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Original Research Article

Development of a green liquid–liquid microextraction method using a solid disperser performed in a narrow-bore tube for trace analysis of some organophosphorus pesticides in fruit juices

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ABSTRACT

A green analytical approach for the determination of trace amounts of organophosphorus pesticides has been proposed using solid-based disperser liquid–liquid microextraction performed in a narrow-bore tube followed by gas chromatography–flame ionization detection. In this method, a sugar cube and acetone (at μL-level) are used as solid disperser and co-disperser, respectively, to facilitate formation of a cloudy state and accelerate mass transfer of the analytes from aqueous solution into the organic phase. The proposed method made possible the determination of analytes in the range of 2–1.0×10^4 μg L^{-1} with good linearity (coefficients of determination, ≥0.996) and favorable repeatability (relative standard deviation <5% for both intra-day, $n = 6$, and inter-day, $n = 4$, at a concentration of 50 μg L^{-1} of each analyte). Moreover, detection limits and enrichment factors of the analytes ranged from 0.2 to 1.4 μg L^{-1} and 466 to 616, respectively. Relative recoveries (72–102%, obtained at three fortification levels) confirmed the usefulness of the method for analysis of the analytes in fruit juices. The method was shown to be fast, reliable, and environmentally friendly with low organic solvent consumption compared to conventional dispersive liquid–liquid microextraction.

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1. Introduction

Organophosphorus pesticides (OPPs), as widely used inhibiting chemicals in the agricultural field, are preferred over other pesticides due to better activity against pests and relatively moderate environmental persistence. OPPs do decompose in the environment to some extent, but residues of these compounds can remain in the environment and contaminate surface water along with fruits and vegetables as a result of extensive and incorrect applications. Because of their high toxicity and potential risk for human health, development of simple, rapid and sensitive analytical methods for both identifying and determining OPPs has become an important issue.

On the other hand, for the analysis of pesticides, sample preparation is a necessary step to separate the analytes from complex matrices, concentrate them and obtain high sensitivity. Commonly used sample preparation methods are liquid–liquid extraction (LLE) (Tahboub et al., 2005; Wang et al., 2008), and solid-phase extraction (SPE) (Ballesteros and Parrado, 2004; García-Ruiz et al., 2005; Juan-Garcia et al., 2005; Zhu et al., 2005). These traditional methods are time-consuming, labor-intensive, expensive and require large volumes of hazardous organic solvents. Thus, much attention is being paid to develop more efficient, simple and solvent-free sample preparation methods or those employing fewer organic solvents such as solid-phase microextraction (SPME) (Fernández et al., 2001; Rodrigues et al., 2011; Tsoukali et al., 2005), stir bar sorptive extraction (SBSE) (Farajzadeh et al., 2010; Liu et al., 2005) and different types of liquid-phase microextraction (LPME) techniques, including single-drop microextraction (SDME) (Ahmadi et al., 2006; Xiao et al., 2006), hollow-fiber liquid-phase microextraction (HF-LPME) (Chen and Huang, 2006; González-Curbelo et al., 2013) and dispersive liquid–liquid microextraction (DLLME) (Farajzadeh et al., 2011a,b; Pizarro et al., 2011; Rodríguez-Cabo et al., 2011; Zgoła-Grześkowiak and Kaczorek, 2011).

Abbreviations: DLLME, dispersive liquid–liquid microextraction; EF, enrichment factor; FID, flame ionization detection; GC, gas chromatography; LOD, limit of detection; LOQ, limit of quantification; LPME, liquid-phase microextraction; MS, mass spectrometry; OPP, organophosphorus pesticide; RSD, relative standard deviation; SDME, single drop microextraction; SPE, solid-phase extraction; SPME, solid-phase microextraction.

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DLLME, presented by Rezaee et al. (2006), as a remarkable approach to LPME, has greatly contributed in miniaturization of the sample preparation step and reducing the time needed for the sample pretreatment that is a considerable factor for an analytical procedure. This method is based on rapid injection of extraction and disperser solvents mixture into an aqueous sample solution containing the target analytes. By this action a cloudy solution is created which increases the contact area between the extraction solvent and aqueous phase leading to a quick extraction procedure. Finally, the extractant can be separated by centrifugation. DLLME provides many advantages such as rapidity and simplicity of operation, high enrichment factor (EF), and low cost.

There are numerous reports about applications of DLLME in preconcentration of OPPs followed by an instrumental analysis (Berijani et al., 2006; Carro et al., 2012; Cunha et al., 2009; Zhao et al., 2007). Despite its merits, however, the use of a disperser solvent at mL-level is a noticeable issue that increases solvent consumption and solubility of the analytes in aqueous solution and hence reduces partition coefficients of polar analytes into the extraction solvent. Therefore during the past few years other microextraction approaches, such as vortex-assisted liquid–liquid microextraction (VALLME) (Pizarro et al., 2014; Yang et al., 2011; Zacharis et al., 2012; Zhang and Lee, 2012), air-assisted liquid–liquid microextraction (AALLME) (Farajzadeh and Khoshmaram, 2013; You et al., 2013), ultrasound-assisted DLLME (Fontana et al., 2010; Guo and Lee, 2012; Regueiro et al., 2008), and salt-assisted liquid–liquid microextraction (SALLME) (Gupta et al., 2009; Ma et al., 2014) were reported. These techniques have all been developed and have received considerable attention, as these methods show many advantages such as reduction in solvent consumption and high EF and extraction recovery (ER).

