dots derived from *Moringa oleifera* (MO-CDs) utilizing a quenching mechanism for detecting deltamethrin and fenvalerate in vegetable and rice samples, with LODs of 0.040 and 0.26 μM, respectively. Although both sensors exhibited no significant interference when tested with other pesticides, their selectivity towards other pyrethroid members remains uncertain due to the absence of specific recognition elements in their design.

In addition to conventional fluorescence recognition elements, Y. Zhao et al. [127] introduced an intriguing fluorescent dye-protein complex known as 3-hydroxy-2-(4-(piperidin-1-yl)phenyl)-4H-benzo[g]chromen-4-one and albumin (PBC@ALB) complex. This complex serves as a novel host-guest supramolecular recognition system for detecting permethrin in tomatoes, cucumbers, and lettuce. The emission of PBC denoted as T*, is responsive to environmental polarity. Upon the addition of permethrin, the polarity of ALB's binding pocket decreases, resulting in heightened T* emission, which facilitates the construction of a sensing mechanism. This sensor exhibits a detection limit of 0.08 μM (29 ppb), meeting the standards for common foods. Additionally, M. Li et al. [128] developed sensor arrays comprising three nanocomposite complexes (rhodamine B-CD@Au, rhodamine 6G-CD@Au, and coumarin 6-CD@Au) to differentiate deltamethrin, fenvalerate, cyfluthrin, and fenpropathrin in apple, pear, and tomato. Due to the non-linear relationship between fluorescence signal and analyte concentration, a support vector machine (SVM) machine learning technique was employed to aid in the identification and quantification of the four pyrethroids. In upcoming times, paper-based tag arrays integrated with smartphones and utilizing machine learning will emerge as the favored method for rapidly and non-destructive detecting various pyrethroid pesticides in real-world samples.

When considering practical applications, integrating an established fluorescence detection system into a portable platform becomes essential for the sake of convenience and real-time pesticide monitoring. This integration facilitates swift detection and response to potential contamination in various environments, including agricultural fields and food processing facilities. For instance, X. Zhu, Han et al. [132] devised an innovative ratiometric fluorescence core-shell nanosphere using blue-green dual-emission carbon dots (CDs) embedded in molecularly imprinted polymers (MIPs) as the recognition element. These core-shell nanospheres demonstrated fluorescence alterations upon exposure to pyrethroids under UV irradiation. Furthermore, the research team developed a smartphone application to analyze the color channel values of captured images, enabling sensitive quantification of pyrethroids with a LOD of 0.61 μg/L in tea, cucumber, and apple samples. Similarly, Y. Zhang et al. [111] identified a significant fluorescence quenching phenomenon in a peptide nanodots-bridged metal–organic framework (PNMOF) fluorescence probe upon the addition of λ-cyhalothrin. This discovery led to the development of a detection system incorporating a smartphone-facilitated cascade amplification sensing platform, which successfully detected λ-cyhalothrin with a LOD of 2.4 μg/L in apple, cabbage, and pear samples. The integration of smartphones into fluorescence sensing demonstrates a promising direction for on-site detection of pyrethroid residues in agricultural fields.

### *3.1.2. Ultraviolet–visible absorbance spectroscopy (UV–Vis)*

The UV–Vis-based or colorimetric approach has emerged as a pivotal methodology in the field of pesticide residue detection,

offering a rapid and visually intuitive means to identify and quantify these contaminants. This technique relies on the specific interaction between a bioreceptor and the analyte, resulting in discernible color changes that can be observed with the naked eye or measured instrumentally using a UV–Vis spectrophotometer. The simplicity and speed of colorimetric assays make them particularly attractive for on-site and field applications, where quick and reliable detection is essential.

Noble metal nanoparticles, such as copper [144], gold, and silver [145], have emerged as powerful tools in analytical chemistry, particularly for colorimetric detection of pesticide residues. Endowed with localized surface plasmon resonance (LSPR) properties, these nanoparticles exhibit unique optical characteristics that are highly effective in sensing applications. LSPR is initiated by the collective resonance oscillation of conduction band electrons upon exposure to visible light, a phenomenon prominently observed in nanoscale particles of gold, silver, or copper, leading to notable absorbance peaks in UV–visible spectrophotometry. The characteristics of this absorbance, including its intensity and wavelength, depend on factors such as the size, morphology, and elemental composition of the material being studied [146]. Moreover, the spacing between metal nanoparticles plays a crucial role in determining their LSPR behavior, thus rendering them highly effective for rapid, cost-efficient, and on-site colorimetric sensing of various analytes [147].

