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Advance on Method for Determination of Imidacloprid Pesticides Residues in Vegetables

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Abstract

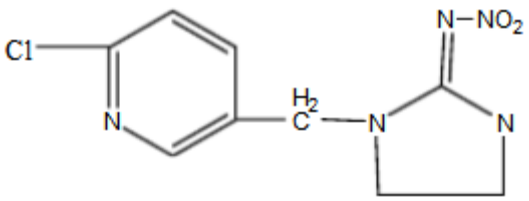
Imidacloprid is a normal kind of neonicotinoid pesticides. It can be detected by Gas Chromatography method, liquid chromatography method, electrochemical method and so on. This paper is mainly introduced the novel developments on determination of Imidacloprid pesticides residues in vegetables and provides technical support for the establishment of new method.

Keywords

Pesticide Residues, Determination of Pesticide Residues, Imidacloprid

1. Introduction

Pesticide residues in fruit and vegetables have recently been one of the primary factors which influencing food safety, especially since China join the WTO. Because the pesticide residues in products are beyond the standard, it is often happened that the export of fruit and vegetables may be impeded [1]. This situation consequently affects the development of the international trade of our country. Otherwise, pesticides and their residues are dangerous for human bodies and will bring serious harm to people. Chemical pesticides have an irreplaceable role in comprehensive prevention and control on crop pests and diseases. But as the long-term and heavily use of them, some stable chemical compositions are continuing accumulating in animals, plants and even in human bodies which are cumulative and chronic poisoning. Neonicotinoid insecticide is one kind of emerging and efficient insecticides that can be used to control piercing-sucking mouthparts insects. It can be applied in vegetables, fruit, paddy and other crops. In 2003, sales volume of neonicotinoid insecticides exceeded carbamate insecticides in the pesticide market of the world. What's more, its sales volume rosed to the third of the insecticide market and showed a tendence of increasing year by year. Imidacloprid is an example of the neonicotinoid broad-spectrum insecticides (The structure is shown in Figure 1). Among these insecticides, imidacloprid's sales volume ranked first in all insecticides and ranked second in all pesticides (only inferior to Glyphosate). Now, more and more attention has been paid to these kind of pesticide residues problems. For example, Japan starts carrying out Positive list system". In this system, the government will limit the amout of these two kinds of pesticides in vegetables on the above and focus on their monitoring. However, there is no research or report about the Near Infrared Spectroscopy Detection Method of these two pesticides.



Imidacloprid

Figure 1. The structure of imidacloprid insecticide.

2. Domestic Research Progress of Imidacloprid

Neonicotinoid insecticide is one kind of emerging and efficient insecticides which used to control piercing-sucking mouthparts insects. It can be applied in vegetables, fruit, paddy and other crops. And imidacloprid is widely applied in control pests whose residual problems have already raised many researchers' concerns. The problem has been reported in the detection method of residues in many samples, such as

cucumber, cabbage, soybeans, garlic, honeysuckle, apple and tea. Detection Technology of Imidacloprid residues: 1.Gas Chromatography Method: Solid Phase Extraction (SPE)-Gas Chromatography (GC)-Mass Spectrometry (MS) Method: Fully using Chromatographic separation, Quantitative function and the qualitative function of mass spectrometry by GC-MS. Now this detection technology has been widely applied in pesticide residues analysis and it's used for detection of pesticide metabolites and degradation products and applied in the multi-residue determination. Such as the Solid Phase Extraction (SPE)-Gas Chromatography (GC) -Mass spectrometry (MS) method that Liu Yongbo and others used to detect the residues of imidacloprid in fruit and vegetables. Samples were extracted with acetonitrile homogeneous and determined the volume by dichloromethane. Its recovery rate was from 80.0 to 89.0 percent and relative standard deviation was less than 5.5% and the detection limit was 0.0063mg/kg.

2.1 Liquid Chromatography

Wang Yong, etc. [2] used High performance liquid phase (HPLC)-diode array detection method. After extraction and concentration by methanol, the sample was extracted by 5% Sodium chloride aqueous solution (NaCl) and Petroleum ether. Then it was counter-extracted aqueous phase. When the sample dryed, they made up to volume and applied reversed C18 column. The mobile phase was methanol/water (its volume ratio was 32:68) and wavelength was 272nm. Results showed that: The regression equation was Y=126.208X-1.348 within the range of 0.1-10.0μg/mL. Its correlation coefficient was 0.99998 and showed a good linear relationship. The detection limit of this method was 0.05mg/kg. The average recovery rate was from 79.6% to 88.2% and the relative standard deviation was from 2.1 to 3.2 percent. This method can meet the analysis requirements of residues. Jiang Hui, et al. [3] used High Performance Liquid Chromatography Method to determine the samples after them extracting by Acetonitrile and having column purification with Florisil and medium alumina. The fortified recovery of this method was from 70 % to 120 % and the relative standard deviation was from 2.3 % to 15.3 %. The minimum detection limit in the samples could reach to 0.02 mg/ kg. The pretreatment of this method is simple. Meanwhile, after treatment, it is proved to have less interference from impurities, be high sensitivity, strong applicability and this method can be used in the detection of Imidacloprid residues in most fruit, vegetables and plant tissue [4-6].

