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Extraction and determination of flubendiamide insecticide in food samples: A review

S. Aghris [a], O. Tahiri Alaoui [b], F. Laghrib [a,c], A. Farahi [a], M. Bakasse [a,d], S. Saqrane [a], S. Lahrich [a], M.A. El Mhammedi [a,*]

[a] Sultan Moulay Slimane University, Laboratory of Materials Science, Mathematics and Environment, Polydisciplinary Faculty, Khouribga, Morocco
[b] Moulay Ismail University, Laboratory of Physical Chemistry, Materials and Environment, Sciences and Technologies Faculty, Errachidia, Morocco
[c] Sidi Mohamed Ben Abdellah University, Engineering Laboratory of Organometallic, Molecular Materials, and Environment, Faculty of sciences, Fes, Morocco
[d] Chouaib Doukkali University, Organic Micropollutants Analysis Team, Faculty of Sciences, Morocco

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ABSTRACT

Flubendiamide (FBD) is the first commercially available phthalic acid diamide that targets ryanodine receptors (RyRs) in insects, which play a major role in lepidoptera control. However, excessive use of FBD can influence the quality of treated products leading to toxic effects on human health. The availability of rapid and convenient methods for evaluating FBD amount in the environment is necessary. Therefore, analytical methods were developed for the determination of residues of FBD and its metabolite desiodo in different food matrices like tomato, cabbage, pigeon pea, apple, chilli and rice. The current review carries forward methods for FBD residues analysis in foods by using several chromatographic techniques including sample preparation steps. The comparison between the different methods employed for quantitative and qualitative analysis of food quality and safety is also discussed. Liquid chromatography (LC) is the predominant analytical method for assessing the quality of foods treated with FBD. Studies related to LC coupled multichannel detector (Ultraviolet (UV), Mass spectrometry (MS)) are also applied to detect pesticide residues. Extraction and clean up steps are essential to obtain reliable results. Moreover, this review reports the allowed limits of residues for the safety of consuming products treated with FBD.

1. Introduction

Pesticides are substances or mixtures of compounds that are used to manage pests including insects, weeds, mammals, and microbes (Percival and Schroeder, 2017). These chemical substances can come from variety of natural sources such as animals, plants, and bacteria. Approximately, 5.2 billion pounds of pesticides are used worldwide each year to control pests and diseases in many fruit and vegetable crops (Mahmood et al., 2016). Their use is not only limited to agricultural crops, but they are also used in households to control mosquitoes, rats, fleas, ticks and other insect pests (Olszewski et al., 2010). For this reason, pesticides are often found in our food products in addition to their presence in the environment. However, pesticides are among the most dangerous chemicals, with their stability and mobility in the environment (Damalas and Eleftherohorinos, 2011). Thus, the successive use of pesticides in crops can cause intense risks on biodiversity (Tossou et al., 2019).

There are different classes of pesticides organized according to their application, including: i) herbicides, used to control weeds and other plants; ii) fungicides, used to control fungi; and iii) insecticides, which play an important role in the control of insect pests (Özkara et al., 2016).

The majority of pesticides are used as insecticides to control a wide variety of insects. These are classified into different categories, such as cholinesterase inhibitors (organophosphates and carbamates), pyrethroids, neonicotinoids, and ryanoids (Ishaaya, 2012). The latter category of insecticides includes several chemical compounds such as FBD, known as diamide insecticides (Caballero et al., 2013; Sharma et al., 2019; Gill and Garg, 2014). FBD is one of the most widely used insecticides on crops, vegetables, and fruits because of its exceptional pest control effectiveness, extremely high intrinsic potency, remarkable selectivity, minimal ecotoxicity, and low residual levels (Secretariat and Center, 2003; Lehotay et al., 2005). However, due to its widespread use, environmental risks and food safety have become an important concern. Therefore, it is important to develop rapid and practical methods for the

* Corresponding author.
E-mail address: elmhammedi@yahoo.fr (M.A. El Mhammedi).

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determination of FBD residues in food products. However, the detection of FBD residues in foods is quite difficult due to the presence of a large amount of interferences in the matrix and also for the low concentrations of pesticide residues in the food samples. Therefore, extraction and cleaning techniques are needed first to prepare the food matrix. This review presents a general approach on insecticidal diamides considering FBD as an example. Extraction and clean-up methods used in the literature for the preparation of the food matrix treated with FBD were reviewed. Moreover, emphasis was put on the most recent research reports regarding the determination of FBD's residues and it's dissipation in different food matrices.

2. Ryanodine receptor-targeting insecticides

Ryanodine receptor-targeting insecticides (Ryanoids) are a novel class of insecticides (Coronado et al., 1994; Fill and Copello, 2002; Lahm et al., 2005, 2007, 2009; Cordova et al., 2006; Li et al., 2012, 2020; Pawar and Bhilave, 2020). This type of insecticide is naturally extracted from tropical South American plant (Ryania speciosa Vahl) (Rogers et al., 1948; Jefferies et al., 1991; Ruest et al., 2002; Sattelle et al., 2008). Ryanodine receptors (RyRs) are intracellular calcium channels in insects placed in the sarcoplasmic and endoplasmic reticulum in neuromuscular tissues and they are important for the control of calcium ions release. They are activated by calcium influx mediated by voltage gated calcium channels upon depolarization of the cell membrane (Brillantes et al., 1994; Marx et al., 1998; Messutat et al., 2001; Masaki et al., 2006; Shiomi et al., 2010; Wang et al., 2013; Liu et al., 2014; Vemu and Dumka, 2014; Yang et al., 2014). There are three types of ryanodine receptors: RyR1 is primarily expressed in skeletal muscle, RyR2 in myocardium, and RyR3 in the brain (Meissner and El-Hashem, 1992; Ogawa, 1994; Laitinen et al., 2001). The ryanodine shows high binding affinity for a class of ligand-gated calcium channels. This affinity is measured by the equilibrium dissociation constant (K_D). This means that as the K_D value is lower, the binding affinity of the ligand for its target is higher for the ryanodine receptor and the calcium trigger ligand, showing the high strength of the binding interaction between a ryanodine and a calcium ligand (Ito et al., 1986; Bull et al., 1989; McGrew et al., 1989; Chu et al., 1990; Ellisman et al., 1990; Stein et al., 1992; Chameau et al., 2001; Ebbinghaus-Kintscher et al., 2006; Rizzuto and Pozzan, 2006; Thomas and Williams, 2012; Santulli and R Marks, 2015). Ryanodine (ryanodyl 3-(pyrrole-2-carboxylate)) is a complex bridged diterpene heptol that is divided into two classes: alkaloid type and nonalkaloid type. The nonalkaloids type were isolated from Persea indica (Lauraceae) while the alkaloidal ryanoids (ryanodines and spiganthines) were isolated from Spigelia anthelmia (Logani-aceae) (Jansen et al., 2009). There are many studies pursued on the synthetic derivatives and structure-activity of the Ryania alkaloids (Soloway, 1976; Waterhouse et al., 1984; Ruest et al., 1985; Jefferies et al., 1992; Isman, 1997; Ujváry, 1999; Cabras et al., 2001; Gossauer, 2003; Nauen, 2006; Mao and Henderson, 2007; Akhtar et al., 2008; Sattelle et al., 2008; Dimetry, 2012; Feng et al., 2012; Wagner et al., 2012; Zhao et al., 2012; Grdiša and Gršić, 2013). Recently, two classes of chemicals that target the RyRs of insects have emerged as new insecticides and have attracted considerable attention. The first chemical class is anthranilic diamides and the second is phthalic acid diamides (Pence and Williams, 2010). These diamides are rapidly replacing the major uses of previous classes of insecticides because of their high efficacy against major pests resistant to these earlier chemicals and appear to have low toxicity to human health and the environment (He et al., 2019; Jactel et al., 2019; Li et al., 2020).