Rasheed et al. [113] synthesized metronidazole-stabilized silver nanoparticles (MTZ-AgNPs) to enable the colorimetric detection of permethrin in tomatoes and apples. The synthesized MTZ-AgNPs, characterized by a localized surface plasmon resonance (LSPR) absorbance peak at 400 nm, initially exhibit a yellow hue, which shifts to pale yellow upon the aggregation of nanoparticles following the addition of permethrin. Integration of this visual color change with smartphone-based and microfluidic paper-based analytical devices facilitated on-site detection of permethrin, achieving a LOD of 0.0104 μM. Although the sensor's selectivity was evaluated against other pesticides, such as bifenthrin, carbendazim, chlorpyrifos, chlorothalonil, isoproturon, and imidacloprid, questions remain regarding the reliability of the detection mechanism, which relies on the interaction between the free hydroxyl groups of metronidazole and permethrin.

Moreover, J. Zhu et al. [114] pioneered the development of a paper-based colorimetric sensor tailored for detecting deltamethrin pesticide. This sensor employed 2-mercapto-6-nitrobenzothiazole (MNBT) functionalized AuNPs as a colorimetric probe. The π-π stacking interaction between deltamethrin and MNBT was confirmed through Fourier-transform infrared spectroscopy (FTIR). Upon the introduction of deltamethrin, the MNBT-AuNPs probe undergoes aggregation, resulting in a color shift from red to grey-purple. The intensity of the resultant color is directly correlated to the concentration of deltamethrin. Notably, this sensor demonstrates exceptional sensitivity, detecting deltamethrin in apples, mandarin oranges, spinach, tomato, and cucumber with a remarkable LOD of 0.173 mg/L.

On the contrary, utilizing aptamers as biorecognition elements for colorimetric detection of pyrethroids offers superior integrity for the specific identification of the analyte. Capitalizing on their inherent merits, such as thermal stability, cost-effectiveness, versatile applications, chemical robustness, ease of modification, and high stability [148,149], Yang et al. [150] isolated a single-stranded DNA aptamer named LCT-1 and a truncated aptamer named LCT-1-39, both exhibiting specificity towards λ-cyhalothrin. These aptamers were employed in the development of a gold nanoparticle (AuNP)-based colorimetric aptasensor. The sensor comprises three key components: citrate-capped AuNPs, a cationic polymer polydiene dimethyl ammonium chloride (PDDA), and a single-stranded DNA aptamer as the recognition element. The detection mechanism relies on the aggregation of AuNPs induced by cationic polymers as the sensing signals. Upon the addition of λ-cyhalothrin, it binds to the aptamer, liberating PDDA from the aptamer-PDDA complex in its native state, thereby causing a color change from red to blue-violet or even blue. The fabricated sensor demonstrates a LOD of 0.0197 μg/mL (LCT-1) and 0.0186 μg/mL (LCT-1-39) and successfully detects λ-cyhalothrin in pear and cucumber samples.

### *3.1.3. Chemiluminescence spectroscopy (CL)*

Chemiluminescence spectroscopy, a valuable analytical method utilized across various scientific fields, holds significant importance in detecting residues of pyrethroid pesticides in agricultural products. This technique functions by exploiting the emission of light resulting from chemical reactions for compound analysis. The process entails initiating a chemical reaction between a target analyte and a specific reagent, producing light in correlation with the analyte's concentration. Subsequently, this emitted light is captured and measured, offering insights into the presence and concentration of the desired compound. Chemiluminescence presents advantages over fluorescence and absorbance spectroscopy, as its measurement differs from the ratio measurements employed in fluorescence and absorption techniques. In fluorescence, this disparity can pose challenges, particularly with fluorescers exhibiting a small Stokes shift, where resolving fluorescence from the exciting wavelength may prove difficult [151]. Additionally, issues arise from the scattering of incident light reaching the detector, particularly in samples with some turbidity. The primary limitation to sensitivity in absorption measurement stems from the necessity to discern a slight difference between two relatively large signals.

