

Magnet Integrated Fabric Phase Sorptive Extraction (MI-FPSE): A Powerful Green(er) Alternative for Sample Preparation

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Abstract: Green(er) sample preparation technologies still dominate as the anticipated improvement in all analytical protocols. Separation scientists all over the world continuously strive to comply with the Green Analytical Chemistry (GAC) demands. To follow this trend, microextraction techniques are constantly evolving to bridge the gap between Green Analytical Chemistry and sample pretreatment. A research group from Florida International University, Miami, Florida has introduced fabric phase sorptive extraction (FPSE) in 2014 that was considered as a new milestone in microextraction technologies at that time. Two years later, the same research group introduced an advantageous innovative configuration that combines the stirring and extraction mechanism into a single sample preparation device, keeping all the benefits originally offered by classical FPSE. Magnet integrated fabric phase sorptive extraction (MI-FPSE) was eventually introduced as a new, advantageous implementation of FPSE. This device exhibits the advantageous role of the increase in extraction kinetics through sample diffusion, resulting in improved extraction efficiency of the microextraction device and supports the need for combining processes for better promotion and implementation of the principles of Green Analytical Chemistry. The applications of MI-FPSE are presented herein, showing the essential role that this technique can play in analytical and bioanalytical sample preparation.

Keywords: green sample preparation; sorptive extraction; fabric phase sorptive extraction; FPSE; magnet integrated fabric phase sorptive extraction; MI-FPSE



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1. Introduction

Due to the increasing concern of consumers as well as different regulatory agencies and environmental advocacy groups, Green(er) sample preparation technologies remain as the primary goal of any new sample preparation technology. Separation scientists across the world are working hard to develop new sample preparation technologies that would comply with the Green Analytical Chemistry (GAC) demands [1]. Green Analytical Chemistry, emerged in 2000, is an offshoot of Green Chemistry that advocates for making analytical laboratory practices more environmentally friendly. GAC has formulated 12 principles that include inhibition of waste, use of safer solvents, design for energy efficiency, decrease derivatization, and improve safety of the operator, among others.

To follow this trend, microextraction techniques are continuously evolving to simplify sample preparation workflow that would be better aligned with the green analytical chemistry principles.

In 2014, Kabir and Furton invented fabric phase sorptive extraction (FPSE) [2,3] which was considered as a new breakthrough in microextraction technologies at that time, while two years later, the same research group introduced an advantageous innovative configuration of FPSE that combines stirring and extraction mechanism into a single sample preparation device, keeping all benefits originally offered by FPSE. Magnet integrated fabric phase sorptive extraction (MI-FPSE) was eventually emerged as a new, advantageous

implementation of FPSE [4,5]. MI-FPSE, by virtue of its integrated magnet, provides rapid diffusion of the sample matrix and consequently quicker extraction equilibrium. As a result, it reduces the overall time needed for the sample preparation and improves the quality of the analytical data by improving the accuracy and precision of the sample preparation process. MI-FPSE also reassures the need for integrating processes that fulfil all Green Analytical Chemistry requirements [1]. Compared to classical fabric phase sorptive extraction that utilizes a single sol-gel sorbent coated membrane, MI-FPSE utilizes two circular disks of sol-gel sorbent coated FPSE membranes stitched together to house the cylindrical bar magnet. The FPSE membrane disks in MI-FPSE may have the same sorbent coating or different sorbent coatings. The possibility of using two different sorbent coatings may offer a huge advantage in analytical/bioanalytical sample preparation where the target analytes are broadly diverse in nature containing polar, nonpolar, and ionic species.

The primary objective of this article is to present the basic principle, available sorbent coatings and applications of MI-FPSE that appeared in the literature so far, showing the essential role that this technique can play in analytical/bioanalytical sample preparation.