The aim of this work was to develop a reliable and environmentally friendly liquid–liquid microextraction method using a solid disperser performed in a narrow-bore tube combined with gas chromatography–flame ionization detection (GC–FID) and gas chromatography–mass spectrometry (GC–MS) for the extraction, preconcentration and determination of some widely used OPPs residues (including dichlorvos, diazinon, chlorpyrifos, profenofos and phosalone) in fruit juice samples. In the presented method, despite conventional DLLME methods, the use of mL-volume of an organic disperser solvent is avoided and the dispersion state is produced by a sugar cube as a solid disperser in the presence of a tiny amount (μL-level) of acetone as a co-disperser. This combination provides an efficient dispersion of extraction solvent into a large volume of aqueous phase (43 mL) by creating numerous microdrops of the extractant. This leads to good extraction efficiency. The influence of different operational parameters on extraction performance of the target analytes is systematically investigated and optimized. The main advantages of the proposed method against the conventional DLLME methods are twofold: a safe substance (sugar cube) instead of disperser solvent is utilized; and a smaller extraction solvent volume (25 μL of toluene) is consumed despite using a large aqueous sample volume. Also, this method is performed in a narrow-bore tube instead of a test tube, and there is no need for a centrifuging step.

2. Materials and methods

2.1. Chemicals and standard solutions

OPPs (dichlorvos, diazinon, chlorpyrifos, profenofos, and phosalone) with purity of >98% were kindly supplied by GYAH Corporation (Karadj, Iran). Analytical grade methanol, *n*-octanol, *n*-hexane, acetonitrile, acetone, dimethyl formamide (DMF), dimethyl sulfoxide (DMSO), sodium chloride, hydrochloric acid, and sodium hydroxide were purchased from Merck (Darmstadt, Germany). *n*-Hexanol and toluene were from Fluka (Buchs, Switzerland). Different types of sugar (as disperser) produced by various manufacturers were purchased from local supermarkets (Tabriz, Iran). Sorbitol sachets were obtained from Pharmachemie Pharmaceutical Company (Tehran, Iran). De-ionized water was obtained from Ghazi Company (Tabriz, Iran) for preparation of aqueous solutions. A 1000 mg L^{-1} (each OPP) mixture stock solution was prepared in acetonitrile and stored in a refrigerator at 4 °C. Fresh working solutions were prepared daily by dilution of the stock solution with de-ionized water to the required concentrations. Also a 1000 mg L^{-1} (each analyte) standard solution of the analytes was prepared in toluene for quality control of system and calculation of some analytical parameters (EF and ER) by its direct injection into the chromatographic system (three times in a day).

2.2. Apparatus

Analysis of OPPs was performed using a Shimadzu GC-2014 gas chromatograph (Kyoto, Japan) equipped with an FID and a split/splitless injector. A CP-Sil-8 (5% diphenyl 95% dimethyl polysiloxane) capillary column 30 m × 0.25 mm i.d. with a 0.25 μm film thickness (Chrompack, Middleburg, the Netherlands) was used for the separation. Helium (99.999%, Gulf Cryo, United Arab Emirates) was used as the carrier gas at a constant linear velocity of 30 cm s^{-1} and make up gas at a flow rate of 30 mL min^{-1}. The injector temperature was constant at 300 °C. Injections (1 μL) were done in a splitless mode (sampling time, 1 min). The oven temperature was set initially at 70 °C (held for 1 min) and then elevated to 300 °C at a rate of 12 °C min^{-1} (held for 4 min). The FID temperature was maintained at 300 °C. Hydrogen gas for FID was generated with a hydrogen generator (OPGU-1500S, Shimadzu, Japan) at a flow rate of 40 mL min^{-1}. The flow rate of air for FID was 300 mL min^{-1}. GC–MS analysis was carried out on an Agilent 7890A gas chromatograph equipped with a 5975C mass-selective detector (Agilent Technologies, CA, USA) and a split/splitless injector operated at 300 °C in a splitless mode with a sampling time of 1 min. Helium was used as carrier gas at a flow rate of 1.0 mL min^{-1}. Capillary column and temperature programming used in GC–MS were the same as those used in GC–FID analysis. Moreover, pH measurements were performed with a Metrohm pH meter model 654 (Herisau, Switzerland). A D-7200 centrifuge from Hettich (Kirchlengern, Germany) and an LBS2 ultrasonic bath (FALC Instruments, Treviglio (BG), Italy) were used in the sample preparation procedure.

2.3. Samples

Packaged samples of apple juice, grape juice, orange juice, and mango juice (Sunich Brand, Saveh, Iran) and fresh tomato, onion, and cucumber were purchased from a local store (Tabriz, Iran). It is noted that one sample of each juice was analyzed in triplicate. Packaged juice samples were exposed to the proposed micro-extraction method without any pretreatment, except mango juice. It was centrifuged at 4000 rpm for 5 min and then the supernatant was diluted as a ratio of 1:2 with de-ionized water before the microextraction procedure was performed. Tomato, onion and cucumber samples were squeezed by a juice extractor (JE600T, Kenwood, England). The obtained juices were centrifuged at 4000 rpm for 5 min and then the supernatants subjected to the proposed procedure.

2.4. Microextraction procedure

Microextraction procedure was performed in a narrow-bore glass tube (100 cm × 8 mm i.d.) with closed bottom using a septum. Prior to the implementation, the narrow-bore tube was

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