



# Fabric phase sorptive extraction: An innovative sample preparation approach applied to the analysis of specific migration from food packaging



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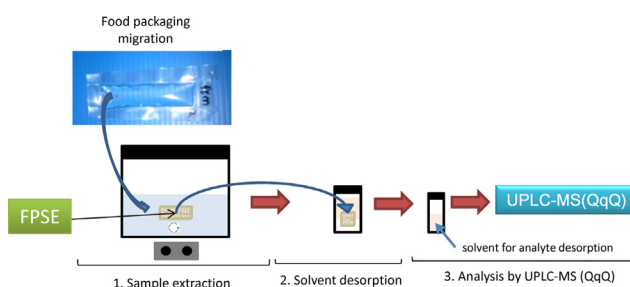
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## HIGHLIGHTS

- Fabric phase sorptive extraction is a quick and sensitive sample treatment approach.
- It was applied to the analysis of common plastic additives.
- Major fabric phase sorptive extraction (FPSE) parameters were optimized.
- FPSE was performed in liquid food simulants spiked with common plastic additives.
- Satisfactory results and good enrichment factors were obtained for most additives.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Additives added to food packaging materials can migrate to food in contact with them during storage and shelf life. A novel simple, fast and sensitive analyte extraction method based on fabric phase sorptive extraction (FPSE), followed by analysis using ultra-high performance liquid chromatography and mass spectrometry detection (UPLC-MS) was applied to the analysis of 18 common non-volatile plastic additives. Three FPSE media coated with different sol-gel sorbents characterized with different polarities including sol-gel poly(dimethylsiloxane), sol-gel poly(ethylene glycol) and sol-gel poly(tetrahydrofuran) were studied. All three FPSE media showed very satisfactory results. In general, compounds with low logP values seemed to have higher enrichment factors (EFs), especially with poly(tetrahydrofuran) and poly(ethylene glycol) media. For compounds with high logP values, the use of sol-gel poly(-dimethylsiloxane) improved the enrichment capacity. Sample preparation time was optimized at 20 min for sample extraction and 10 min for solvent desorption. Acetonitrile was selected as desorption solvent since recoveries were over 70% for 13 out of 18 selected compounds in all FPSE media. The best extraction recovery values were obtained when compounds were dissolved in aqueous acetic acid solution (3%), where 17 out of 18 compounds showed improvement in their signal intensity after FPSE extraction and 10 obtained enrichment factors above 3 for all the tested FPSE media. When FPSE extracts were concentrated under nitrogen, 11 out of 18 compounds reached EFs values above 100.

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## 1. Introduction

During the manufacture of food packaging materials, different additives such as antioxidants, stabilizers, slipping agents and plasticizers are commonly added to the plastic polymers in order to improve the material properties. The direct or indirect contact between food and the packaging material can end up in the transference of these chemicals from the packaging material to food, in a phenomenon called migration. Several works have shown the presence of these compounds such as antioxidants, biocides, plasticizers or other additives in food migrated from packaging [1–3] or tableware [4,5]. Not only intentionally added compounds can migrate to food, also non-intentionally added substances (NIAS) can be transferred from packaging to food [6–9]. These substances, called NIAS, may originate from different sources such as degradation of the polymer, degradation of polymer additives, impurities, neoformed compounds, contaminants or even products resulting from the interaction between the food and the packaging material [10,11]. Migrants can pose a health risk for consumers if they have a toxic effect and for this reason a strict evaluation of the food contact materials prior to launching them into the market is necessary. To comply with European legislation [12], migration tests are required. During migration tests, food packaging materials are brought in contact with a food simulant designed to mimic the mass transfer characteristics of food that will be in contact with the packaging. Food simulants can have an aqueous nature, such as simulant A (ethanol 10%) or simulant B (acetic acid 3%) or they can have a more hydrophobic nature such as simulant C (ethanol 20%), simulant D1 (ethanol 50%) or simulant D2 (vegetable oil). Extractables and leachables are also evaluated from pharmaceutical packaging materials due to their potential deleterious effects [13–15]. Since migrants are commonly at trace level, the importance of very sensitive analytical methods as well as highly effective preconcentration techniques cannot be overemphasized.