Although sorbent based sorptive microextraction techniques such as solid phase microextraction (SPME), stir bar sorptive extraction (SBSE), microextraction by packed sorbent (MEPS) have demonstrated their advantages over classical solid phase extraction, they still suffer from several shortcomings such as (i) availability of limited sorbent coatings; (ii) lack of cation exchanger/anion exchanger/mixed mode sorbents; (iii) not suitable or recommended for field deployment; (iv) poor selectivity; (v) low thermal/solvent/pH stability; (vi) expensive; (vii) low sorbent loading, leading to poor sensitivity; (viii) high viscosity of the polymeric sorbent, resulting in slow mass transfer; (ix) inefficient coating technology, resulting in reduced batch-to-batch reproducibility; (x) prolonged sample preparation time. Fabric Phase Sorptive Extraction and Magnet Integrated Fabric Phase Sorptive Extraction have eloquently addressed most of the issues listed above.

2. Fabric Phase Sorptive Extraction (FPSE)

Fabric phase sorptive extraction was invented to address most of the sample preparation challenges that other sorptive microextraction techniques failed to offer individually. It unites the extraction principles of solid phase microextraction (SPME) (equilibrium-based extraction) and solid phase extraction (SPE) (exhaustive extraction). FPSE has adopted sol-gel coating technology for the sorbent coating on a flexible, porous fabric such as cotton/polyester/fiberglass substrate. Sol-gel coating technology chemically attaches the organic/organic-inorganic hybrid polymer to the fabric substrate by means of a sol-gel linker that results in unprecedented thermal, chemical, and solvent stability to the sorbent coating with pH stability ~1–13. The sol-gel coating technology, unlike classical polymer coating and immobilization technique used in SPME or SBSE, is a highly controllable chemical deposition process that provides unprecedented batch-to-batch reproducibility in the sorbent coating process. It is worthy to mention that extraction reproducibility between different SPME fibers and Twister stir bars are among many challenges the analysts face when using SPME or SBSE as the sample preparation techniques. In addition to superior batch to batch coating reproducibility in FPSE membranes, the porous morphology of the sol-gel sorbent facilitates the rapid permeation of the sample through the sorbent as well as the FPSE membrane and the analytes get extracted onto the sol-gel sorbent coated FPSE membrane via numerous analyte-sorbent intermolecular/ionic interactions. FPSE has developed more than 30 different sorbent chemistries that include polar, non-polar, medium polar, cation exchanger, anion exchanger, mixed mode cation exchanger, mixed mode anion exchanger and zwitterionic multi-mode sorbents. Depending on the nature of the target analytes, FPSE sorbents exploit London dispersion, dipole–dipole interaction, π - π interaction, hydrogen bonding and electrostatic interactions. Only very small volumes of organic solvent (100–500 μ L) are sufficient to achieve quantitative analyte back-extraction. The small volumes of the organic solvent as the eluent ensures high preconcentration factor

and remove the need of evaporation and sample reconstitution, thus minimizes analyte loss, and increases overall absolute recovery of the analytes [6].

3. Magnet Integrated Fabric Phase Sorptive Extraction (MI-FPSE)

MI-FPSE is an upgraded format of FPSE that was created (a) to improve the extraction reproducibility; (b) to expedite the analyte mass transfer from the bulk of the sample solution; (c) to eliminate the necessity of an external magnet and (d) to use more than one sorbent chemistry simultaneously in the same device in order to expand the extractive efficiency, in the case of various target analytes of different properties present in the sample (neutral, acidic, basic, polar, nonpolar). Due to the precise control of the spinning rate (rpm), MI-FPSE demonstrates higher reproducibility and improved absolute recovery values. The seamless and precisely controllable stirring speed facilitates the rapid permeation of the sample matrix through the FPSE membrane in MI-FPSE. These result in faster analyte mass transfer from the bulk of the solution to the FPSE membrane and consequently improves the rate of extraction in comparison to classical FPSE mode. Additionally, extraction equilibrium is achieved in substantially shorter time [4]. Compared to classical microextraction techniques, another major advantage of MI-FPSE is the ability to change the disk size from ¼" to 2" depending on the sample size. As such, a smaller MI-FPSE device is preferred for biological samples, whereas a large MI-FPSE device is recommended for environmental samples when large volume of sample can be collected easily.

