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Review Article

Fabric phase sorptive extraction: A sustainable approach in analysis of pharmaceutical product

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ABSTRACT

As the pharmaceutical industry continues to evolve, there is an increasing demand for sustainable and efficient analytical techniques in the analysis of pharmaceutical products. This manuscript explores the application of Fabric Phase Sorptive Extraction (FPSE) as a novel and sustainable approach for the extraction and analysis of pharmaceutical compounds. FPSE, a recent advancement in sample preparation, offers a greener alternative by utilizing a fabric-like sorbent material. The environmentally friendly nature of FPSE, with reduced solvent consumption and waste generation, aligns with the principles of green analytical chemistry. Case studies involving the analysis of various pharmaceutical products showcase the versatility and applicability of FPSE in different matrices.

1. Introduction

1.1. Extraction

Extraction techniques are used to isolate a specific compound from a mixture. A few different extraction methods exist, such as liquid/liquid, liquid/solid, and acid/base extractions. A solute is isolated from impurities or unreacted starting materials by moving it from one phase to another. Natural product extraction has also used some more recent or environmentally friendly extraction techniques such as supercritical fluid extraction (SFC).[1], Microwave-assisted extraction (MAE), and pressurized liquid extraction (PLE).[2] Traditional extraction methods including maceration, percolation, and reflux extraction frequently employ organic solvents, demand a large quantity of solvent, and take a long time to complete.[3] The ambient temperature, initial particle size, solvent-to-ratio solvent, and time are just a few of the factor's characteristics, that might affect how effective the extraction is.[4,5]

1.2. Microextraction

Microextraction is an extractive method that uses a minimal extraction phase volume in comparison to the sample volume. Microextraction techniques are becoming increasingly popular in various research areas due to their simplicity, rapid sampling, high recovery, low cost, and high enrichment factor,[6] as well as environmental friendliness. Solid-Phase Microextraction (SPME), Liquid-Phase Microextraction (LPME), nanoparticle coatings, and packed sorbents (MEPS) are all fast-growing areas of microextraction technique that are yet to be fully explored.[7] Microextraction techniques facilitate the miniaturization, automation, onsite analysis, and time-effectiveness of sample preparation.[8] In the field of

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bioanalytical applications, The most recent developments in microextraction techniques for biomarker analysis have been reviewed.[9] There are several types including Dispersive Liquid-Liquid Microextraction (DLLME), Liquid-Phase Microextraction (LPME), and Solid-Phase Microextraction (SPME).[8]

1.3. Types of microextraction

1.3.1. Solid-phase microextraction (SPME)

SPME is a solventless technique involving Fibers coated with an extracting phase to extract different kinds of analytes from a variety of liquid or gaseous media.[10] In analyte extraction during equilibrium, Fibre is proportional to the sample concentration.[11] SPME is a thorough sample preparation method that offers several advantages over other sample preparation methods that have been around for a while, including reduced organic solvent use, simplicity, speed, and improved sample clean-up.[12] Because of these benefits, SPME has become a very popular technique in a variety of bio/analytical research fields, including environmental, clinical, biological, and food studies.[13]

1.3.2. Liquid-phase microextraction (LPME)

Liquid-Liquid Extraction (LLE) was miniaturized to create Liquid-Phase Microextraction (LPME), which drastically reduced the amount of solvent used.[14] Solvent film presents inside or at the tip of the needle dissolves analytes, forming a droplet.[9] The fundamentals of LPME are the idea that analytes of interest can be extracted or partitioned out of the sample matrix and placed in a small volume of an immiscible organic solvent, often one that is lighter than water.[15] After being collected, frequently via a micro syringe or a straightforward phase separation, this solvent phase containing the extracted analytes is then subjected to analysis using the necessary analytical instruments.[16]

1.3.3. Dispersive liquid-liquid microextraction (DLLME)

DLLME prepares samples using a mixture of three components: aqueous sample, dispersive solvent, and extraction solvent. DLLME extraction involves rapid injections of extracting and dispersing solvents into aqueous samples, followed by centrifugation**.** DLLME is based on the idea of creating a cloudy or turbid mixture by forming a fine dispersion of an extraction solvent (usually an organic solvent) within the sample solution. The analytes divide between the sample solution and the scattered organic solvent droplets in this emulsified condition. The organic solvent phase containing the extracted analytes is separated and collected for further analysis when this partitioning equilibrium has been established.[17]

Fabric phase sorptive extraction

Based on this observation and innovation, Kabir and Furton developed FPSE in 2014 as a novel, environmentally friendly technique for preparing samples.[18,19]Figure 1