2.1. Anthranilic diamides

Anthranilic diamides are very demanding commercial class of insecticides. This class was discovered by the Dupont company during the last decade (Zhou et al., 2019; Boaventura et al., 2020). The most important characteristics of these diamides are their broad spectrum insecticidal efficacy and their environmental and ecological safety (Boaventura et al., 2020). A class of anthranilic diamides was being developed with two representative compounds including chlorantraniliprole and cyantraniliprole (Fig. 1). Chlorantraniliprole was the first commercialized diamide and exhibits an exceptional activity against lepidopteran pests (European Food Safety Authority et al., 2019; Kadala et al., 2019; Zhou et al., 2019; Jouraku et al., 2020; Satpathy et al., 2020). It activates ryanodine receptors by stimulation of calcium release from the sarcoplasmic reticulum of muscle cells causing impaired regulation, paralysis, and ultimately death of target species (Ahlawat et al., 2019; Chen et al., 2019; He et al., 2019; Héma et al., 2019; Jallow et al., 2019; Plata-Rueda et al., 2019; Silva et al., 2019; Passos et al., 2020; Shah and Shad, 2020; Williams et al., 2020). However, the researcher continued to pursue a wide range of polar groups on the anthranilic core with an emphasis on nitrile substitution (Barrania, 2019; Bolzan et al., 2019; Carscallen et al., 2019; Mao et al., 2019; Gao et al., 2019; Meng et al., 2019; O'Neal et al., 2019; Sharma et al., 2019; Sreedhar, 2019; Truong and Pessah, 2019; Jiang et al., 2020). This effort resulted in the discovery of cyantraniliprole, a second product candidate in this chemical class possessing excellent interspectral activity against a range of insect orders, including lepidopteran and hemipteran pests (Hopkinson and Pumpa, 2019; Plata-Rueda et al., 2019; Ran et al., 2019; Zhang et al., 2019). This insecticide product exhibits a very low toxicity to vertebrates and non-target organisms.

2.2. Phthalic diamides

The phthalic diamide shares the same mode of action to anthranilic diamides. The chemical structure of phthalic acid diamides is marked by three parts as shown in Fig. 2A: (*a*) the phthaloyl moiety that have yielded the important commercial product namely FBD, (*b*) the aliphatic amide moiety, and (*c*) the aromatic amide moiety (Boaventura et al., 2020; Gonring et al., 2019; Lin et al., 2020; Zhang et al., 2020; Zuo et al., 2020). The initial leads of this compound had discovered by Nihon nohyaku in 1993 in a pyridine dicarboxamide herbicide and is being co-developed by N. nohyaku and bayer in july 2005 (Bolzan et al., 2019).

3. Flubendiamide

Flubendiamide or 1, 2-benzenedicarboxamides N0-[1, 1-dimethyl-2-(methyl-sulfonyl) ethyl]-3-iodo-N-{4-[2, 2, 2tetrafluoro-1-(trifluoromethyl) ethyl]-0-tolyl} is a new insecticide belongs to phthalic acid diamides, where its chemical structure reveals several interesting features (Tohnishi et al., 2005). This diamide compound is characterized by three substituents (Fig. 2B): a heptafluoro isopropyl group in the anilide moiety, a sulfonylalkyl group in the aliphatic amide moiety, and an iodine atom at the 3-position of the phthalic acid moiety (Singh Battu et al., 2008; Kato et al., 2009; Paramasivam and Banerjee, 2011). The heptafluoro-isopropyl side chain confers to the compound lipophilic character and is required for the very high insecticidal activity. FBD has also an iodine atom substituent, which are accounts for superior activity by comparison with the chloro analogue. The introduction of this halogen done by Sandmeyer reaction (Fig. 3A) on the corresponding amino phthalic (Li et al., 2006). The two amines are subsequently introduced in a regioselective manner from the phtalanhydride and the isoimide (Fig. 3B). The introduction of heptafluoroisopropyl is fixed via a radical reaction of 2- bromo-heptafluoropropane on the aniline side (Fig. 3C) (Jeanguenat, 2013). FBD is widely used as a strong potent for controlling lepidopterous pests including Helicoverpaspp, Heliothisspp, Spodopteraspp, Plutellaspp, Trichoplusiaspp and Hyrotisspp (Das et al., 2017). It is also very safe for mammalians (Isaacs et al., 2012). In addition, it has recently been approved for use on major crops such as diamondback moth, cabbage whitefly, grapevine caterpillar, corn, cotton, tobacco, seeds and stone nuts, grapes and vegetables (cucurbits,

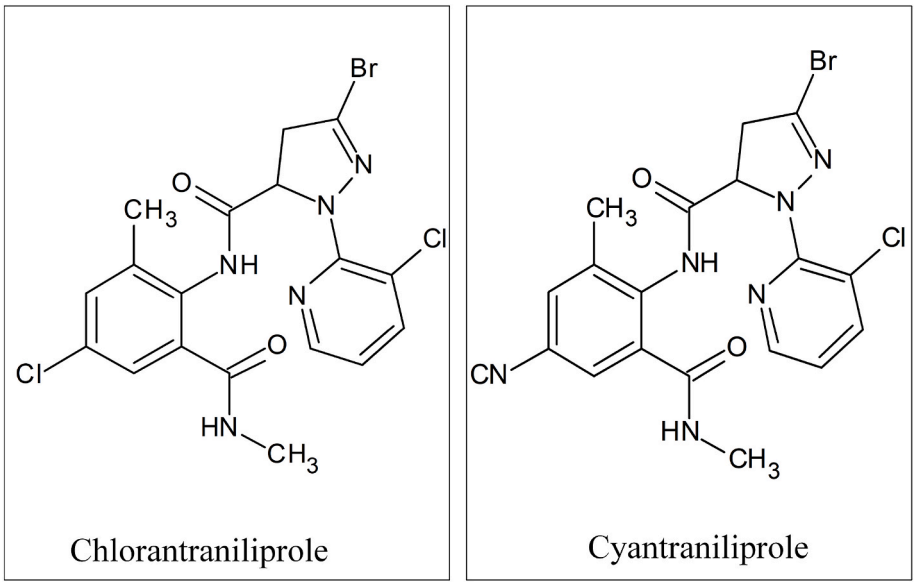

Fig. 1. Chemical structure of clorantraniliprole and cyantraniliprole.