Figure 1 presents the images of FPSE membranes, MI-FPSE devices and the extraction process.

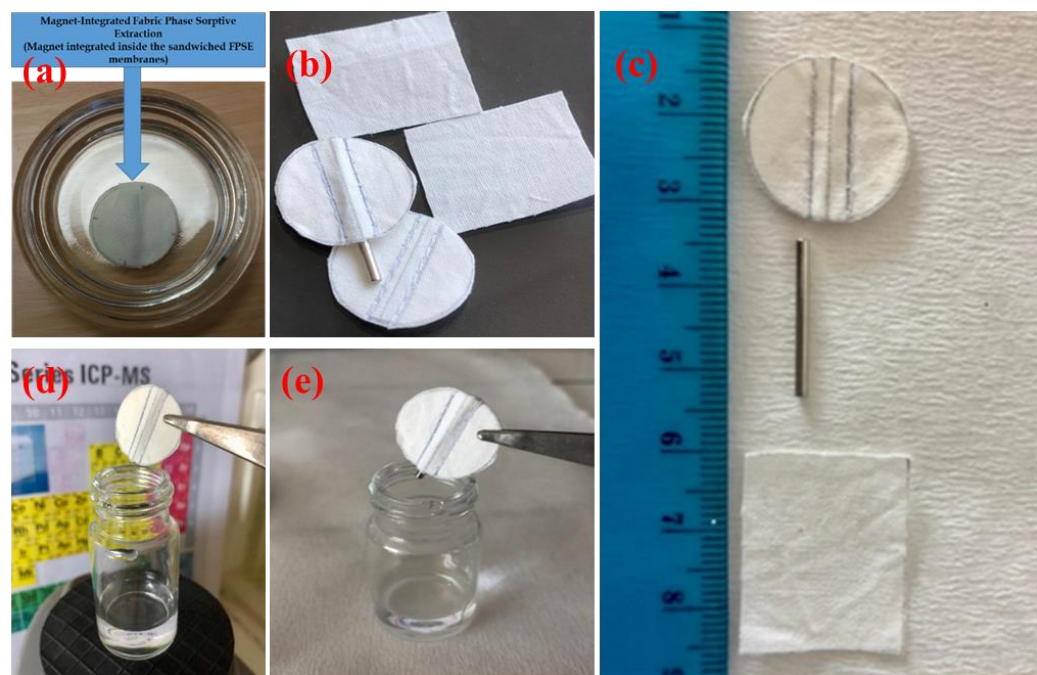


Figure 1. Fabric phase sorptive extraction and magnet integrated fabric phase sorptive extraction (MI-FPSE) membranes: (a) image of an MI-FPSE membrane; (b) side-by-side images of classical FPSE membranes and MI-FPSE membranes; (c) size comparison between classical FPSE membrane and MI-FPSE membrane; (d) extraction operation of MI-FPSE; (e) desorption operation in MI-FPSE.

4. Mechanism of Extraction

Unlike other microextraction techniques, MI-FPSE is unique in a sense that it incorporates the extraction mechanism of solid phase microextraction (equilibrium-based extraction) and solid phase extraction (exhaustive extraction) by design. MI-FPSE membranes are porous and permeable. The sol-gel sorbent coating does not obstruct the through pores of the fabric substrate. As a result, when MI-FPSE device spins inside the aqueous