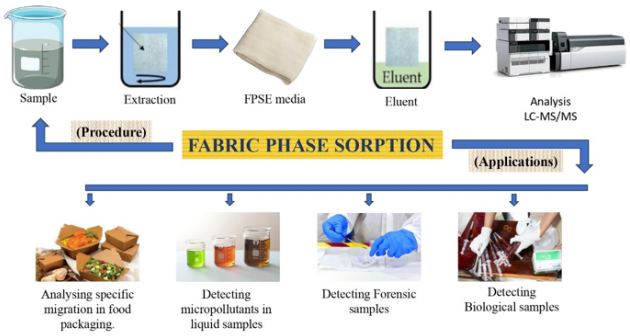

Figure 1: Typical FPSE workflow

Figure 1 illustrates typical workflow of FPSE. This innovative technique combines the chemical solution desorption coating technology advantages with the large surface area of fabric substrates in order to prepare samples with superior efficiency. A sample preparation method is called FPSE. which integrates the benefits of the chemical solution desorption coating technology and the rich surface chemistry of fabric substrates.[20] The working principle of FPSE is based on the kinetic principle of extraction, the extracting phase's volume can be increased by thickening the coated sorbent. will increase the amount of analyte extracted.[18] In FPSE, a fabric substrate is coated with a sol-gel material that selectively extracts the target analytes from a sample matrix. The coated fabric is then used as a Sorbent for analyte extraction from the sample. Then, the sorbent is desorbed of the isolated analytes. using a suitable solvent, and the resulting solution is analyzed using a suitable analytical technique.[20] FPSE has several advantages over traditional sample preparation techniques, including high extraction efficiency, low cost, and ease of use. It has been used to extract a variety of analytes from different sample matrices with success, including environmental, biological, and food samples.[18,21]

2. Methodology of FPSE

By removing all sample pretreatment and posttreatment processes, FPSE has significantly simplified the sample preparation procedure.[22] When the sample preparation procedure is underway, FPSE simultaneously employs an exhaustive extraction method and equilibrium-driven extraction.[23] The chemicals and materials required for FPSE include fabric substrates, sol-gel precursors, and solvents.

Making FPSE media with PEG coating using sol-gel: Fabric substrates should be surface-cleaned and activated. the process of preparing sol solutions for coating a substrate, and the development of sol-gel PEG coatings on the substrate are each step in the preparation of sol-gel PEG coated FPSE media. The FPSE and back-extraction solvent desorption and the creation of standard solutions for FPSE

are steps in the process.Method development in FPSE is simple, and In FPSE back-extraction, the amount of solvent used must be kept as low as possible.[23]

2.1. Principle and mechanism

The combination of several kinds of substrates made of natural or synthetic fabrics that have been chemically coated with an ultra-thin layer of hybrid organic-inorganic sorbents made of sol-gel provides its benefits in that there is no need for any matrix alteration or cleanup steps.[24] The resulting FPSE medium is versatile, porous, and has a noticeably increased major contact surface area, all of which aid in the rapid and efficient extraction of analytes. London dispersions, hydrogen bonds, dipole-dipole interactions, and p-p interactions are a few molecular interactions that help with the extraction of analytes. Target analytes can be concentrated using modest amounts of organic solvent without solvent evaporation or sample reconstitution.[25] As an add-on, a washing phase can be employed before the analytes are eluted to remove a variety of undesirable substances from the matrix before chromatography. until equilibrium has been achieved, analytes are transferred either in the sample headspace or in a matrix of samples. It takes a long extraction time to reach equilibrium since the extracting phases are viscous. Additionally, a low mass of sorbent (particularly in SPME) frequently prevents the extraction causing a significant buildup of target analytes, and lowering the sensitivity of the process as a whole.[26] If FPSE media are properly cleaned using an organic solvent system, allowed to dry, and kept in a dry environment, they can be used repeatedly without losing their ability to perform adsorption and extraction.[27]