2.2 Solid Phase Extraction (SPE) by Ion Exchange-High Performance Liquid Chromatography Method

It used column purification after liquid-liquid distribution by dichloromethane and Strong Cation exchange- Solid Phase Extraction (SCX-SPE). Wu Di, et al. detecting by Solid Phase Extraction (SPE) by ion exchange-High performance liquid chromatography method and then found that the recovery rate of peel Imidacloprid was from 83% to 103% and its relative standard deviation (RSD) was less than 10%. The detection limit of this method was 0.05 mg/kg [7].

2.3 Liquid-Phase Time-of-Flight Mass Spectrometry (LC/MSD TOF) and Liquid Chromatography/Ion Trap Mass Spectrometry (LC /MSD Trap)

Imma Ferrer, et al. used this method to determine Imidacloprid. They found that LC /MSD TOF was a powerful tool for identification of pesticides in vegetables and provided new analytical skills for environmental food chemistry. LC /MSD TOF can be used to quantitative analysis of two or three order of magnitude. Its accuracy was less than 3mg/kg (normally less than 2mg/kg). The method can perform identification of standards, elemental composition of fragment ions and structural debris, especially identified by MS/MS ion trap. The limit of chlorine-neonicotinoid insecticides residues in salad vegetables was from 0.003mg/kg to 0.01μg/g. This concentration is equal or even superior to the indicator of the European Union about insecticides in the vegetables [8].

2.4 Reversed-Phase High Performance Liquid Chromato -graphy (RP-HPLC)

Liu Guosheng and others chose Spherisorb C18 Column and made the mobile phase which was Acetonitrile-Water (its volume ratio was 27:33). Its flow rate was 0.6 mL/min. The detection wave length was 254 nm. Relative standard deviation was 0.33% when determinate Imidacloprid quantitatively. And the recovery rate was between 96.9% and 103.0% [9].

2.5 Rapid Pressure Solvent Extraction-High Performance Liquid Chromatography

Under the condition of 100 Pa extraction pressure and 80 ℃, the average recovery rate was bigger than 88% and its standard deviation was smaller than 25%. Compared to the traditional method of extracting Imidacloprid from the soil, this method's PSE recovery rate was higher than ultrasoundt extraction and oscillation extraction. Reproducibility and recovery rate of this were good or even better than Soxhlet extraction method. Meanwhile, PSE avoids the promble of repeated cleaning by using ultrasound extraction or oscillation extraction and then saves solvent which makes it spare more time than Soxhlet extraction method.

3. Electrochemical Method

Glassy Carbon Electrode Voltammetry: This method researched the electrochemical behavior and determination of Imidacloprid at the glassy carbon electrode. A sensitive reduction peak can be observed when volt was 1.24 V (vs.Ag/AgCl) in the NH3-NH4Cl bottom liquid of 0.2 mol/L by using Square Wave Voltammetry (SWV) method to detect

Imidacloprid. There was good linear relationship between this peak current and the density of Imidacloprid from 6.72 $\times 10^{-6}$ mol/L to 1.68 $\times 10^{-4}$ mol/L. After conducted six parallel experiments for the Imidacloprid solid which concentration was 1.38 $\times 10^{-5}$ mol/L, the relative standard deviation was 2.36 %. [7-8] Prussian blue carbon nanotubes modified glassy carbon electrode method: Used the Glassy carbon electrode modified by Carbon nanotubes to be the bottom liquid which was 0.1mol/L NH3-NH4Cl buffer solution. Then determing Imidacloprid pesticide by Linear Sweep Voltammetry method. In the range of 1.34×10^{-7} mol/L to 2.94×10^{-5} mol/L, the peak current appeared in 1.04 V and the concentration of Imidacloprid showed a good linear relationship. This method's detection limit was 5.0×10^{-8} mol/L [8].

4. Other Methods

Micellar Electrokinetic Capillary Chromatography (MECC): Linear range was from 0.156 to 16.0 mg/L and the detection limit of Ephedra Imidacloprid detected by this method was 0.08mg/L. The average recovery rate was 93.8% and relative standard deviation was 4.20% [9].

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