sample matrix on a magnetic stirrer, it creates a strong vortex that forces aqueous samples to permeate through the MI-FPSE membrane, resulting in rapid analyte mass transfer from the bulk of the aqueous sample to the sol-gel sorbent coated on the membrane. The MI-FPSE membrane behaves like an SPE disk during the extraction process. At the same time, as the FPSE membrane is submerged into the sample matrix, it acts like an SPME fiber in its direct immersion extraction mode. Analytes diffuse from the bulk of the solution towards the MI-FPSE membrane and slowly establishes equilibrium. The combination of the extraction mechanism of SPME and SPE by design is one of the key advantages of MI-FPSE that is absent in other microextraction techniques. In addition, the overall selectivity of MI-FPSE is determined by (i) the substrate surface chemistry (hydrophobic or hydrophilic); (ii) nature of the functional group connected to the sol-gel precursor used in the sol solution formulation; and (iii) nature of the organic/inorganic polymer used in the sol solution. As a result, the selectivity of the MI-FPSE can be fine-tuned based on the properties of the target analytes [6]. This unique advantage is absent in classical microextraction techniques where pristine polymer (such as polydimethyl siloxane, polyethylene glycol) is the only source of the selectivity parameter that cannot be changed or modified.

Sol-gel based sorbents are intrinsically porous with sponge-like internal structure that provides very high specific surface area. As a result, a small volume of organic solvent (100–500 μL) can exhaustively elute the adsorbed analytes from the MI-FPSE membrane in minutes. The low volume of the eluent provides higher preconcentration factor and eliminates the requirement of solvent evaporation and sample reconstitution from the sample preparation workflow. As such, MI-FPSE is considered as a lean sample preparation technique.

A detailed, step-by-step procedure for creating FPSE membranes are presented elsewhere [7]. Several review articles on FPSE can also be consulted for obtaining a thorough insight of this emerging technology [8–10].

5. Building Blocks of Magnet-Integrated Fabric Phase Sorptive Extraction

Magnet-integrated FPSE membranes are comprised of several building blocks: (i) a fabric substrate; (ii) a sol-gel inorganic precursor/organically modified inorganic precursor; (iii) a sol-gel active inorganic/organic polymer and (iv) a cylindrical bar magnet. In addition to these building blocks, carbonaceous particles possessing high specific surface area such as Carboxen, graphene, carbon nanotubes can also be used in tandem with inorganic/organic polymers to enhance the selectivity parameter of the extracting sorbents.

The types and roles of different building blocks of MI-FPSE membranes are presented below:

(i). Fabric substrate:

In all classical sorbent-based sample preparation techniques, a neutral substrate is used that hosts the sorbent coating. For example, solid phase extraction (SPE) uses silica particles to coat the C_{18} , C_8 and other ligands. Solid Phase Microextraction (SPME) utilizes a small segment of fused silica glass rod that retains the polymeric sorbents. The substrates are inactive and do not contribute any role in the analyte extraction process. However, in MI-FPSE, the fabric substrate is not simply a support for the sorbent but also actively participates in the ultimate selectivity of the MI-FPSE membrane via hydrophilic/hydrophobic surface property. From a large number of potential candidates, cellulose, polyester and fiberglass fabrics are primarily utilized as the substrates for MI-FPSE membrane. Fabric substrates used in MI-FPSE holds sol-gel active functional groups that chemically binds the sol-gel sorbent networks during the sorbent coating process. Sorbent loading on the FPSE membrane primarily depends on the concentration of surface hydroxyl groups of the fabric substrate, therefore, the amount of sorbent loading per unit area of cellulose fabric is substantially higher than polyester fabric. During the extraction, MI-FPSE substrate acts like a fishing bait to attract the analytes via hydrophilic/hydrophobic surface property and induce the analytes to interact with sol-gel based high-efficiency sorbent. Due to the well-maintained porosity of the fabric, FPSE membranes mimic a solid phase extraction

disk. During the extraction process, aqueous samples containing the analyte(s) of interest permeates through the MI-FPSE membrane continuously and facilitate rapid and near exhaustive extraction. Figure 2 depicts the chemical structures of different FPSE substrates.