According to their extraction mechanism, there are two categories of solid sorbent-based sample preparation techniques: (a) Exhaustive extraction, which is comparable to solid-phase extraction (SPE), involves completely extracting every analyte from the sample.[28] (b) Equilibrium extraction, on the other hand, as shown in solid-phase microextraction, entails creating an equilibrium between the analytes in the sample and the sorbent phase, which results in the extraction of analytes depending on their partitioning actions. (SPME).[17] Polymeric sorbents such as PDMS, PEG, and SBSE dominate classical microextraction techniques. Analytes are dissolved by extracting polymers during extraction. These adsorbent materials enhance the SPME Fibre coating's surface area and selectivity, improving its ability to extract and concentrate analytes from complex sample matrices.[29] With high viscosity, these polymers lead to slow mass transfer and extraction rate, especially with heavier analytes, these polymeric coatings can be enhanced by adding carbonaceous particulates like divinyl benzene and carboxen. In contrast, FPSE uses Organic and inorganic Through chemical bonding, polymers are connected to a fabric substrate. the sol-gel sorbent coating method.[30] Chemical solution desorption sorbent forms mesopores and micropores that form a porous 3D network.[31] The hydrophilic/hydrophobic surface properties of a fabric substrate pull aqueous samples and analytes during analyte extraction. The sol-gel sorbent and analytes interact with the FPSE membrane as they reach it. The sorbent loading is one significant distinction. Comparing FPSE to SPME, SBSE, and TFME, sorbent loading is often much higher in the former two. This increased loading enables FPSE to collect more analytes.[32,33] However, even after thorough extraction, FPSE might not make use of the entire analyte retention potential.

2.2. Instrumentation techniques used with FPSE

2.2.1. HPLC

HPLC can be used in conjunction with fabric phase sorptive extraction (FPSE) to analyse extracted analytes. FPSE is a Flexible, quick, and delicate micro-extraction device that can be used for environmental, food, and biological samples.[27] A study published in 2019 developed a novel chromatographic separation and sample preparation technique for the trace detection of cosmetic and environmental samples using FPSE and HPLC-photodiode array detection (PDA).[34] In this method, Before being separated and evaluated by chromatography, the analytes obtained from the FPSE membrane were eluted using methanol. Following filtration through a 0.45 μm porous filter, the resulting samples were then transferred into HPLC vials.[33]

2.2.2. UPLC-MS/MS

FPSE and UPLC-Tandem Mass Spectrometry can be used together[35]. Chemicals and materials for Fabric substrates should be surface-cleaned and activated, and the sol solutions should be ready for coating, and creation of the sol-gel PEG coated FPSE media are all part of the equipment needed for FPSE-UPLC-MS[36]. The approach includes applying sol-gel PEG coatings to the substrate, preparing standard solutions for fabric phase sportive extraction, and performing back-extraction and fabric phase sportive extraction. The identification of distinct chemicals in diverse sample matrices[37].

2.2.3. LC-MS/MS

liquid chromatography-tandem mass spectrometry (LC-MS/MS) can be used in conjunction with FPSE to analyse extracted analytes. In this method, before being separated and evaluated by chromatography analytes were eluted from the FPSE membrane using methanol, and then they were filtered through a porous PTFE filter. As a result, the extracted analytes may be detected effectively using LC-MS/MS[31].

2.2.4. GC/MS

GC is a useful tool for analysing organic molecules that are volatile and semi-volatile. The analytes are introduced to a gas chromatograph after desorption, where they are separated according to their volatility and engage in interactions with a stationary phase in a capillary column. The separated analytes are then detected and measured using a variety of detectors, such as a mass spectrometer (MS) or a flame ionization detector (FID)[22].

2.2.5. UV spectroscopy

UV spectroscopy can be used as a detection method for analytes extracted using FPSE. FPSE can be used in combination with HPLC-UV for the quantitation of analytes in human urine. UV spectroscopy can be a useful tool in FPSE instrumentation for the detection and quantitation of analytes in various samples[31,38]Int.

2.2.6. LC-FD

Liquid chromatography with fluorescence detection, often known as LC-FD, is a typical analytical technique for identifying and measuring the extracted analytes. For the detection of diverse chemicals in environmental samples, such as anthracycline, FPSE followed by LC-FD has been employed[39].

3. Development Stages of FPSE

3.1. Fabric substrate selection and pretreatment

FPSE, a microextraction method depends on the surface chemistry of fabric substrates. For nonpolar analytes, it entails selecting a hydrophobic polyester substrate, and for polar or medium-polar analytes, a hydrophilic 100% cotton cellulose substrate is used[40] Chemical solution desorption sorbents are attached to the fabric by chemical bonds, so the fabric must have surface hydroxyl functional groups. Important phases in this procedure include cleaning the fabric substrate to get rid of undesirable finishing compounds and treating it to add hydroxyl groups[41].

