Dispersive liquid–liquid microextraction based on solidification of floating organic droplet coupled with HPLC-DAD for analysis of diazinon, chlorpryfos and phosalone residue in apple juice samples

Yousef Nikkhahi and Ramin Maleki*

Research Department of Analytical Chemistry, Iranian Academic Center for Education, Culture & Research (ACECR), Urmia Branch, Urmia, Iran.

Organophosphorus pesticides are widely used in treatment of insects for increasing horticultural production. Therefore, their residue in agricultural products such as fruits and fruit juices can become a risk to humans [1]. Among the analytical methods, high performance liquid chromatography with diode array detector (HPLC-DAD) is effective in used in pesticide residue analysis [2,3]. The direct analysis by HPLC-DAD is limited to mgL^{-1} levels so pesticide residue analysis requires a pre-concentration step in order to improve the analytical detection limit. In this work, a simple liquid phase microextraction method based on solidification of floating organic droplet in conjunction with HPLC-DAD has been developed for the quantitative analysis of diazinon, chlorpryfos and phosalone residue in apple juice samples. The effect of extraction and disperser solvent (nature and volume), extraction time, extraction temperature and salt effect was investigated. Under the optimal conditions (Extraction solvent: 50 µL of 1-undecanol; disperser solvent: 500 µL of acetone; extraction time: 1 min.; temperature (25±1°C) and NaCl (2%w/v)) the linear dynamic ranges were: 5.0-50.0 $\mu g\ L^{-1}$ for diazinon, 2.5-50.0 $\mu g\ L^{-1}$ for chlorpryfos and 0.5-50.0 $\mu g\ L^{-1}$ for phosalone. The relative standard deviation (RSD, n=5) for all analytes were below 6.5 % and the recovery values were in the range of 94-97%.

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Determination of lanthanum with liquid –liquid microextraction based on solidification organic phase by spectrophotometry in real sample.

M. Masrournia*[1], M. Chamsaz [2], V. ghasamsham[1], A. Mohamadhasan[1]

[1]*Department of Chemistry, Faculty of Sciences, Mashhad Branch, Islamic Azad University, Mashhad, Iran.*
[2]*Department of Chemistry, Faculty of Sciences, Ferdowsi university of Mashhad, Mashhad, Iran*
E-mail:masrournia@yahoo.com, masrour@mshdiau.ac.ir

Interest in the development of methods for the determination of actinides and lanthanides in natural and industrial samples remains constant. The main restrictions in determination of the inner transition elements in environmental samples come from the high content of the matrix and the extremely low concentration levels of the elements in those samples, which are below the detection limits of the conventional instrumental techniques. Therefore, an efficient separation and preconcentration technique is frequently required for determination of these elements in complex matrices. In this project, we used liquid –liquid microextraction, which organic solvent is solidified using an ice bath. As a novel sample preparation this method combining sampling, extraction, and pre-concentration all together. Compared with traditional extraction methods it has the advantages of simplicity of operation, rapidity, low cost, high recovery and high pre-concentration factor. However, the main drawback of the proposed method is the limitation of selecting an extracting solvent, because just a few organic solvents are close to the melting point of room temperature. In this study was used morin (3,2,4,5,7-pentahydroxy flavone) as a reagent to form a complex with lanthanium. All the parameters influencing the complex formation, microextraction, and determination were studied and optimized. At the optimum conditions, the LOD and RSDfor determination were 0.002 $\mu g\ mL^{-1}$ and 2.2 % respectively. The calibration curve was linear in the range of 0.0062 to 2.5 $\mu g\ mL^{-1}$ and the enrichment factor based on the slopes ratio after and before preconcentration was 48. The feasibility of the proposed method was successfully confirmed by the extraction and determination of lanthanium ions in real sample and suitable results were obtained.

Keyword: lanthanum, liquid –liquid microextraction, spectrophotometry, real sample

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Synthesis of polypyrrole /Fe_3O_4 for the magnetic solid phase extraction followed by dispersive liquid liquid microextraction of abamectin from water samples

Faezeh Khalilian[a,*], Mohammad Rezaee[b], Masoud KashaniGorgabi[a], Mohammad Farajvand[a]

[a]*Department of Chemistry, Shahr-e-Rey Branch, Islamic Azad University, Tehran, Iran*
[b] *Nuclear Fuel Cycle Research School, Nuclear Science & Technology Research Institute, Atomic Energy Organization of Iran, Tehran, Iran*

Abamectin is a widely used insecticide and anthelmintic. It is used to control insect and mite pests of a range of agronomic, fruit, vegetable and ornamental crops. Abamectin is also used as a veterinary antihelmintic. Abamectin has moderate to high acute toxicity and the main target organ for abamectin is the nervous system. [1]

In this study a magnetic solid phase extraction method using polypyrrole /Fe_3O_4 nanocomposite, as a novel sorbent, along with dispersive liquid liquid microextraction was developed to the extraction of abamectin from aquatic media. Firstly Fe_3O_4 magnetic nanoparticles (MNPs) coated by polypyrrole (PPy) was successfully synthesized. [2,3] and then it was characterized using scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FT-IR). An amount of 50m g of the entire nanocomposite was used for the first step of extraction from 5-ml sample. After extraction for a specified time, the sorbent was isolated from solution using a 1.4 T magnet. The sorbent was eluted with methanol as a desorption solvent. Following, the primary extractor was mixed with CCl_4 as a next step extraction solvent. The entire solution, which contains abamectin, was rapidly injected into 5 ml double distilled water. After centrifugation of cloudy solution, the sedimented phase was injected into HPLC-UV system. The parameters affecting extraction process including the amount of Fe3O4@PPy particles, salt effect, extraction time along with desorption solvent and time were optimized using one at a time strategy. Under optimized condition, an enrichment factor of 602 along with an acceptable RSD and a suitable dynamic range was obtained.

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