Several sample preparation techniques can be used for the extraction of compounds from liquid samples such as food simulants. Due to the minimal or zero consumption of toxic and hazardous organic solvents, potential for easy automation, simplistic operation and many other advantages over conventional exhaustive sample preparation techniques, solid-phase microextraction (SPME) [16] has positioned itself as a premier green sample preparation technique for volatile compounds. Nevertheless, its use is less common for the analyses of non-volatile compounds. Other option for the extraction of liquid samples is stir bar sorptive extraction (SBSE), but only two sorbent phases are commercially available [17]. The volume of extraction solvent in SBSE is much higher than in SPME (~50–250 times) and consequently a higher extraction sensitivity would be expected. However, this technique does not offer anticipated advantages over SPME [18], probably because mass transfer of the analyte through the sorbent is slow due to its thickness and viscosity. Considering the necessity of both higher sorbent loading as well as larger surface area to extraction-phase volume required to increase the sensitivity without prolonging extraction equilibrium time, thin-film microextraction (TFME) was introduced by Janusz Pawliszyn's research group [19]. Pristine poly(dimethylsiloxane) membrane film [19]; glass wool coated with poly(dimethylsiloxane)/divinylbenzene and Carboxen/poly(dimethylsiloxane) [20]; carbon mesh supported divinylbenzene/poly(dimethylsiloxane) [21] are among many different formats of TFME presented to date [22]. The new format of SPME has demonstrated higher extraction sensitivity compared to SBSE [23]. However, TFME films are not yet commercially available.

Fabric phase sorptive extraction (FPSE), has also been recently developed by Kabir and Furton [24,25]. It is a new generation

sample preparation technique that effectively addresses the majority of the shortcomings related to conventional sorptive sample preparation techniques. FPSE integrates the advanced material properties of sol-gel sorbents [25]; hydrophobic/hydrophilic property of natural or synthetic fabric as an active substrate, and the permeability of the substrate that creates a pseudo flow-through system, mimicking solid phase extraction to stimulate fast mass transfer from aqueous sample matrix to the FPSE media. High loading of sol-gel sorbents in the form of ultra-thin coating uniformly distributed on the fabric substrate, high primary contact surface area of the microextraction device, inherent capillary force of the substrate to transport aqueous/organic solvent through its surface, built-in pores to facilitate permeating aqueous solution through its body, complimentary hydrophilic affinity of the substrate to trap analytes for interaction in fabric phase sorptive extraction, synergistically promote near-exhaustive extraction in a short period of time. The same factors also facilitate quantitative analyte recovery when exposed to a small volume of suitable organic/organo-aqueous solvent for analyte back-extraction. Due to the strong covalent bond between the fabric substrate and the sol-gel sorbent, FPSE media can be exposed to any organic solvent or harsh chemical environment without compromising the integrity of the microextraction device. Due to the flexibility of FPSE media, it can be inserted directly into the aqueous sample matrix even in presence of high amount of particulates, debris, biomasses or other matrix interferences. FPSE is a simple sample preparation technique consisting of only two main steps: (1) sample extraction, where sample is put in contact with the FPSE media and analytes are retained in the sorbent and (2) solvent desorption, where FPSE media is brought in contact with a small volume of organic solvent and analytes are back-extracted to this solvent. One of the main advantages of FPSE is its high primary contact surface area (1000 mm<sup>2</sup>) for sorbent-analyte interactions. This fact and the possibility of enhancing the diffusion of analytes through the FPSE media with magnetic stirring or sonication make this technique a powerful system for obtaining fast extraction equilibrium. Other advantage includes the high number of sorbents available, with unique but different physico-chemical properties, that has allowed its application to different matrix and analytes.

FPSE has been applied recently to the analysis of estrogens in urine with very satisfactory results [26], the determination of non-steroidal anti-inflammatory drugs from environmental water samples [27]; triazine herbicides or emerging contaminants in environmental waters [28] [29], amphenicols residues from raw milk [30]; androgens and progestogens from environmental and biological samples [31]; residual sulfonamides from raw milk [32]; benzodiazepines in blood serum [33]; alkyl phenols in environmental samples [34] or UV stabilizers in sewage samples [35].

The main objective of this work was to develop a method based on FPSE and ultra-high performance liquid chromatography coupled to mass spectrometric detection, UPLC-MS(QqQ) for the specific migration analysis of some common non-volatile additives in packaging materials. In these work three different sol-gel coated FPSE media with different polarities provided by Dr. Abuzar Kabir's research group, were tested: sol-gel poly(dimethylsiloxane) (sol-gel PDMS, non-polar), sol-gel poly(tetrahydrofuran) (sol-gel PTHF, medium polar) and sol-gel polyethylene glycol) (sol-gel PEG, highly polar) and several extraction parameters such as sample extraction time, suitable solvent for solvent desorption, and solvent desorption conditions were optimized. The optimization was carried out for 18 common plastic additives (8 plasticizers, 5 antioxidants, 4 UV absorbers and 1 antistatic agent) and 3 different food simulants (A: ethanol 10%, B: acetic acid 3% and D1: ethanol 50%). Very good results in terms of recovery and reproducibility were obtained.

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