Pharmaceutica Analytica Acta
ISSN: 2153-2435

Samanidou and Kabir, Pharm Anal Acta 2015, 6:7
DOI: 10.4172/2153-2435.1000e177

Editorial Open Access

Fabric Phase Sorptive Extraction in Pharmaceutical Analysis

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Micro extraction techniques represent the current state of the art in analytical sample preparation characterized with solvent-free/solvent-minimized extraction, miniaturization and automation to treat samples with pharmaceutical, food, forensic, and environmental significance for instrumental analysis. To be consistent with today's challenges of sustainability and environmental issues, Green Analytical Chemistry (GAC) demands are taken into consideration when developing a new analytical methodology in order to comply with the principles of green chemistry. To this direction, the analysis of raw and untreated samples as well as the miniaturization in sample volume requirement leading to the reduction of organic solvents used for sample pretreatment, thus complying with the priorities of GAC as defined by Anastas and Namiesnik [1,2]. Moreover, waste generated during sample preparation exercise is minimized while economy in consumables and energy demands is achieved.

In pharmaceutical analysis, the challenge of greener approach can be met by embracing modern solvent less or solvent-minimized sample preparation techniques. The milestones of micro extraction techniques undoubtedly include, among various techniques, the first reported use of Solid Phase Extraction (SPE) in 1974 by R. Adams and co-workers [3], the introduction of Solid-Phase Micro extraction (SPME) by Pawliszyn and co-workers [4] in 1987, Stir-bar Sorptive Extraction (SBSE) by Pat Sandra and co-workers [5] in 1999, Micro extraction by Packed Sorbent (MEPS) by Mohamed Abdel-Rehim in 2004 [6].

These innovative techniques gave birth to a new era in analytical sample preparation distinguished by their capability of isolating target analyte(s) from simple to complex sample matrices. In the field of pharmaceutical analysis, the predominant sample matrix is of biological origin and often includes plasma, serum, whole blood, urine, saliva, tissues, etc. These samples need to be free from endogenous interference so that the active ingredient of the drug and its metabolites can be determined with high confidence in pre-clinical trials, clinical trials, in therapeutic drug monitoring as well as in clinical toxicology.

Among all biological matrices, blood plasma or blood serum samples are the most common in pharmaceutical and clinical analysis. Whole blood is also a matrix of interest in forensic toxicology.

The isolation of target analytes from these samples includes protein precipitation followed by solid phase extraction/liquid-liquid extraction/solid-phase micro extraction as well as other multi-step extraction and clean-up approaches. Protein precipitation may cause significant analyte loss especially when the target analyte is medium polar or non-polar and therefore should be avoided if possible.

Fabric Phase Sorptive Extraction (FPSE) is a highly promising and versatile sample preparation technique that has been recently introduced by Kabir and Furton [7,8].

Till now, it has been applied in the isolation of various analytes such as antibiotics in milk [9], triazine herbicides in environmental waters [10], non-steroidal anti-inflammatory drugs in environmental water samples [11], selected estrogens [12] and benzodiazepines in blood serum [13], endocrine disruptors alkyl phenols from ground water, river water, sewage water, sludge and soil samples [14]

FPSE utilizes a flexible fabric surface (cotton, polyester, glass fibre, cotton-polyester blend) as the substrate platform for creating sol-gel hybrid organic-inorganic sorbent coatings. FPSE device can be introduced directly into the vial containing the sample eliminating any prior pre-treatment step such as filtration, protein precipitation, and centrifugation (Figure 1). The flexible fabric support in FPSE allows near-exhaustive extraction of the target analyte(s) from untreated sample matrix in a very short period of time.

The high primary contact surface area and the open geometry of FPSE device combined with sol-gel derived sponge-like porous sorbent in the form of ultra-thin coating enable the rapid sorbent-analyte interaction so that target analyte(s) can be extracted from complex biological sample matrices at high efficiency in a fraction of time compared to its conventional analogs. Magnetic stirring enhances the analyte mass transfer rate from the bulk to the FPSE device. Following fabric phase sorptive extraction, preconcentrated analyte(s) of interest are back-extracted using a suitable organic solvent compatible to the analytical technique. Selecting an organic solvent compatible with multiple chromatographic techniques opens up the possibility of simultaneously injecting same sample into different chromatographic techniques (e.g., GC-MS, LC-MS, CEC) to obtain a holistic chromatographic profile of the sample of interest. This may facilitate metabolomics biomarker research in future.

Fabric phase sorptive extraction (FPSE), by design, has innovatively integrated solid phase micro extraction (SPME) and solid phase extraction (SPE) into a single technology platform in two ways. First, it resembles with the direct-immersion SPME where the extracting phase remains submerged into the aqueous solution containing the analyte of interest during the extraction. The aqueous solution may be diffused by applying different strategies e.g., magnetic stirring, sonication, etc. On the other hand, the porous substrate in FPSE and the sponge-like porous network of sol-gel sorbent coating establish a quasi-flow-through system that mimics an SPE disk. Second, FPSE makes use of all major SPE phases including octyl (C8), octadecyl (C18), cyano (CN) moieties as well as popular SPME phases including polydimethylsiloxane (PDMS), polyethylene glycol (PEG), etc. The successful combination of SPE/SPME extraction mechanisms as well as integration of the sorbent chemistries used in both the techniques result in a robust sample preparation device capable of achieving near exhaustive extraction under equilibrium extraction conditions. FPSE also substantially minimizes the number of steps in sample

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Received July 12, 2015; **Accepted** July 13, 2015; **Published** July 15, 2015

Citation: Samanidou VF, Kabir A (2015) Fabric Phase Sorptive Extraction in PharmaceuticalAnalysis.PharmAnalActa6:e177.doi:10.4172/21532435.1000e177

preparation, and consequently reduces the risk of potential analyte loss and experimental errors which are very important especially in the field of chemical and pharmaceutical analysis [15].