Taheri et al. [152] devised an indirect competitive chemiluminescent enzyme immunoassay (CLEIA) utilizing a novel broad-specific monoclonal antibody for identifying fenpropathrin, deltamethrin, and λ-cyhalothrin residues in orange, eggplant, and cowpea samples. This immunoassay protocol closely resembles the conventional indirect competitive enzyme-linked immunosorbent assay (ic-ELISA), differing only in the substrate employed; here, chemiluminometric signals are produced from the HRP-luminol-$H_2O_2$ system rather than the chromogen used in ELISA. The developed assay exhibits noteworthy sensitivities toward fenpropathrin, deltamethrin, and λ-cyhalothrin, with $IC_{50}$ values of 1.9, 3.4, and 4.3 ng/mL, respectively, along with minimal cross-reactivity (5 %) towards cypermethrin when tested against other pyrethroid compounds. Furthermore, the assay's analysis of several real samples was authenticated by a gas chromatography-electron capture detector (GC-ECD), demonstrating a strong correlation between the two methodologies.

In addition to antibodies, J. J. Huang et al. [153] developed a chemiluminescence sensor using a dual-dummy-template MIP capable of recognizing 10 pyrethroids on conventional 96-well microplates. The chemiluminescence assay generates a signal based on the bis(2,4,6-trichlorophenyl)oxalate–$H_2O_2$–imidazole system, demonstrating high sensitivity in the detection of pyrethroids in

chicken muscle samples with LOD ranging from 0.3 to 6.0 pg/mL. Additionally, this sensor requires a short assay time (12 min) and is reusable up to four times, showcasing its promising application in the detection of pyrethroid residues.

### 3.1.4. Near-infrared spectroscopy (NIRS)

Near-infrared spectroscopy (NIRS) has emerged as a powerful analytical technique in various fields, showcasing its versatility and efficiency in non-destructive analysis. One particularly impactful application lies in the realm of pesticide detection, where NIRS has proven to be a valuable tool for rapid and reliable assessment. The near-infrared region of the electromagnetic spectrum, ranging from 700 to 2500 nm [154], is particularly informative for analyzing molecular vibrations and electronic transitions. This enables NIR to provide detailed information about the chemical composition of a sample. In the context of pesticide detection, the technique exploits the unique spectral signatures associated with specific functional groups in pesticide molecules. By illuminating a sample with near-infrared light and analyzing the resulting absorption or reflection spectra, NIR allows for the identification and quantification of pesticides in a variety of matrices, including crops [155], soil [156], and water [157].

Ishkandar et al. [158] utilized a visible shortwave near-infrared (VSNIR) spectrometer to assess deltamethrin residue in cabbage, subsequently determining deltamethrin concentration through partial least square regression (PLSR) analysis of the collected VSNIR spectral data. The study encompassed an adequate sample size, comprising sixty organic cabbages categorized into control (no deltamethrin) and low, medium, and high concentrations of deltamethrin. To enhance the study's credibility, a gas chromatography-electron capture detector (GC-ECD) served as the reference method for deltamethrin concentration determination. Despite achieving commendable results with $R^2$ values of 0.98 and 0.94 for calibration and prediction models, respectively, the authors advocate for expanding the scope by including additional cabbage sample replicates sourced from diverse farms with varying agronomic practices and spanning multiple harvest seasons. This proposed extension aims to refine the model, positioning the method as a robust and promising technique for detecting pesticide residues at different concentrations in cabbage samples.

In a recent study conducted by G. Yu et al. [159] visible/near-infrared (Vis/NIR) spectroscopy, in conjunction with the multiscale Deep spectra network, was employed to detect λ-cyhalothrin and β-cypermethrin residues in Hami melon, resulting in noteworthy $R^2$ values of 0.758 and 0.835, respectively. The application of the multiscale Deep spectra network for analyzing the acquired Vis/NIR spectra demonstrated superior performance when compared to other chemometric methods such as partial least square regression (PLSR) and support vector regression (SVR), establishing it as the most effective among the regression models. However, the study acknowledges that there is room for improvement in the repeatability and adaptability of the established multiscale Deep spectra network model. Addressing these aspects could further enhance the reliability and applicability of the method in detecting pesticide residues in Hami melons, contributing to the continued development of advanced techniques for food safety assessments.