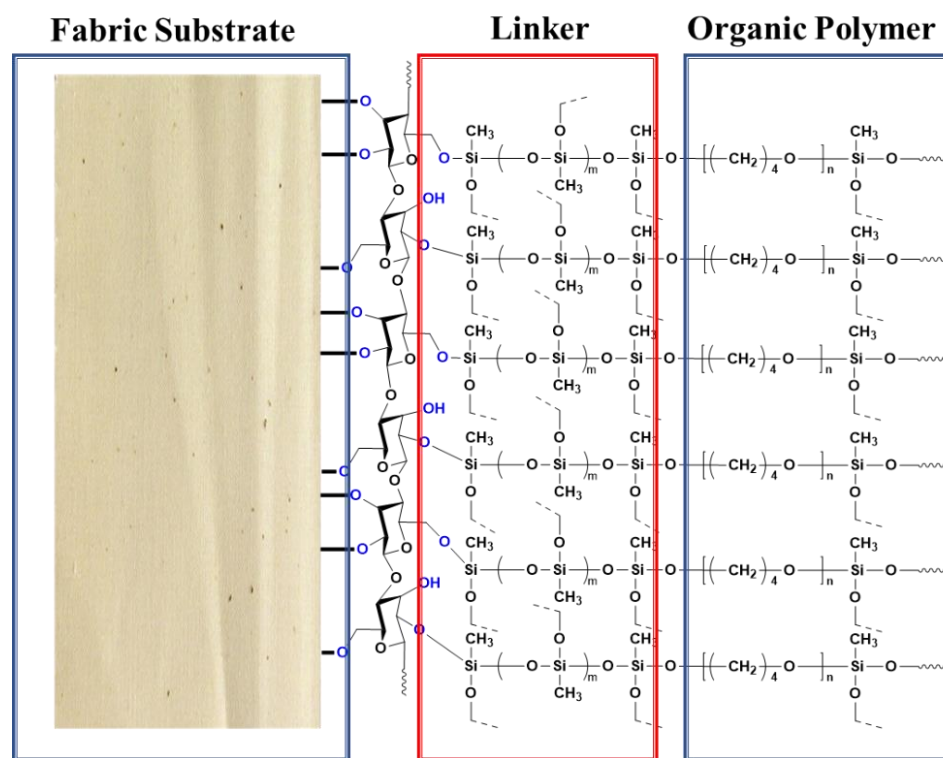


Figure 2. Building blocks of MI-FPSE membrane.

(ii). Inorganic/organically modified sol-gel precursor:

Inorganic/organic ligand containing silane precursor plays a very important role in sol-gel sorbent coating process. It forms the three-dimensional networks of sol-gel sorbent and randomly incorporates the inorganic or organic polymer into the expanding networks. It also serves as a linker to bind the sol-gel sorbent networks to the fabric substrate via condensation. When organic ligand containing silane precursor is used, the organic pendant moiety dynamically influences the ultimate selectivity of the MI-FPSE membrane. As a result, the ultimate polarity and selectivity of the MI-FPSE membrane can be adjusted as needed by a judicious selection of the sol-gel precursor. Among a large number of available so-gel precursors, tetramethoxysilane (TMOS), tetraethoxysilane (TEOS), methyl trimethoxysilane (MTMOS), phenyl trimethoxysilane (PTMOS), 3-aminopropyl trimethoxysilane (3-APTOS), octadecyl trimethoxysilane (C₁₈-TMOS), octyl trimethoxysilane (C₈-TMOS), titanium isopropoxide, zirconium isopropoxide, tetramethoxy germane are worthy to mention.