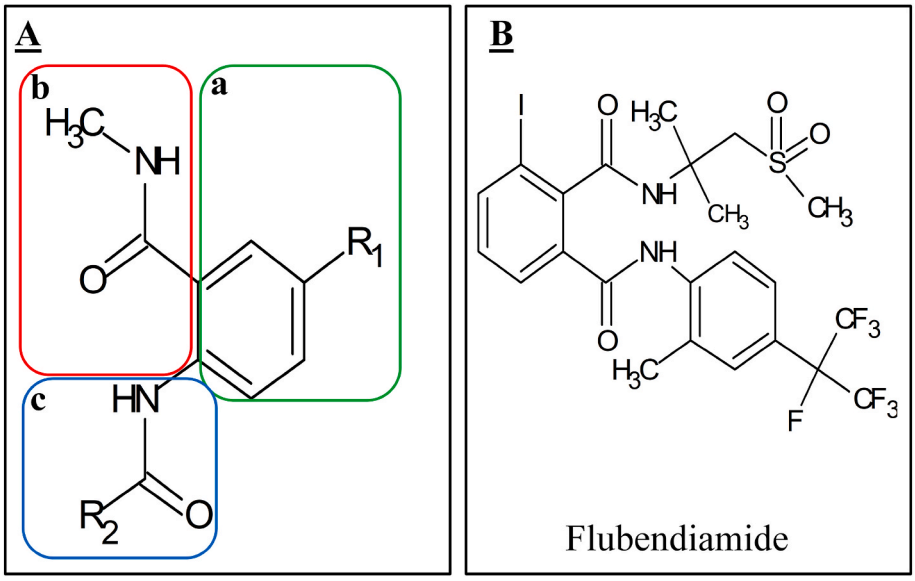

Fig. 2. Chemical structure of (A) phthalic diamide and (B) flubendiamide.

fruiting vegetables and okra vegetables) (LU and LI, 2012).

3.1. Mechanism of action

The mode of action of FBD has been detailed by Ebbinghaus-Kintscher and colleagues (Ebbinghaus-Kintscher et al., 2006). They affirm that FBD is a selective activator of the insect ryanodine receptor, inducing ryanodine-sensitive cytosolic Ca^{2+} transients that were independent of the extracellular Ca^{2+} concentration (Fig. 4). A study reported by Lummen et al. (Lümmen et al., 2007) on isolated neuronal cells showed that FBD activates the intracellular release of ryanodine-sensitive calcium. Another separate study effected by Masaki et al. (2006) showed that FBD stimulates Ca^{2+} pump activity by reducing the coupling between RyRs and the pump, resulting a decrease in internal calcium concentration. This specific mode of action of FBD produced several disruptions of muscle function in the target insect with symptoms of poisoning including rapid cessation of feeding, contractile paralysis, and regurgitation resulting in insect death.

3.2. Acute toxicity

FBD shows low oral, dermal and inhalation toxicity (EFSA, 2013). However, their successive use can lead to the accumulation of its residues on crops at harvest time (Regueiro et al., 2015). The presence of trace amounts of FBD residues and degradation of organic compounds in agricultural crops may result in potential health risks. Although in small quantities, their accumulation in the body can have adverse effects on human health, which should be controlled to ensure “food safety” (Boobis et al., 2008). For this reason, the European Union directives have set maximum residue limits (MRLs) of 0.2 ppm for FBD in food, agricultural products or feed (Tohnishi et al., 2005; Wilkowska and Biziuk, 2011). FBD presents a low acute risk toward birds and mammals, beneficial insects including honey bees and natural lepidoptera predators, and terrestrial plants. Although, the continued use of FBD will result in negative effects due to one degradation product of FBD in water is highly toxic to fish (Tohnishi et al., 2005; Lahm et al., 2009; EFSA, 2015). FBD is less absorbed in rats when administered orally and has a

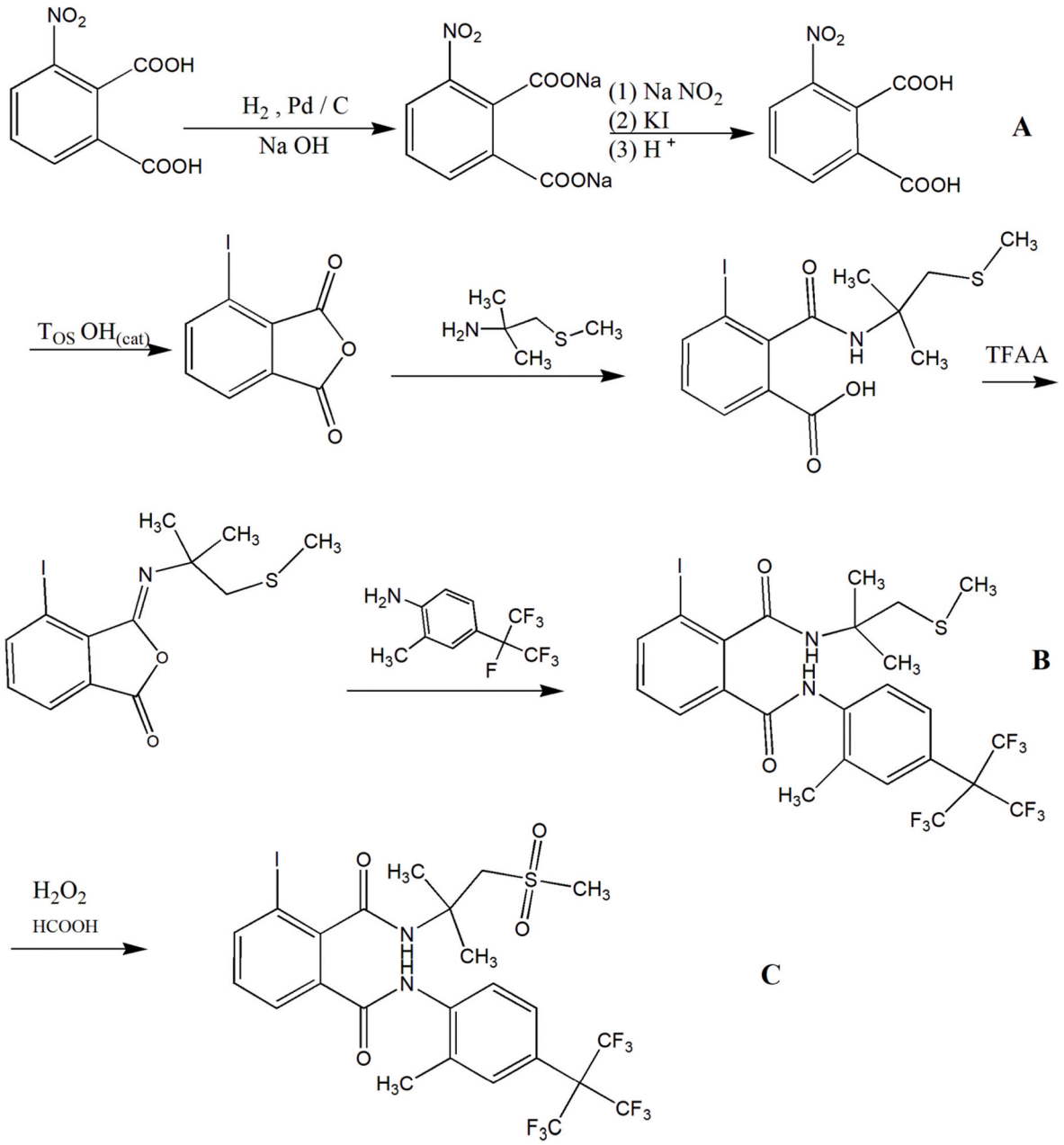

Fig. 3. Schematic illustration of the synthesis of flubendiamide.

wide distribution with the highest concentrations in target organs such as the liver, thyroid gland, kidney, bloodstream. Numerous studies confirm that FBD does not have genotoxic effects and does not possess mutagenic and carcinogenic activity, selective neurotoxic and immunotoxic action (Ludlow, 2010). Various studies on the degradation of FBD in soil, which generally degrades in two phases, the first one being faster, leading to photolysis, and the second one being quite slow, establishing that the substance is sufficiently stable in soil under aerobic and anaerobic conditions (Ludlow, 2010). FBD records a low risk assessment towards earthworms, soil macro-organisms and soil microorganisms.