### 3.1.5. Surface-enhanced Raman spectroscopy (SERS)

Surface-enhanced Raman spectroscopy (SERS) has emerged as a powerful and sensitive analytical technique with diverse applications, and one particularly promising field is the detection of pesticide residues. Recent technological advancements have led to the development of alternative detection techniques, such as SERS, which provide added advantages such as ultrasensitive detection, faster turnover, simpler protocols, *in situ* sampling, on-site capability, and reduced cost [160]. SERS allows for the rapid and sensitive detection of pesticide residues on food surfaces, enabling quantitative measurement without the need for sample pretreatment [161]. Furthermore, SERS has been employed for the simultaneous detection of multiple pesticide residues, providing a rapid and straightforward method for multi-residue analysis [162]. The use of SERS in combination with nanomaterials, such as gold nanoparticles, silver nanoparticles-plated-zinc oxide nanoflowers, silver nanorods, silver nanoparticles, and silica-titanium dioxide-silver composites has further enhanced its detection capabilities, allowing for the detection of specific pyrethroids with high-performance substrates [75,162–166].

SERS operates on the principle of enhancing Raman signals through the excitation of localized surface plasmon resonances in noble-metal nanostructures, leading to a significant amplification and localization of the incident electromagnetic field [167]. Surface-enhanced Raman Spectroscopy (SERS) involves two concurrent mechanisms: electromagnetic (EM) enhancement, influenced by the local electric field increase near nanoparticles through surface plasmon resonance (LSPR), and chemical (CM) enhancement, resulting from charge-transfer interactions between molecules and nanoparticle surfaces. The CM depends on the specific Raman-active molecule and its interaction with nanoparticles, while the EM is influenced by surface plasmon resonance. Together, these mechanisms synergistically enhance the overall Raman signal in SERS [168].

R. Hou et al. [162] introduced a Surface-Enhanced Raman Spectroscopy (SERS) technique for the identification of various insecticides, including deltamethrin, in tea leaves and apples. The SERS analysis employed citrate-capped gold nanoparticles (AuNPs) as the substrate. Spectral data were subjected to principal component analysis (PCA), and a partial least squares (PLS) model was established. This method successfully detected deltamethrin with a LOD of 0.02 μg/cm on the surfaces of both tea leaves and apple peels, *in situ*. Similarly, H. Zhang et al. [169] utilized AuNPs as a SERS substrate to detect deltamethrin in *Corydalis*, a Chinese medicinal material. They optimized the Quick, Easy, Cheap, Effective, Rugged, and Safe (QuEChERS) method, incorporating multi-walled carbon nanotubes (MWCNTs) as dispersive solid-phase extraction sorbents to prepare the sample for SERS analysis, achieving a LOD of 0.484 mg/L. Both SERS techniques provide a direct, rapid, and highly sensitive approach to monitor the presence of these insecticides on plant surfaces, ensuring the safe production of commercial tea, fruits, and herbs.

In their respective studies, Hidayah et al. [170] and Dong et al. [163] investigated the application of silver and gold nanoparticles as substrates to enhance the SERS signal for detecting deltamethrin in brewed tea and strawberries, respectively. Dong et al. [163] employed multiplicative scatter correction (MSC) for spectral treatment, followed by partial least squares (PLS) and backward interval partial least squares (BIPLS) modeling, successfully detecting deltamethrin in strawberries with a LOD of 0.1 mg/L. They compared the

enhancement effects of AuNPs and AgNPs, concluding that AuNPs exhibited more uniform particle size, concentrated distribution, closer agglomeration state, and better permittivity of the surrounding medium than AgNPs, resulting in higher detection sensitivity. On the other hand, Hidayah et al. [170] compared AuNPs, AgNPs, and Au-Ag nanoalloys, determining that AgNPs outperformed the others as Raman signal enhancers. They successfully detected deltamethrin in tea with a LOD of 0.01 mg/L, despite encountering inconsistencies in the enhancement of Raman signals due to the Brownian motion of the liquid SERS substrate.