3.1.1. Chemical solution desorption sorbent coating on the treated fabric substrate

Sol-gel sorbent coatings are constructed from a) any number of inorganic or chemically altered sol-gel precursors. B) Organic/inorganic polymers that sol-gel. C) Solution compatibility. D) Acid catalyst, water to hydrolyze. There are many There are several organically altered precursors to silanes, but methyl trimethoxysilane is the most widely utilized[42]. Other preferred silanes both 3-aminopropyl trimethoxysilane and phenyl trimethoxysilane are precursors. In FPSE, polymers include poly (dimethyl diphenyl siloxane) (PDMDPS), poly (tetrahydrofuran) (PTHF), poly (dimethyl siloxane) (PDMS), and poly (ethylene glycol) (PEG)[43].

3.1.2. The dip coating technology

sol-gel dip coating is conducted with the chemical solution deposition prepared in 2 steps. Pretreated fabric segments are before beginning the coating procedure, submerged into the sol solution[44]. Following the immersion of the sol solution in the fabric substrate, the sol-gel coating process occurs. This process typically lasts 12 hours. Sol solution; The FPSE membrane covered with chemical solution deposition sorbent is allowed to air dry for an hour[45].

3.1.3. The FPSE membrane

The specially designed conditioning device integrated within the system is used to the FPSE membrane with an air-dried sol-gel sorbent coating for thermal conditioning. FPSE membrane is thermally treated for 24 hours with continuous helium gas flow in a gas chromatograph (GC) oven at 50°C with sol-gel sorbents[46]. An additional cleaning protocol is implemented to eliminate any unbonded components within the sol solution and reaction byproducts. Rinse the membrane in a 50:50 mixture of methylene chloride and methanol while sonicating for an hour. Once the rinsing solvent has been drained, the dried FPSE membrane is in the air for one hour before undergoing 24-hour thermal conditioning in helium at 50°C. As a result, the FPSE membrane has completed the Sol-gel coating technique[47].

3.1.4. FPSE membrane resize

With FPSE, the size of the membrane can be adapted to meet the needs of the analysis, unlike traditional approaches like SPME or SBSE. FPSE membrane discs with a diameter of 1 cm are sample for small sample volumes like blood, plasma, or saliva. It is advised to use a bigger membrane size, such as 5-20 mL, for samples larger than 2.5 × 2.0cm. Because of the increased contact surface area, a larger membrane size results in quicker extraction equilibrium, but it also requires more quantitative back-extraction with an organic solvent, conceivably diluted analytes before injection. Due to the elimination of the Reconstitution of the sample and evaporation of the solvent in FPSE, the amount of liquid utilised in FPSE back-extraction must be kept to a minimum[43].

4. Applications of FPSE

Innovative sample preparation techniques, such as Fabric Phase Sorptive Extraction, have been used in a variety of fields, including the processing of environmental, food, and pharmaceutical samples[48]. The high-efficiency extraction of various pharmaceutical chemicals from biological fluids like blood serum using a possible sample preparation technique that uses fewer solvents is FPSE[49].

Table 1: Use of FPSE for analysis of pharmaceutical products

Compound	Sample	Determination technology	Refences
Cytostatic drugs	Effluent WWTP	LC-MS/MS	50
NSAIDs	Ultrapure water	GC-MS	51,52
Fungicides	Ultrapure water	GC-MS/MS	53
Hormones	Effluent WWTP Hospital	LC-MS/MS	51
alkyl phenols	Wastewater, sewage water, groundwater, river water	GC–MS	54
Estrogens	Ultrapure water	LC-FD	39,55
Triazine herbicides	Ultrapure water	LC-DAD and LC-MS/MS	50,56
Organophosphorus pesticides	vegetable samples	GC-MS	57
Azole antimicrobial drug	Human plasma and urine	FPSE-HPLC-PDA	19,58
Benzodiazepines	blood serum	HPLC	59
Antidepressant	biological fluids	HPLC	60
Concentration of trace drug amounts, enhancing sensitivity	extract drugs and their metabolites in blood or urine.	HPLC-UV and GC-flame ionization detection	61
Fungicides	Pond wate	LC-MS	62
Tetracyclines	milk	HPLC-UV	63
Fluoroquinolones	Lake water, River water	LC-UV	64
Ketoconazole	human plasma and urine	HPLC-PDA	65
Ciprofloxacin	plasma	HPLC-PDA	66

5. Advantages of FPSE than other sample preparation techniques

1. *High selectivity:* The unique sorbent material used in FPSE allows for excellent selectivity for the target analytes[67].
2. *Low detection limit:* The high concentration of analytes on the sorbent material allows FPSE to achieve low detection limits[68].
3. FPSE can be used to extract a variety of analytes, including polar and non-polar compounds.[60]
4. Minimal sample preparation is necessary for FPSE, which can save time and lessen the possibility of sample contamination[20,69].
5. *Environmentally friendly:* FPSE is a technology that produces less waste and uses less solvent than conventional extraction techniques[45].
6. *Versatility:* A wide variety of sample matrices, including biological, environmental, and food samples, can be used with FPSE[70].