4. Sample preparation methods for determination of FBD residues

A variety of pre-treatment and extraction techniques were used for determing FBD residues in food samples. In laboratories, there are no standard methods for extracting pesticides. Furthermore, the extraction procedure follows a common path that includes the release of the desired analyte from the matrices, followed by a cleanup process that refers to a step or series of steps in the analytical procedure in which the majority of the potential interference co-extracts are removed by physical or chemical methods (Fig. 5).

4.1. Pre-treatment and extraction methods

The process of extracting pesticides from the sample is a fundamental aspect of the analytical process (Jing and Amirav, 1997). Solid-liquid extraction (SLE) is the initial step in the sample preparation process for most agro-environmental samples such as crops and soil, as well as for animal samples such as eggs and tissues. As described earlier, FBD is highly soluble in water, which paves the way for a wide range of extractants capable of efficiently extracting FBD from solid samples (Ebbinghaus-Kintscher et al., 2006). Acetone, acetonitrile and methanol

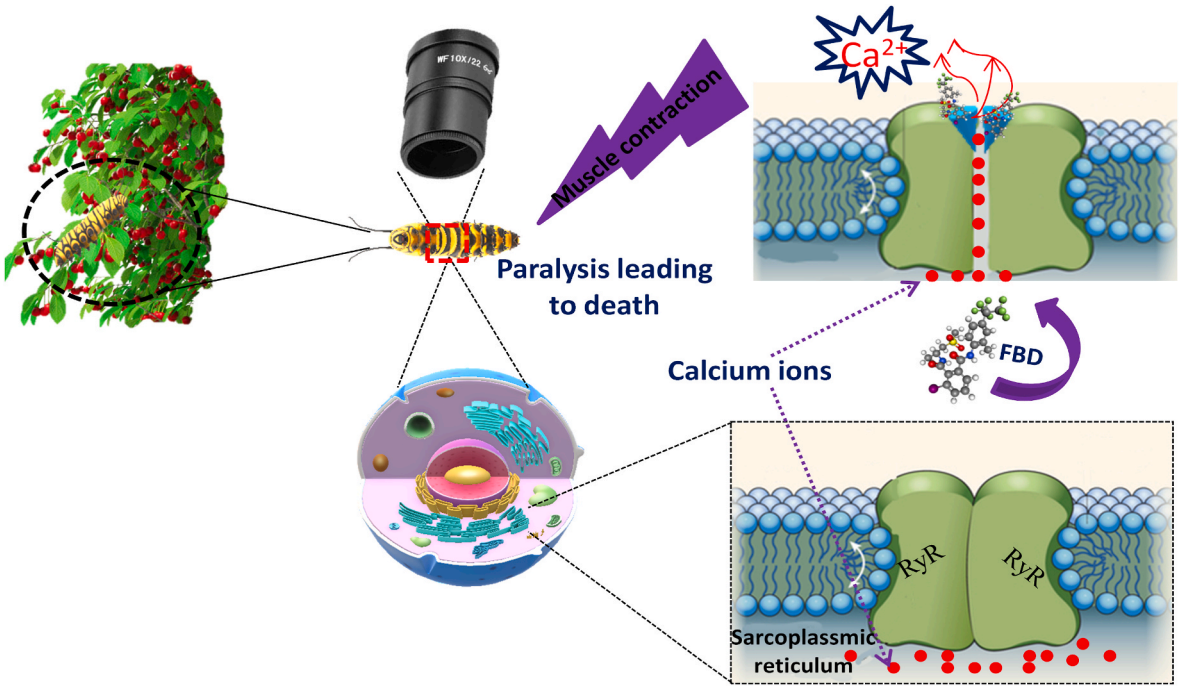

Fig. 4. Mode of action of flubendiamide on lepidoptera pest.

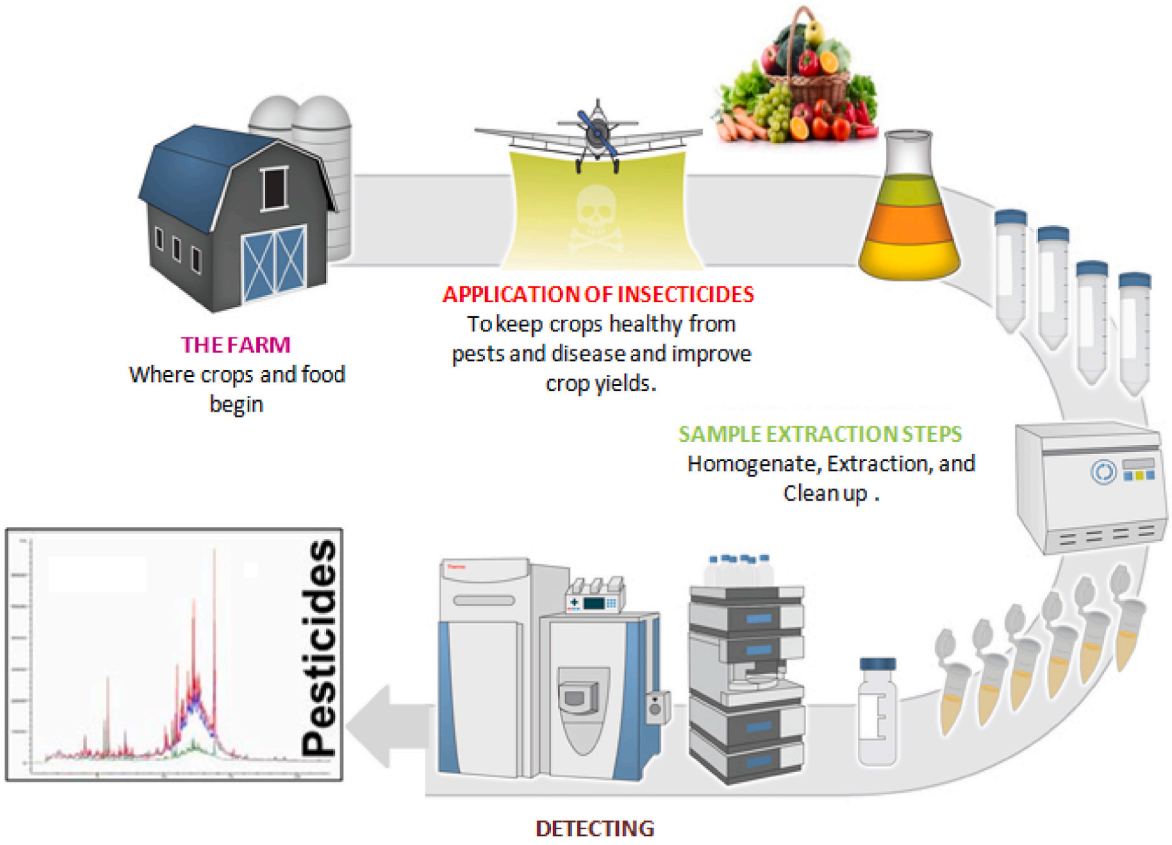

Fig. 5. Analytical procedure for determining pesticide in food matrices.

are mainly used for the extraction of FBD (Table 1). Especially, acetonitrile has an exceptional permeability to the water-soluble component of the sample due to its good miscibility with water. In addition, the solvent minimizes the co-extraction of hydrophobic components such as lipids, pigments and wax; the addition of salts also allows them to be quickly separated from the aqueous phase. It is preferable to shake the samples to extract them, as long as this gives acceptable results for the samples taken and/or the standard reference materials (if available).