Furthermore, in a study conducted by Jiao et al. [164], silver nanoparticles-plated-zinc oxide nanoflowers (Ag@ZnO NFs) were utilized as a Raman signal enhancer for the quantification of deltamethrin in wheat, achieving a LOD of 0.16 μg/kg. The researchers employed the mean centering (MC) coupled successive projection algorithm-partial least squares regression (SPA-PLS) to establish optimal detective performance based on the obtained Surface-Enhanced Raman Spectroscopy (SERS) spectra. In a separate study by S. Zhang et al. [171] a flexible SERS substrate composed of Ag nanoparticle-coated bacterial nanocellulose (AgNP@BNC) was employed to detect λ-cyhalothrin on apple surfaces. The synthesis of the AgNP@BNC SERS substrate was accomplished through a straightforward, cost-effective, efficient, and scalable magnetron sputtering technology. The optimized AgNP@BNC SERS substrate demonstrated highly sensitive pesticide detection, with a detection limit of $7.8 \times 10^{-8}$ M for λ-cyhalothrin on apple surfaces. In conclusion, SERS stands out as a powerful and versatile analytical technique, offering ultrasensitive and rapid detection of pyrethroid residues on various surfaces through recent technological advancements and the application of nanomaterials.

#### *3.1.6. Surface plasmon resonance (SPR)*

Surface Plasmon Resonance (SPR) emerge as a promising technique for pesticide residue detection, capitalizing on its capacity for real-time and label-free analysis through the interaction of target molecules with a sensor surface [172]. However, there has been limited exploration of SPR sensors for pyrethroid residue detection in agricultural products, mainly due to the small molecular size of pyrethroid pesticides, which makes it challenging to induce significant changes in the refractive index upon binding to the sensor surface. X. Liu et al. [173] introduced an innovative approach that integrates SPR sensor technology with $Fe_3O_4$ magnetic nanoparticles (MNPs) assays for the direct detection of deltamethrin in soybeans. Deltamethrin-specific antibodies were linked to the $Fe_3O_4$ MNPs, acting as both labels to enhance the refractive index change upon capturing the target analyte and "vehicles" to transport the analyte from the sample matrix to the sensor surface. This strategy successfully detects deltamethrin with a detection limit of 0.01 ng/mL, improving the direct SPR detection format by four orders of magnitude. The developed sensor's selectivity was also assessed using fenvalerate and atrazine, demonstrating no significant interference with deltamethrin detection. This innovative detection strategy exemplifies the SPR sensor's potential to advance precision for monitoring the safety of agricultural products.

### *3.2. Electrochemical-based approaches*

Electrochemical-based detection methods present a promising avenue for analyzing pyrethroids in agricultural settings, owing to their inherent advantages in sensitivity, selectivity, and ease of operation. These techniques capitalize on the electrochemical characteristics of pyrethroids, exploiting phenomena like redox reactions or alterations in charge transfer to precisely detect and quantify these compounds. The fundamental principle underlying electrochemical detection entails the conversion of chemical information into an electrical signal, which can be conveniently measured and interpreted. In comparison to conventional analytical approaches, electrochemical methods typically offer swifter analysis times, necessitate minimal sample preparation, and can be seamlessly adapted for on-site analysis, rendering them especially well-suited for agricultural applications. Over the past decade, researchers have made significant strides in developing various recognition elements, electrochemical nanomaterial modifiers, and innovative

**Table 4**
Summary of the electrochemical-based detection method for pyrethroids in the agricultural field in the past 10 years.

| Electrochemical technique | Nanomaterial Modifier | Recognition element | Pyrethroids | Sample matrix | Limit of detections (LODs) | References |
|---|---|---|---|---|---|---|
| CV | AuNPs@ZIF-67 | Antibody | Fenpropathrin, deltamethrin | Lettuce, baby cabbage | 0.0258 nM, 1.712 nM | [174] |
| CV | CS@AgNWs | Haemoglobin | α-cypermethrin | Chilli | 14 nM | [176] |
| SWV | – | – | β-cyfluthrin | Lemongrass tea, chamomile tea | – | [180] |
| CV | MOF-5 | Antibodies | Bifenthrin | Okra, brinjal, capsicum | 4 ng/L | [175] |
| CV, CA | – | MIP | Cypermethrin | Kale, cucumber, carrot, coriander, and cilantro juices | 0.015 mg/L | [181] |
| CV | Ag-N@ZnO/CHAC | MIP | Cypermethrin | Mackerel, crayfish | $6.7 \times 10^{-14}$ M | [179] |
| DPV | – | MIP | Deltamethrin, fenvalerate, fenpropathrin, cyhalothrin | Tangerine, Gong orange, and Ponkan | 0.01–0.30 mg/kg | [182] |
| CV | – | GST (enzyme) | Cypermethrin | Tomato | 0.002 mg/L | [177] |

CV, cyclic voltammetry; AuNPs@ZIF-67, metal-organic framework loaded with gold nanoparticles; CS@AgNWs chitosan-silver nanowire nanocomposite; SWV, Square wave voltammetry; MOF-5, Metal-organic framework; MIP, molecularly imprinted polymer; CA, chronoamperometry; EIS, electrochemical impedance spectroscopy; Ag-N@ZnO/CHAC, silver and nitrogen co-doped zinc oxide activated carbon; GST, glutathione-s-transferase; DPV, differential pulse voltammetry.

electrochemical techniques aimed at enhancing the detection performance of pyrethroid pesticides in agricultural commodities, as evidenced by recent advancements summarized in Table 4.

