COMPARATIVE STUDY ON CLEANUP PROCEDURES FOR THE DETERMINATION OF ORGANOPHOSPHORUS PESTICIDES IN VEGETABLES

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Abstract

A study was carried out to compare the cleanup procedures for the determination of organophosphorus pesticides in vegetables. Eleven organophosphorus pesticides were extracted with acetone and methylene chloride. Extracts were cleanup by solid-phase extraction (SPE) mixed-mode column using quaternary amine and aminopropyl (SAX/NH_2) or octadecyl (C_{18}) sorbents. The pesticides were determined by gas chromatography with flame photometric detector. The recovery results obtained from the SPE SAX/NH_2 and C_{18} cleanups in carrot, cucumber and green mustard samples were in the range of 71.0 % to 115 %. Lower recoveries were obtained for polar pesticides, methamidophos and dimethoate. These results were compared to the method currently used in the laboratory which does not include any cleanup**.**

Abstrak

Satu kajian telah dijalankan untuk membandingkan kaedah-kaedah pembersihan untuk menentukan racun perosak organopfosforus di dalam sayur-sayuran. Sebelas racun perosak organofosforus diekstrak dengan aseton dan dwikloro metana. Ekstrak dibersihkan dengan pengekstrakan fasa pepejal (SPE) menggunakan amina quaterina dan aminopropil (SAX/NH_2) atau turus oktadesil, C_{18}. Racun perosak ditentukan dengan kromatografi gas yang dilengkapkan dengan pengesan fotometrik nyala. Pengembalian racun perosak dalam tiga jenis sayur-sayuran iaitu lobak merah, timun dan sawi hijau adalah di antara 71.0 % dan 115 %. Pengembalian yang rendah diperolehi untuk racun perosak yang berkutub iaitu methamidophos dan dimethoate. Keputusan ini dibandingkan dengan kaedah tanpa pembersihan yang digunakan di makmal pada masa ini.

Introduction

Organophosphorus (OP) pesticides have replaced the organochlorine pesticides due to concern regarding the persistence and polluting effect of these compounds to the environment. However, due to its low persistency, a greater number of applications to a crop may be necessary during the course of the growing season. Numerous methods have been developed for the analysis of OP pesticides in fruits and vegetables. Some of these methods advocate the use of solid-phase extraction (SPE) cartridges. A method using acetonitrile for extraction of pesticide residues in fruits and vegetables was reported [1]. The pesticides were detected on gas chromatograph (GC) equipped with flame photometric detector (FPD). A simple and efficient cleanup method for GC determination of twenty-three OP pesticides in crops was reported [2]. The sample was extracted with acetone and benzene. Cleanup was performed on silica catridges. It was reported that water-soluble pesticides such as dichlorvos and dimethoate gave poor recoveries in all crops. A method for the determination of twenty-eight OP pesticides in fatty and non-fatty foods was reported [3]. Extraction was carried out using acetone and mixture of acetone-water. Carbon-celite was used as cleanup. A multi-residue method for determination of forty-three OP insecticides in plant and animal tissues was reported [4]. The OP insecticides were extracted with methanol-dichloromethane (1 : 9) and cleaned up using gel permeation chromatograph and silica gel mini columns. Determination of OP pesticides in fruits and vegetables using octadecyl, carbon and aminopropyl cartridges was reported [5]. The pesticides were determined by GC equipped with mass selective detector. Gravity-fed C_{18} SPE as cleanup for detection of pesticides in spinach, oranges, tomatoes and peaches was also reported [6]. The method was used to analyze forty-eight OP pesticides. A method using SPE cleanup of chlorpyrifos, methidathion and methyl parathion in oranges was reported [7]. Samples were extracted with anhydrous sodium acetate with ethyl acetate. The ethyl acetate extract was concentrated and cleaned up by passing through tandem