Table 1
Clean up preparation in the analysis of flubendiamide residues in foods.

Aghris et *al.*					
Matrix	Clean-up method	Sample preparation	Recovery (%)	RSD (%)	Ref
Pigeon/Pea Cabbage/Tomato	D-SPE	**Extraction:** 10 mL acetonitrile, 50 mL polypropylene, 1.0 g NaCl, and 4.0 g $MgSO_4$ **Clean-up:** 10 mg of PSA + 150 mg of $MgSO_4$	85–99	–	(Paramasivam and Banerjee, 2011)
Cabbage	LLP	**Extraction:**1.5 g sodium chloride, 6 g anhydrous magnesium sulfate, 30 mL of acetonitrile. **Clean-up:** $MgSO_4$ (150 mg), PSA (25 mg), C18 (25 mg).	72.4–119	15	Chen et al. (2014)
Cabbage	D-SPE	**Extraction:** 30 mL acetonitrile, 1.5 g sodium chloride, 6 g anhydrous magnesium sulfate **Clean-up:** 150 mg $MgSO_4$, 25 mg PSA	80.7–99.4	4.90	Sharma et al. (2014)
Cucumber	D-SPE	**Extraction:** 30 mL acetonitrile – 50 mL Teflon -3 g sodium chloride, 9 g anhydrous sodium sulfate (remove moisture). **Clean-up:** 12 mL acetonitrile +400 mg PSA +1.15 g anhydrous magnesium sulfate.	91–101	2.77	Sahoo et al. (2009)
Cabbage, Cauliflower, Brinjal/Chilli	D-SPE	**Extraction:** 15 mL acetonitrile, 3 g sodium chloride, 9 g anhydrous sodium sulfate **Clean-up:** 40 mg of PSA + 140 mg of $MgSO_4$	90.2–97.9	1.05–5.26	Mukherjee et al. (2012)
Chili	SPE	**Extraction:** 100 mL acetonitrile, 50 mL chloroform. **Clean-up:** 500 mg activated charcoal	89–95	6	(Gopal and Mishra, 2008)
Tomato	SPE	**Extraction:** 100 mL acetonitrile, 50 mL chloroform. **Clean-up:** 500 mg activated charcoal	84.4–96.4	2.30	Sharma and Parihar (2013)
Brinjal	SPE	**Extraction:** 100 mL acetonitrile, 50 mL chloroform. **Clean-up:** 500 mg activated charcoal	89–95.8	6	(Chen et al., 2014)
Chilli	SPE	**Extraction:** 100 mL acetonitrile, 50 mL chloroform. **Clean-up:** 500 mg activated charcoal	89–101	–	Takkar et al. (2012)
Jatropha Plant Leaves	AC	**Extraction:**100 mL acetonitrile + 0.002 M hydrochloric acid **Clean-up:** 10 g of alumina +50 mL of 9:1 hexane: ethyl acetate	–	–	Sharma et al. (2011)
Melon	LLP	**Extraction:** acetone (100 mL) dichloromethane (100 mL) **Clean-up:** n-hexane (6 mL) + acetonitrile (8 mL)	92–103.06	1.7–3.4	Kabir et al. (2018)
Rice	LLP	**Extraction:**1.5 g of sodium chloride, 6 g of anhydrous magnesium sulfate **Clean-up:** $MgSO_4$ (150 mg), PSA (25 mg), and C18 (25 mg)	92–101	–	(Wu et al., 2014)
Cabbage	SPE	**Extraction:**1.5 g of sodium chloride, 6 g of anhydrous magnesium sulfate **Clean-up:** $MgSO_4$ (150 mg), PSA (25 mg), and C18 (25 mg)	92–99.8	–	Chen et al. (2015)

D-SPE: Dispersive solid phase extraction, **PSA:** Primary secondaryamine, **GPC:** Gel permeation chromatography, **SPE:** Solid phase extraction, **LLP:** Liquid Liquid partitioning, **AC:** Adsorption chromatography.

This practice is acceptable for the extraction of many pesticide residues from many crops, but may be problematic for systemic pesticides present in some pasty foods where agitation does not allow easy penetration into the matrix. Soxhlet extraction has been used since the early 1900s (De Castro and Priego-Capote, 2010). Despite the fact that this technique is time consuming and requires the use of relatively large volumes of solvent, it is cost-effective and robust. Thus, the efficiency of the process is high, which makes it still useful today (Brits et al., 2016). Increasingly, new extraction techniques are being sought, with shorter extraction time and minimal use of solvents including ultra-turax macerators and sonication. These are faster and more efficient techniques for extracting organic analytes from solids or semi-solids.

Singh Battu et *al.* (Singh Battu et al., 2008) analyzed the residues of FBD in a number of crops included cabbage, tomato, pigeonpea and chilli based on extraction with high-speed homogenization using LC equipped with diode array detector (DAD). In this study, they evaluated different extractants like dichloromethane, acetone, acetate, ethyl acetate hexane and acetonitrile. The results showed that acetone and dichloromethane had low solvating capacity and very low recoveries, less than 20%. Similarly, with ethyl acetate and hexane, the recovery percentages were very low. However, acetonitrile was selected as the optimal extractant because the matrix interference peaks were smaller and good recoveries were achieved (85%–94%). Because acetonitrile is convenient for application to samples with high sugar content and because it evaporates easily, it has also been selected as an extractant for FBD residues. Tian et *al.* developed a new method for simultaneous determination of residues of FBD in edible mushrooms (Tian et al., 2020). The samples were prepared using the QuEChERS methodology. This methodology was chosen because it allows the simultaneous extraction of polar and non-polar compounds. The extraction was performed with the use of 10 mL of acetonitrile; the mixture was then shaken for 10 min. Next, 1 g of NaCl and 4 g of anhydrous $MgSO_4$ were added and the final extract were shaken again for 5 min. This method was successful in separating and extracting the sample, and also had satisfactory recoveries which ranged from 73.5 to 110.2%. In addition to the extractants described above, MeOH has been used for the extraction of FBD (Diandian and Wenzhu, 2012; Chen et al., 2014; Ma et al., 2015; Wang et al., 2018). Chen et al. (2014) used MeOH to extract FBD in cabbage. Wang et al. (2021) used MeOH–water (90:10) containing 0.1% formic acid to extract six amide pesticides including cyantraniliprole, mandipropamid, boscalid, fluopicolide, thifluzamide and flubendiamide, in vegetables and fruits. As shown in Table 1, in a few cases, MeOH is used as an extractant for FBD. However, instead of concentrating the sample extract under reduced pressure, adjusting the concentration of MeOH with water allows a smooth transition to the next clean-up process of the sample extract. Therefore, MeOH is also regarded as an effective extractant for FBD.