A reliable recognition element for an electrochemical sensor typically exhibits several crucial characteristics, including selectivity, sensitivity, stability, affinity, compatibility, regenerability, and cost-effectiveness. As indicated in Table 4, over the past decade, four types of recognition elements have been utilized for the detection of pyrethroids. These encompass antibodies, transport proteins, MIPs, and enzymes. Xiang et al. [174] and Chansi et al. [175] demonstrated the efficacy of antibodies as recognition elements for pyrethroids in electrochemical sensing. Xiang et al. [174] utilized monoclonal antibodies to selectively detect specific members of pyrethroids, whereas Chansi et al. [175] employed polyclonal antibodies, enabling the detection of a wide range of pesticides albeit with compromised selectivity towards individual analytes. In a notable departure, Bhandari et al. [176] employed hemoglobin, a transport protein, to facilitate electrochemical signal generation between α-cypermethrin and the oxygen-containing hemoglobin, presenting a rare recognition approach in the literature. Similarly surprising, Borah et al. [177] utilized glutathione-S-transferase (GST), an enzyme, to detect cypermethrin. This novel approach exploited the distinctive effect of cypermethrin on the GST-catalyzed reaction between glutathione (GSH) and 1-chloro-2,4-dinitrobenzene (CDNB) in methanol. Additionally, MIPs have become the most extensively studied recognition elements for the electrochemical detection of pyrethroids, owing to their well-established synthesis protocols and high selectivity towards target analytes, which has led to their widespread use in this field.

An electrode, serving as a transducer, stands as a pivotal element within an electrochemical sensor. Enhancing electrodes with nanomaterial modifiers plays a crucial role in refining sensor performance, enhancing electron transfer rates, sensitivity, selectivity, and stability [178]. For instance, Y. Li et al. [179] employed the sol-gel method to fabricate silver and nitrogen co-doped zinc oxide (Ag-N@ZnO), subsequently loading it onto activated carbon derived from coconut husk to create Ag-N@ZnO/CHAC. The impact of this modification was assessed via electrochemical impedance spectroscopy (EIS), revealing a decrease in resistance from 78 Ω (for the ZnO-modified electrode) to 64 Ω (for the Ag-N@ZnO/CHAC glassy carbon electrode), indicating an expanded surface area and improved electron transfer rate. Furthermore, the utilization of metal-organic framework (MOF) as an electrode modifier has been demonstrated by Chansi et al. [175] and Xiang et al. [174]. Chansi et al. [175] synthesized a metal-organic framework (MOF-5) through chemical means using zinc acetate and terephthalic acid, offering a well-defined crystal structure with available functional groups to enhance bioconjugation and adsorption. Xiang et al. [174] chemically synthesized ZIF-67 (a MOF) characterized by a regular dodecahedron structure and a large specific surface area suitable for the conjugation of gold-labeled antibodies. In conclusion, the strategic incorporation of nanomaterial modifiers, including metal-organic frameworks, holds immense promise in advancing the capabilities and performance of electrochemical sensors.

Electrochemical-based approaches are generally less reported than optical-based approaches for the detection of pyrethroid residues in food samples. In terms of sensitivity, comparing electrochemical and optical-based methods is challenging because different studies have demonstrated varying LOD due to the diverse range of food matrices analyzed. Nevertheless, a general conclusion can be drawn for both approaches, as summarized in Table 5.

## 4. Conclusion and perspectives

Conventional analytical techniques, such as GC, LC, SFC, and MECC, coupled with various detectors, have demonstrated effectiveness in detecting trace amounts of pyrethroid residues and identifying pyrethroid types, including enantiomers. However, to improve pyrethroid recovery, especially in complex food matrices, future research should prioritize optimizing sample preparation processes. Developing tailored sample extraction and clean-up methods specifically designed for different types of agricultural produce is crucial for ensuring comprehensive food safety and accommodating a broader range of foods containing pyrethroid residues.

