

Titania-based second-generation fabric phase sorptive extraction media: Synthesis, characterization, and preliminary evaluation

Natalia Manousi¹, [Antonio Ferracane](#)², Natasa P. Kalogiouri¹, Kenneth G. Furton³, Peter Q. Tranchida², Luigi Mondello^{2,4}, George A. Zachariadis¹, Erwin Rosenberg⁵, Victoria F. Samanidou¹, Abuzar Kabir³

¹ Laboratory of Analytical Chemistry, Department of Chemistry, Aristotle University of Thessaloniki, GR-54124 Thessaloniki, Greece.

² Department of Chemical, Biological, Pharmaceutical and Environmental Sciences, University of Messina, Messina, Italy. E-mail: antonio.ferracane@unime.it

³ International Forensic Research Institute, Department of Chemistry and Biochemistry, Florida International University, Miami, FL, USA

⁴ Chromaleont s.r.l., c/o Department of Chemical, Biological, Pharmaceutical and Environmental Sciences, University of Messina, Messina, Italy

⁵ Institute of Chemical Technologies and Analytics, Vienna University of Technology, 1060 Vienna, Austria

Introduction

Currently, the development of new sorptive devices with improved efficiency has become a major focus of contemporary research in the field of sorbent-based microextraction techniques. Fabric phase sorptive extraction (FPSE) is a novel sample preparation technique that has established itself as a powerful tool for the analysis of food, biological and environmental matrices [1]. Traditionally, in FPSE sol-gel coated silica-based fabric substrates are used for the extraction of the target analytes from real samples. In this work, the synthesis and characterization of second-generation FPSE titania-based media is presented. The different FPSE membranes were evaluated for their ability to extract organophosphorus pesticides from apple juice samples, prior to their determination by gas chromatography-mass spectrometry (GC-MS).

Methods

Different sol-gel coated titania based FPSE membranes were synthesized. The novel FPSE media were characterized by Fourier-transform infrared spectroscopy (FT-IR) and scanning electron microscopy (SEM). The performance of the different titania-based FPSE membranes was evaluated towards their ability for the extraction of organophosphorus pesticides from apple juice samples prior to their determination by gas chromatography-mass spectrometry. The main factors that affect the extraction performance were investigated to ensure high extraction efficiency and increased sample throughput. Accordingly, the method was validated and applied for real sample analysis.

Results

Among the different sol-gel coated titania based FPSE membranes, sol-gel octadecyl coated membranes showed the best performance for the extraction of organophosphorus pesticides. The sol-gel octadecyl coated titania-based FPSE membranes were compared to the conventional silica-based analogues demonstrating promising results for their utilization in pesticides residue monitoring in complex matrices. Under optimum conditions, extraction of the target analytes was performed within 30 min under stirring at 800 rpm using 20 mL of sample, while elution was carried out by immersing the membrane in 400 μ L of acetone for 2 min. The FPSE-GC-MS method showed good linearity, accuracy, precision and sensitivity. Moreover, the sol-gel octadecyl coated titania based membranes were found to be reusable.

Innovative aspects

- Sol-gel coated titania-based FPSE membranes were prepared and characterized
- An FPSE-GC-MS method was developed for the extraction of organophosphorus pesticides
- The new extraction membranes exhibited superior performance

References

- [1] Kabir, A and Samanidou V. Fabric Phase Sorptive Extraction: A Paradigm Shift Approach in Analytical and Bioanalytical Sample Preparation, *Molecules* **2021**, 26, 865.