(iii). Sol-gel active inorganic/organic polymer

One major advantage of MI-FPSE is the commercial availability of a large number of inorganic and inorganic-organic hybrid polymers that can be used in sol-gel sorbent synthesis. Sol-gel active polymers such as poly(dimethylsiloxane) (PDMS), poly(dimethyldiphenylsiloxane), poly(ethylene glycol), poly(tetrahydrofuran), octadecyl trimethoxysilane (ODS-TMS), octyl trimethoxysilane (OTMS) are effectively integrated into the sol-gel networks via sol-gel synthesis and serves as the principal source of the selectivity and extraction affinity towards the target analyte(s). The polymer used in the sol-gel sorbent coating process provides diversified intermolecular interactions, which are ascribed to their functional makeup and broadly differs from one polymer to the other. As such, the proper

selection of the polymer in the sol solution design carry enormous weight to the success of the FPSE membrane for a given set of compounds.

6. Sorbents Developed for MI-FPSE

MI-FPSE has not only combined the extraction mechanism of solid phase extraction and solid phase microextraction by design, but it has also integrated the sorbents traditionally used for both the techniques. For example, SPME and SBSE traditionally use polymeric sorbents as their extraction sorbents, whereas, SPE utilizes organic ligands such as C₈, C₁₈ as the extraction sorbents. In addition, microextraction techniques do not offer clear solutions for ionizable compounds such as organic acids and organic bases. MI-FPSE has developed more than 30 unique sorbents, many of which were exclusively used either by SPE or by SPME and affiliated sample preparation techniques. The sorbents include nonpolar, medium polar, polar, cation exchanger, anion exchanger, zwitterionic, mixed mode and multi-mode chemistries. Table 1 presents a list of MI-FPSE sorbent chemistries with other pertinent information.

Table 1. MI-FPSE sorbent chemistries with other pertinent information.

MI-FPSE Sorbent		Sorbent Type	Sorbent Loading (mg/cm ²)/disk
1.	Sol-gel Polydimethylsiloxane (sol-gel PDMS)	Non-polar	2.30
2.	Sol-gel Polydimethyldiphenylsiloxane (sol-gel PDMDPheS)	Non-polar	1.93
3.	Sol-gel C ₁₂	Non-polar	3.54
4.	Sol-gel C ₁₈	Non-polar	4.88
5.	Sol-gel Graphene	Non-polar	N/A
6.	Sol-gel Multi-walled Carbon Nanotubes (sol-gel MWCNT)	Non-polar	N/A
7.	Sol-gel Polytetrahydrofuran (sol-gel PTHF)	Medium polar	3.96
8.	Sol-gel Polyethylene glycol-polypropylene glycol-polyethylene glycol (sol-gel PEG-PPG-PEG)	Medium polar	5.68
9.	Sol-gel Polypropylene glycol-polyethylene glycol-polypropylene glycol (sol-gel PPG-PEG-PPG)	Medium polar	5.25
10.	Sol-gel Methacrylate	Medium polar	4.51
11.	Sol-gel C ₄	Medium polar	1.90

Table 1. Cont.

MI-FPSE Sorbent		Sorbent Type	Sorbent Loading (mg/cm ²)/disk
12.	Sol-gel C ₈	Medium polar	2.33
13.	Sol-gel Polycaprolactone-polytetrahydrofuran-polycaprolactone (sol-gel PCAP-PTHF-PCAP)	Medium polar	4.64
14.	Sol-gel Polycaprolactone-polydimethylsiloxane-polycaprolactone (sol-gel PCAP-PDMS-PCAP)	Medium polar	6.14
15.	Sol-gel Polycaprolactone diol	Polar	3.46
16.	Sol-gel Polycaprolactone triol	Polar	N/A
17.	Sol-gel UCON	Polar	N/A
18.	Sol-gel Sucrose	Polar	N/A
19.	Sol-gel Sucralose	Polar	N/A
20.	Sol-gel Chitosan	Polar	N/A
21.	Sol-gel Carbowax 20 M (sol-gel CW20M)	Polar	8.64
22.	Sol-gel polyethylene glycol 10,000 (sol-gel PEG 10,000)	Polar	6.36
23.	Sol-gel polyethylene glycol 300 (sol-gel PEG 300)	Polar	4.45
24.	Sol-gel Cation exchanger	Cation exchanger	N/A
25.	Sol-gel Anion exchanger	Anion exchanger	N/A
26.	Sol-gel Zwitterionic sorbent	Cation and anion exchanger	N/A
27.	Sol-gel Mixed Mode sorbent	Medium polar, cation exchanger/medium polar, anion exchanger	N/A
28.	Sol-gel Zwitterionic-Multi Mode sorbent	Medium polar, cation exchanger, anion exchanger	N/A

7. Applications

Till now, there are only three publications appeared in the literature that reported the application of this technique to sample pretreatment.