While ELISA is typically performed in laboratory settings, there is a need to explore the development of portable

**Table 5**
Comparison of electrochemical sensors and optical-based methods for the detection of pesticide residues in agricultural commodities.

| Criteria | Electrochemical-based detection | Optical-based detection |
|---|---|---|
| Sensitivity | High sensitivity; can detect low concentrations (nanomolar or lower). | High sensitivity; varies depending on the technique used. |
| Specificity | Depends on the type of recognition elements and electrochemical technique used to induce specific redox reactions. | Depending on the type of recognition elements; techniques such as SERS & NIRS can provide molecular fingerprints. |
| Portability | Portability relies on miniaturized potentiostats or handheld devices. | Portability can be achieved by integrating with a smartphone for FL and UV–Vis, while other techniques require more sophisticated equipment. |
| Response Time | Rapid response, often within seconds to minutes. | Rapid and real-time analysis capability. |
| Interference | Susceptible to interference from other electroactive substances. | Susceptible to interference from background fluorescence or light. |
| Stability | Limited stability; biological recognition elements and nanomaterial modifiers may degrade over time. | Depends on the type of fluorescence probe, plasmonic nanoparticles, chemiluminescence substrates, and SERS substrates. |
| Calibration | Requires frequent calibration for accurate results. | Calibration is less frequent but may require complex calibration in certain techniques. |
| Sample Preparation | Simple sample preparation. | May require complex preparation to reduce matrix effects. |
| Environmental Sensitivity | Less sensitive to environmental changes. | Sensitive to environmental factors such as light, temperature, and humidity. |

immunochromatographic assays that utilize mAbs validated through ELISA for field applications. Given that these assays often rely on an indirect competitive format with mAbs as the biorecognition element, future research should focus on innovative assay designs that enhance the color contrast between positive and negative samples, simplifying the quantification of pyrethroids in field conditions.

Innovative approaches are emerging that address the limitations of conventional methods, offering quicker, more cost-effective, and user-friendly detection solutions in agricultural settings. The effectiveness of these innovative techniques largely depends on the stability and specificity of the recognition units—such as enzymes, antibodies, and aptamers—which can be influenced by environmental factors like temperature and pH. To ensure reliable on-site analysis, future research should aim at developing engineered recognition units that maintain their stability under varying environmental conditions, thereby overcoming the current limitations.

Fluorescence-based methods hold significant potential for further development, particularly in the design of signaling probes, including fluorophores, semiconductor quantum dots, carbon dots, and metal-organic frameworks. Similarly, colorimetric methods could benefit from exploring noble metal nanoparticles with distinct LSPR properties, which could serve as indicators of pyrethroid concentration in samples. Both fluorescence and colorimetric approaches are well-suited for integration with smartphone-based sensing platforms, offering straightforward and easily interpretable results, even visually.

Although electrochemical methods are currently less popular compared to optical methods, their potential for practical application in detecting pyrethroid residues in agricultural produce should not be overlooked. Future studies could focus on integrating electrochemical sensors with smartphones by coupling them with miniaturized potentiostats, facilitating field analysis in various settings.

In the foreseeable future, machine learning (ML) could be employed to interpret complex data generated by innovative techniques such as NIRS and SERS, enhancing the accuracy and efficiency of pyrethroid pesticide residue detection. Concurrently, integrating the Internet of Things (IoT) could enable real-time monitoring and data sharing among various stakeholders, including farmers, consumers, and regulatory authorities. This connectivity would support data-driven decision-making in pesticide management, promote supply chain transparency, and streamline regulatory compliance and reporting processes, ultimately ensuring the safety and quality of agricultural produce.

## CRediT authorship contribution statement

**Dirong Goh:** Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Formal analysis, Data curation, Conceptualization. **Ahmad Faizal Abdull Razis:** Supervision, Project administration, Funding acquisition, Conceptualization. **Nor Azah Yusof:** Supervision. **Norida Mazlan:** Supervision. **Noordiana Nordin:** Supervision. **Choo Yee Yu:** Writing – review & editing, Supervision.

## Data availability statement

This manuscript is a literature review and does not involve the generation or analysis of primary data. All data supporting the findings of this study are derived from publicly available resources, which have been appropriately cited within the text.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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