4.2. Clean-up approaches

Clean up is an essential step to clear away co-extractives interfering substances from the matrice (Ballesteros and Ramos-Martos, 2010). Therefore, following the extraction procedure, clean-up of the sample extract is positioned as the next important step (Mekonen et al., 2014). For most samples, it is critical to clean up the sample extracts using an appropriate procedure and to reduce matrix interference in chromatographic analysis to the greatest extent possible (Schenck and Hobbs, 2004). To eliminate matrix interference before the use of highly selective and sensitive analytical equipment such as liquid chromatography tandem mass spectrometry (LC–MS/MS), a time-consuming clean-up

process was required. Apparently, column chromatography and liquid-liquid extraction (LLE) were applied to clean up the sample extracts. The most extensively utilized clean-up method as a replacement to the above-mentioned is solide phase extraction (SPE). Among the many adsorbents used in SPE are normal phase adsorbents such as silica gel and Florisil, reverse phase adsorbents such as C18 and C8, ion exchange adsorbents and GCB. In SPE, a sample extract dissolved in a tiny amount of organic solvent is placed into a cartridge that has been pre-equilibrated with an appropriate amount of organic solvent or water. Extracted pesticides are eluted selectively after the matrix components are washed away while target pesticides are kept on the adsorbent. SPE is a clean-up procedure that outperforms standard column chromatography in terms of the amount of organic solvent used, speed of operation, and simplicity. As expected, SPE has been shown to clean up sample extracts in residue analysis procedures for FBD. Several adsorbents have been applied to clean-up FBD in sample extracts by SPE such as primary secondary amine (PSA) (Paramasivam and Banerjee, 2011; Mukherjee et al., 2012; Chen et al., 2015; Słowik-Borowiec and Szpyrka, 2018; Sharma et al., 2019; Park et al., 2021; Reddy et al., 2021; Wang et al., 2021), activated charcoal (Sahoo et al., 2009; Sharma et al., 2018; Takkar et al., 2012; Sharma and Parihar, 2013), C18 (Abbas et al., 2017; Ares et al., 2017; Kabir et al., 2018; Kralj et al., 2018; Sherma and Rabel, 2018; Huh et al., 2019; Li et al., 2020; Sharma et al., 2019; Wang et al., 2021), silica gel (Kabir et al., 2018; Sherma and Rabel, 2018; Ma et al., 2021; Souza et al., 2017), Florsil (Hwang et al., 2018; Malhat et al., 2018; Słowik-Borowiec and Szpyrka, 2018;; Lee et al., 2019; Ruiz et al., 2020; Kim et al., 2021), graphitized carbon black (GCB) (Lee et al., 2018; Słowik-Borowiec and Szpyrka, 2018; Wang et al., 2021). And hydrophilic-lipophilic balanced (HLB) (Ballesteros and Ramos-Martos, 2010; Abbas et al., 2017; Casado et al., 2018; Hou et al., 2019; Lu et al., 2019; Li et al., 2020; Jiafeng et al., 2021; Park et al., 2021). According to reports, HLB is effective for cleaning sample extracts containing pesticides with a wide range of physicochemical properties, ranging from hydrophilic pesticides like FBD to hydrophobic pesticides. HLB has been applied as a clean-up method of samples of animal origin (Li et al., 2020; Jiafeng et al., 2021) and in food samples (Hou et al., 2019; Ballesteros and Ramos-Martos, 2010). In d-SPE, various adsorbents were utilized in the QuEChERS procedure based on the properties of the samples. In the original QuEChERS (Sharma et al., 2019), the mixture of MeCN extract (12 mL), PSA (400 mg), and anhydrous $MgSO_4$ (115 mg) was stirred by hand or vortex. After centrifugation of the mixture, the supernatant was collected to complete the cleanup. The method has favorable accuracy with RSD values of 1.05–5.26%. Furthermore, combining different adsorbents can lead to larger clean-up effect. In fact, d-SPE has been extensively used to clean up extracts of vegetables, fruits, cereals, and others (see Table 1). As a conclusion, d-SPE is a tailor clean-up method that may be used by arbitrarily combining adsorbents based on matrix component characteristics in samples. Lipid-rich foods like olives and avocados are regarded as a difficult sample matrix for developing a pesticide residue analytical method. Furthermore, contamination of the ion source with lipid could result in a reduction of analytical sensitivity due to ion suppression. Because of these reasons, lipid removal is essential. To remove lipids and proteins in a sample extract, freezing this latter can be done to precipitate these components (Hildmann et al., 2015; Bernal et al., 2019). Nguyen Huu Vinh et *al.* (Vinh et al., 2010) applied a frozen low temperature clean-up to MeCN extracts of 262 pesticides including FBD in milk, butter and peanut samples. This method is the simplest approach to remove lipids from a sample extract. However, the procedure is time consuming and does not completely eliminate lipids. Afterward, clean-up processing is frequently required. Gel permeation chromatography (GPC) also aids in the separation of low-molecular-mass chemicals like insecticides from higher-molecular-mass matrix components like lipids. Hildmann et al. (2015) applied a small-scale GPC that halves the consumption of extractant (mobile phase) to the removal of lipids in a multi-class residue analytical method for 78 pesticides including FBD in egg samples. Although GPC has been effective at removing high molecular mass lipids, it has been difficult to remove low molecular mass lipids. For that reason, the sample extract was finally cleaned up with C18 SPE.

5. Analytical techniques for FBD detection in food matrices

Consumption of fruits and vegetables is considered part of a regular, balanced diet and a healthy, active lifestyle. In order to achieve good quality and better yields, insecticides are used in the cultivation of fruits and vegetables. Apart from their benefits, insecticide residues left on fruits and vegetables can be very harmful when consumed by humans. To this end, several analytical techniques such as gas chromatography (GC), LC-MS/MS, and high performance liquid chromatography (HPLC) coupled with detectors such as nitrogen and phosphorus detector, photometric flame detector, diode array detector, fluorescence detector and MS detector to determine pesticide residues in food matrices. Table 2 summarizes various methods for determining FBD in food matrices.

5.1. Gas chromatography

The most common chromatographic technique is GC, used for separating compounds based on their volatility. A number of studies have been reported for FBD analysis by coupling GC with various detectors such as electron capture detector (ECD), flame photometric detector (FPD), nitrogen phosphorus detector (NPD) and flame ionization detector (FID). In comparison to traditional detectors, the mass detector (MS) has higher sensitivity, accuracy, reproducibility, and effectiveness in removing interferences (Kende et al., 2006). Nowadays, it is possible to combine MS analyzers with triple quadrupole mass spectrometer (QqQ)-MS (Tobin et al., 2014). Further, to eliminate the matrix interference, selective ion monitoring (SIM) is used. Similarly, Sharma et *al.* (Sharma et al., 2018) reported a method for the identification and quantification of FBD and deltamethrin in cucumber using GC-MS in (SIM) mode based on the use of target and two qualifier ions. Recently, the use of GC methods in detection of FBD are found inappropriate due to its volatility and poor thermal stability.