The first application is a method developed by Alampanos et al. in 2021 [11] and refers to the magnet integrated fabric phase sorptive extraction of selected endocrine

disrupting chemicals from human urine and subsequent analysis by high-performance liquid chromatography–photodiode array detection.

In this research paper, the magnet integrated fabric phase sorptive extraction (MI-FPSE) device was evaluated to create a new analytical methodology suitable for the determination of selected and common endocrine disrupting chemicals (EDCs) as test analytes in human urine using high-performance liquid chromatography–photodiode array detector. The sol-gel Carbowax 20 M coated MI-FPSE device was identified as the most suitable membrane as the result of optimization experimentations in terms of elution solvent mixture, volume of the sample, extraction, and the elution time, stirring speed during the extraction, ionic strength, and pH of the sample matrix. The method was validated with regard to linearity, sensitivity, selectivity, precision, accuracy, and stability and efficiently applied to the bioanalysis of real samples. The MI-FPSE device proved its performance superiority over classical FPSE device and found to be very promising as a substantial progress in the field of analytical/bioanalytical sample preparation, especially in terms of greener sample preparation approaches.

Manousi et al. in 2022 [4] proposed the use of magnet integrated fabric phase sorptive extraction as a stand-alone extraction device to monitor benzoyl urea insecticides in water samples by high performance liquid chromatography–diode array detection (HPLC-DAD). Benzoyl urea insecticides, a group of pesticides often used in agriculture, that are persistent in environmental samples and therefore their monitoring is necessary to prevent adverse effects to human health and the environmental ecosystem. In this work, the application potential of MI-FPSE was assessed for the first time in environmental pollution monitoring. More specifically MI-FPSE was employed for the extraction and preconcentration of several benzoyl urea insecticides including diflubenzuron, triflumuron, hexaflumuron, lufenuron and chlorfluazuron from environmental water samples followed by their chromatographic determination by high performance liquid chromatography–diode array detection (HPLC-DAD). The most important parameters that may affect the performance of the newly developed methodology were comprehensively optimized and the MI-FPSE-HPLC-DAD method was validated.

Since the method favours handling of relatively high sample volume, high preconcentration factors can be achieved easily.

The validated method was then successfully employed for the analysis of several environmental samples including tap, mineral, river, and lake water samples. Additionally, the ComplexGAPI [12] evaluator tool was used to demonstrate the green potential of developed method.

Last but not the least, in 2022 Manousi et al. [5] demonstrated also the applicability of MI-FPSE in food analysis. In this work, MI-FPSE using a sol–gel poly(tetrahydrofuran) coated FPSE cellulose membrane was applied for the first time to selectively extract six triazine herbicides from herbal infusions prior to their determination by HPLC. After optimization of all crucial parameters that affect adsorption and desorption steps, the developed method was validated in terms of accuracy, precision, linearity, limits of detection (LODs) and limits of quantification (LOQs). Finally, the methodology was successfully applied for the analysis of several herbal infusion samples.

8. Conclusions

Magnet integrated FPSE has already proved its applicability in the field of bioanalysis, food analysis and environmental analysis.

More applications are expected to be developed in the near future by taking advantage of the wide variety of the FPSE sorbents that can be developed by sol-gel technology in order to selectively extract any compounds, polar or non-polar, in order to be analysed by any chromatographic separation technique.

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