5.1. Limitations of FPSE

Fabric Phase Sorptive Extraction (FPSE), despite its numerous merits, is not without limitations. The availability of suitable sorbent materials for FPSE may be somewhat restricted, potentially limiting its versatility across diverse analytes. Complex sample matrices, particularly those found in biological samples or pharmaceutical formulations, can introduce matrix effects that may compromise the selectivity of FPSE. Saturation of the sorbent material, especially in the presence of highly concentrated samples, may result in diminished extraction efficiency and sensitivity. Compatibility issues between certain sample matrices or target analytes and FPSE conditions may hinder its effectiveness. Moreover, the extraction kinetics of FPSE may not be optimal for all analytes, possibly leading to extended extraction times. Challenges associated with efficient desorption of analytes from the sorbent material could affect the accuracy and reproducibility of the method. The scalability of FPSE for large-scale analyses and standardization across laboratories might be constrained by factors such as cost, equipment requirements, and variations in sorbent types. Additionally, limited commercial availability of FPSE devices and standardized sorbent materials could impact its widespread adoption, and while FPSE is considered greener due to reduced solvent usage, associated costs with acquiring specialized materials and equipment should be taken into account. Addressing these limitations is essential for a comprehensive evaluation of FPSE's suitability in pharmaceutical analysis[71,72].

5.2. Future scope

The future scope of Fabric Phase Sorptive Extraction holds significant promise in advancing pharmaceutical analysis. Ongoing research efforts can focus on expanding the range of available sorbent materials, enhancing their specificity for diverse analytes, and addressing matrix effects in complex samples. Further optimization of extraction kinetics and desorption efficiency, along with the development of standardized protocols, will contribute to the method's broader applicability and reproducibility. Exploration of FPSE in emerging fields, including environmental monitoring of pharmaceutical residues and point-of-

care diagnostics, offers exciting avenues. Collaborative endeavors between researchers, industry, and regulatory bodies are essential to propel FPSE into routine analytical practices, establishing it as a sustainable and efficient tool for pharmaceutical analysis in the years ahead[73,74].

6. Conclusion

SPME and SPE are sample preparation methods that are combined into a single platform by FPSE. To extract analytes from various sample matrices, FPSE use a permeable fabric, either natural or synthetic, coated with a sorbent material derived from sol-gel Regarding the extraction of analytes from biological, dietary, and environmental sample materials, FPSE has proven to be a reliable and efficient procedure. FPSE has several advantages over standard sample preparation techniques, such as reduced solvent use higher extraction yield, and simple method development. FPSE is a promising technique for removing a wide range of analytes from various sample matrices for removing a wide range of analytes from various sample matrices.

7. Abbreviations

(FPSE): Fabric phase sorptive extraction; *(SPE):*-solid-phase extraction; *(SPME):*-solid-phase microextraction; *(GAC):*- Green analytical chemistry; *(SFC):*-supercritical fluid extraction; *(MAE):*-Microwave-assisted extraction; *(PLE):*-pressurised liquid extraction; *(LPME):*-Liquid-Phase Microextraction; *(DLLME):*-Dispersive Liquid-Liquid Microextraction; *(PEG):*-poly(ethylene glycol); *PDMS:*-poly(dimethyl siloxane); *(SBSE):*- stir bar sorptive extraction; TFME:- thin film microextraction; *(HPLC-PDA):*- High-performance liquid chromatography photodiode array detection; *(UPLC-MS/MS):*-Ultra-Performance Liquid Chromatography-Tandem Mass Spectrometry; *(LC-MS/MS):*-liquid chromatography-tandem mass spectrometry (LC-MS/MS); *(GC/MS):*-Gas chromatography; *(UV spectroscopy):*-Ultraviolet–visible spectroscopy; *(LC-FD):*- liquid chromatography-fluorescence detection; *(PDMDPS*):-polymers include poly(dimethyl diphenyl siloxane); (*PTHF*):-poly(tetrahydrofuran);

8. Source of Funding

None.

9. Conflict of Interest

None.

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