5.2. Liquid chromatography

The majority of the reviewed studies claim that the detection of FBD have been carried out by LC coupled with various detectors as summarized in Table 2. Detectors such as ultraviolet (UV), photodiode array (PDA), diode array detector (DAD), and mass (MS) detectors are used because of their sensitivity. Gopal and Mishra (2008) developed a method for the determination of FBD in rice. The insecticide was separated and quantified by reversed-phase LC with UV-diode array detection at 220 and 260 nm. Recoveries were ranged from 80% to 92.5% and limit of quantification (LOQ) was 25 μg/kg. Sharma et al. (2015) used PDA detector for the analysis of FBD and its metabolite (des-iodo FBD) in Chili; the recovery was in the range from 79 to 98%.

Recently, MS detection has become the most accepted and successful methodology in FBD analysis due to its increased sensitivity and selectivity. In LC-MS, ionization is usually performed by atmospheric pressure ionization (API) sources, in which the API can ionize both polar and non-polar analytes. In addition, mass analyzer such as triple quadrupole (QqQ) and quadrupole-trap (Q-Trap) are used for qualitative and quantitative analysis of FBD. Ares et al. (2017) developed a method to extract trace FBD from honey of different botanical origins using a single quadrupole. Another study conducted by Chen et al. (2018) reported a method for FBD estimation in cabbage using a QqQ-MS-equipped LC-MS/MS rapid resolution method. Further, Buddidathi et al. (2016) reported a method for simultaneous determination of FBD and its metabolite Des-Iodo FBD in Capsicum and Grape using a modified QuEChERS method equipped with LC-MS/MS. Caboni et al. (2008)

Table 2
Analytical techniques for determining flubendiamide in food matrices.

Aghris et *al.*							
Matrix	Sample treatment (time)	Reagents (g, mL)	Recoveries	Baseline separation	LOQs (μg/Kg)	System (SP, time)	Ref.
Vegetables	SE + clean-up + EV (>12 h)	0.5 g, > 900 mL	85–99%	Yes	10	LC-PDA (C_{18}, 15 min)	(Singh Battu et al., 2008)
Capsicum fruit	QuEChERS (<30 min)	8.1 g, 15 mL of ACN	96–100%	Yes	50	LC-PDA; LC-MS/MS (C_{18}, 20 min)	Buddidathi et al. (2016)
Fruits and vegetables	QuEChERS + EV (<30 min)	3.0 g, 20 mL of ACN	87–112%	Yes	0.8	LC-MS/MS (C_8, 8 min)	Caboni et al. (2008)
Cabbage	QuEChERS + EV (<30 min)	7.7 g, 30 mL of ACN	81–92%	Yes	0.3 (LLOD)	LC-MS/MS (C_{18}, 12 min)	(Chen et al., 2014)
Cabbage	QuEChERS (<30 min)	7.7 g, 30 mL of ACN	80–91%	Yes	15 (LLOD)	LC-MS/MS (C_{18}, 12 min)	Chen et al. (2014)
Cardamon	QuEChERS (NP)	NS (ACN)	>83%	NP	50	LC	(De Castro and Priego-Capote, 2010)
Rice	SE + CP + EV (>5 h)	9 g, > 400 mL	80–92	Yes	25	LC-UV (NS, 14 min)	(Gopal & March et al., 2009)
Pea	SE + clean-up + EV (>12 h)	0.5 g, > 900 mL	87–96%	Yes	50	LC-UV-Vis (C_{18}, 5 NS)	Kale et al. (2012)
Fodder berseem clover	QuEChERS (<20 min)	21.1 g, 15 mL of ACN	87–99%	Yes	10	LC-MS/MS (C_{18}, 5 min)	Kaur et al. (2016)
Tomato	SE + clean-up + EV (>24 h)	25.5 g, 400 mL	98–102%	Yes	10	LC-PDA (C_{18}, 12.5 min)	Mohapatra et al. (2011)
Cabbage, tomato and pea	QuEChERS + EV (<30 min)	5.2 g, 10 mL of ACN	85–99%	Yes	10	LC-UV-Vis (C_{18}, 13 min)	Paramasivam & Banerjee (2011)
Tomato	QuEChERS + EV (<30 min)	5.2 g, 10 mL of ACN	97–99%	Yes	10	LC-UV-Vis (C_{18}, 13 min)	Paramasivam & Banerjee (2012)
Cabbage	QuEChERS + EV (<30 min)	5.2 g, 10 mL of ACN	96–98%	Yes	10	LC-UV-Vis (C_{18}, 13 min)	Paramasivam & Banerjee (2013)
Gherkin	QuEChERS + EV (<30 min)	5.7 g, 20 mL of ACN	87–93%	Yes	10	LC-DAD (C_{18}, 12 min)	(Paramasivam et al., 2014)
Bee pollen	QuEChERS (<30 min)	1.75 g, 7 mL (5 mL of ACN)	80–84	No	5	LC-MS/MS (C_8, 19 min)	(Percival and Schroeder, 2017
Tomato	SE + clean-up + EV (>12 h)	0.5 g, > 900 mL	86–96%	Yes	10	LC-PDA (C_{18}, NS)	(Sharma & Pawar and Bhilave, 2020)
Cabbage	SE + clean-up + EV (>12 h)	25.5 g, 400 mL	81–100%	Yes	10	LC-PDA (C_{18}, 15)	(Sharma et al., 2014a)
Tomato	SE + clean-up + EV (>12 h)	0.5 g, > 900 mL	85–101%	Yes	10	LC-PDA (C_{18}, 15 min)	(Sharma et al., 2014b)
Chili	SE + clean-up + EV (>12 h)	0.5 g, > 900 mL	79–98%	Yes	10	LC-PDA (C_{18}, 15 min)	Sharma et al. (2015)
Gherkin	QuEChERS + EV (<30 min)	8.4 g, 15 mL of ACN	98–101%	Yes	10	LC-PDA (C18, 20 min)	(Souza et al., 2017)
Brinjal	SE + clean-up + EV (>12 h)	0.5 g, > 900 mL	89–96%	Yes	50	LC-PDA (C18, 15 min)	Takkar et al. (2012)
Cabbage	QuEChERS (<20 min)	7.75 g, 10 mL (5 mL of ACN)	–	No	1.1 × 10–6 (LOD)	PD-LVI-LC-MS/MS (C18, 15 min)	(Zhang et al., 2016)
Tomato	SE + clean-up + EV (2–3 h)	50 g, 100 mL	82–90%	Yes	10	LC-DAD (C18, 10 min)	(Kooner et al., 2010)
Korean melon	SE + clean-up + EV (<30 min)	20 g, 900 mL	92–103.6%	Yes	20	HPLC-UV-Vis (C18, 13 min)	Kabir et al. (2017)
Okra	SE + clean-up + EV (40 min)	10 g, 50 mL	85%–88%	Yes	0.01	HPLC-UV-Vis (RP18, 10min)	(Sharma et al., 2011)
Rice	SE + clean-up + EV (40 min)	5 g, 30 mL	86–94%	–	10	LC-MS/MS (C18, 30 min)	Chen et al. (2015)

ACN, acetonitrile; **CP,** column partitioning; **DAD,** diode array detector**; dSPE,** dispersive SPE; **EV**, evaporation; **LOD**, limit of detection; **MS/MS**, tandem mass spectrometry**; PDA,** photodiode array detector; **PD-LVI,** pre-column dilution large volume injection; **QuEChERS**, quick, easy, cheap, effective, rugged and safe; **SE**, solvent extraction.

developed a method for measuring FBD residues obtained from a variety of vegetables and grains. They then extracted the residue with acetonitrile and homogenized with magnesium sulfate and sodium chloride. After centrifugation, the top layer was analyzed by LC-MS/MS. In reference to the use of LC-MS and MS/MS, a new method has been reported with the use of ultra performance liquid chromatography (UPLC) because of its sensitivity and high chromatographic efficiency in analyzing FBD in fruits and vegetables. Ma et al. (2015) reported a method for the determination of six amide pesticide residues in vegetables and fruits with Electrospray Ionization –Time of Flight (ESI-TOF) detection. Regueiro et al. (2017) have reported a method for the quantification and confirmation capabilities of UPLC coupled with QqQ and hybrid Q-TOF-MS in FBD residue analysis.

5.3. Enzyme linked immunosorbent assay ELISA

In recent years, immunochemical techniques have received much attention for the rapid identification and detection of FBDs in various food matrices due to their attractive features of a rapid, simple, portable and inexpensive detection method. ELISA stands for “enzyme-linked immunosorbent assay,” which is a simple and quick method for detecting soluble substances like antibodies, hormones, peptides, and others. It is considered one of the analytical approaches offering specificity and sensitivity to a specific type of insecticide due to the antigen-antibody interaction (Li et al., 2021). Over the past decades, traditional antibodies, such as polyclonal and monoclonal antibodies (pAbs and mAbs), have been widely used to develop immunoassays for small

molecules. Nanobody (Nb)-based immunoassays have recently proven to be a powerful tool for detecting environmental compounds such as insecticides in complex matrices like food matrices. The widespread availability of molecular biotechnology allows for the gene engineering of Nbs to facilitate speed of detection, improve analytical sensitivity, and either increase the specificity or even broaden the application range of Nbs. A study conducted by Zhang et *al.* showed that the application of an indirect competitive ELISA (icELISA) based on monoclonal antibody could lead to detection of anthranilic diamide insecticides namely cyantraniliprole in pakchoi vegetable (Zhang et al., 2015). The inhibition concentration (IC_{50}) values were ranging from 0.43 to 6.15 μg L^{-1}. The method achieved a detection limit of 1.57 μg L^{-1}. The interesting feature of this method is that no obvious cross-reactivity was observed for the analytes studied. The accuracy of the method was verified using the HPLC method and recoveries were 94–101%. These results proved that the ic-ELISA could be used as a sensitive device for monitoring of target analytes.

5.4. Other extraction and detection approaches

High or low water content can have very different effects on the behavior of food products, leading to a range of undesirable consequences such as microbial growth, mycotoxin formation, alteration of the sensory quality of the final product, unstable production conditions and unclear commercial problems. Therefore, it has issued routine and rapid methods for determining the water content of food samples. The water content of food samples is most often determined using rapid instruments such as capacitance-based or microwave analyzers, which are inexpensive and widely available. Whereas near-infrared spectroscopy is most accurate indirect method for water content determination (Zhangetal., 2019).

Various detection methods have been used to estimate FBD from fruits and vegetables. Traditional analytical methods for detecting pesticides, such as gas and liquid chromatography, have great selectivity and sensitivity. However, these approaches have drawbacks such as being time consuming, needing highly skilled personnel, and requiring the use of expensive instruments. Therefore, advanced approaches for the determination of pesticides were reported using sensor-based techniques such as electrochemical impedance spectroscopy (EIS) (Lahrich et al., 2016), differential pulse voltammetry (DPV) (Farahi et al., 2014, 2015, 2016), square wave anodic stripping voltammetry (SWASV) (El Harmoudi et al., 2013), differential pulse anodic stripping voltammetry (DPASV) (Elkasmi et al., 2016), and square wave voltammetry (SQW) (Ajermoun et al., 2019, 2020; El Harmoudi et al., 2017; El Mhammedi et al., 2010; El Mhammedi et al., 2007a, 2007b; El Mhammedi et al., 2008; El Mhammedi et al., 2009). These electrochemical techniques are very sensitive and offer several advantages including low-cost, simplicity, rapid operation and low detection limits compared to the conventional chromatographic methods (Laghrib et al., 2020).

Recently, numerous sensor (Rawtani et al., 2018; Gao et al., 2019; Aghris et al., 2021a, 2021b, 2022) and biosensors such as optical (Biswas et al., 2016; Zhang et al., 2016; Singh et al., 2017; Wang et al., 2017), electrochemical, piezoelectric (Kaur et al., 2004; March et al., 2009; Cervera-Chiner et al., 2020), and molecular imprinted polymer (MIP) (Wang et al., 2016; Saylan et al., 2017; Tan et al., 2019; Singh et al., 2020; Xu et al., 2020) were majorly used for the detection of pesticides. Apart from the benefits offered by these detection methods, the main disadvantage discovered was the small number of pesticides detected, making the method vulnerable.

6. Conclusions and future perspectives

The determination of FBD residues in food as complex matrices is a major concern to several areas such as health and quality control. Therefore, analytical methods have to be approving for bring out both research and monitoring programs. In this field, reproducible analytical methods are appropriate to get the effective separation, selective identification, and accurate quantification of FBD analyses in food products. The extraction of flubendiamide is often followed by clean-up with SPE, D-SPE or GPC. Then, analytical separation with LC and GC coupled with tandem mass spectrometry for identification and quantification was used. Although the advantages in separation and detection of the chromatographic techniques, clean-up remains important to obtain reliable data. However, the presented techniques used for pesticide residues detection present limitations that are laborious, times consuming and destructive methods. In this context, electrochemical sensors and biosensors have attractive analytical characteristics and may become useful tools in detection of pesticides due to the fact that their determinations are simple, fast, sensitive, selective and had lower detection limits compared to the conventional chromatographic methods. Generally, the monitoring of insecticide levels in the different food matrices appears to be necessary to ensure human safety.

Given the danger of FBD to human health, surveillance activities should be increased, and strict measures should be taken when high levels of FBD are detected in food products. Farmers and extension agents should be trained in good agricultural practices by ministries of agriculture and related agencies to help reduce this threat. Simpler extraction and cleanup procedures need to be developed for FBD analysis. Electrochemical sensors and biosensors are being applied for detection of vast number of pesticides in various food systems. Using Biosensors for pesticide detection, is a highly promising and cost-effective technique. Apart from detection, removal of pesticides from various food categories is much more important. With the help of nanotechnology, it could be possible to eradicate these pesticides from the food matrices. In future, research must be focused on biosensors for FBD insecticide, so that the use of sophisticated instruments can be omitted.

CRediT authorship contribution statement

S. Aghris: Conceptualization, Methodology, Supervision, Visualization, Writing – original draft, Writing – review & editing. **O. Tahiri Alaoui:** Conceptualization, Methodology, Supervision, Visualization, Writing – original draft, Writing – review & editing. **F. Laghrib:** Methodology, Writing – original draft. **A. Farahi:** Writing – original draft, Supervision. **M. Bakasse:** Conceptualization, Writing – review & editing. **S. Saqrane:** Conceptualization, Writing – review & editing. **S. Lahrich:** Conceptualization, Methodology, Supervision, Visualization, Writing – original draft, Writing – review & editing. **M.A. El Mhammedi:** Conceptualization, Methodology, Supervision, Visualization, Writing – original draft, Writing – review & editing.

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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