Unlike the substrates used in SPE (silica particles) and SPME (fused silica fibre), the fabric substrates used in fabric phase sorptive extraction are not inert. They are either hydrophilic (cotton cellulose) or hydrophobic (polyester) or both (cotton-polyester blend) and play key role in determining the overall polarity and selectivity of the FPSE device.

Sol-gel coating technology used in FPSE provides a facile pathway to create high efficiency sol-gel sorbents chemically bonded to the fabric substrate. Due to the strong covalent bonding between the sol-gel sorbent network and the fabric substrate, FPSE devices demonstrate remarkably high solvent and chemical resistance. As such, the same device can be reused many times without losing its sorption capability. After extraction, a small volume of organic solvent (~500 µL) can be used for solvent mediated back-extraction which preserves the analyte pre-concentration to a great extent and therefore no post-sample preparation treatment process for example solvent evaporation followed by sample reconstitution (typically applied after SPE) is required.

A large number of pharmaceutical products are either weakly acidic or weakly basic and often require pH adjustment of the sample matrix prior to extraction in order to ensure that the molecules are in their neutral form. To extract these entities directly in their ionized form, FPSE has introduced sol-gel anion exchange coating, sol-gel cation exchange coating and mixed mode coatings (neutral + anion exchanger; neutral + cation exchanger) which turned FPSE more effective for pharmaceutical analysis.

Selection of a suitable sorbent for a particular compound class in both SPE and SPME is still dependendent upon the knowledge, experience and judgent of the analyst and often require numerous trial and error based experimentation. This empirical approach may lead to wrong sorbent selection, resulting in compromised sensivity of the analytical method. Fabric phase sorptive extraction, on the other hand, utilizes a rational data driven approach for sorbent selection. Each sorbent chemistry in FPSE is presented with a second-order mathematical model that correlates logK_{ow} values of a broad range of test analytes (log K_{ow} values range from 0.3 to 5.07) with their extraction efficiencies (Figure 2). This model and the associated extraction efficiency calculator can be conveniently used to predict extraction efficiency value for a given analyte using its logKow value. Table 1 presents a comparison between the model predicted values *vs.* actual extraction efficiency values for selected alkylphenols. This model based sorbent selection strategy is indeed a clear manifestation of GAC since the entire sorbent selection process does not require a single experimentation.

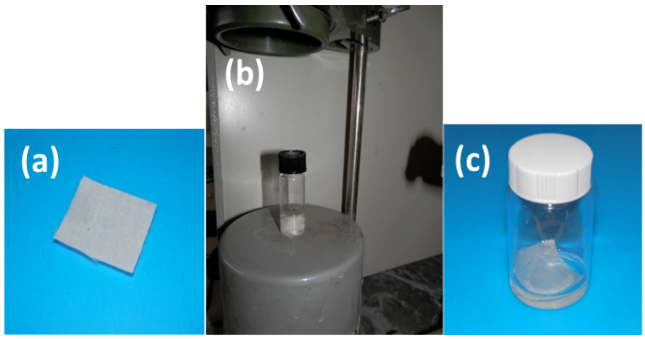

Figure 1: Fabric phase sorptive extraction: (a) a fabric phase sorptive extraction (FPSE) device; (b) extraction using FPSE device; (c) solvent mediated back-extraction.

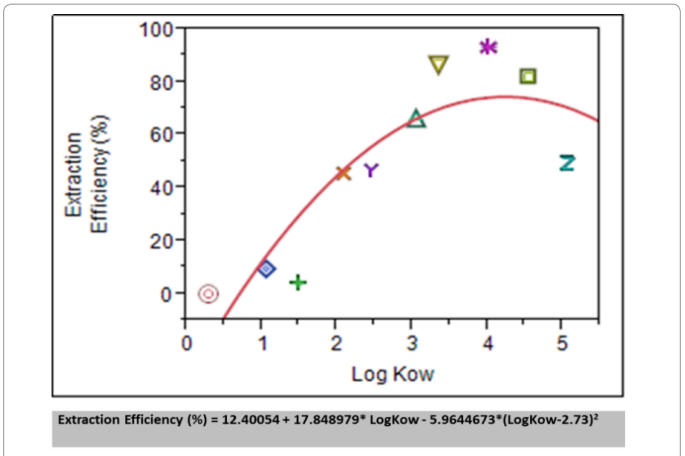

Figure 2: Extraction efficiency calculation model for sol-gel PTHF coated FPSE device.

Compound	Log K_{ow}	Predicted Extraction Efficiency (%)	Actual Extraction Efficiency (%)
4-*tert*-butylphenol	3.29	70.2	74.0
4-*sec*-butylphenol	3.46	72.5	75.6
4-*tert*-amylphenol	4.03	79.1	78.0
4-cumylphenol	4.12	79.9	78.3

Table 1: Extraction efficiency data for selected alkyl phenols [14].

In conclusion, FPSE has presented a viable and greener alternative to conventional SPE and SPME and their many offspring. Due to its simplicity, cost-effectiveness, versatility and robustness, the applications of FPSE in pharmaceutical analyses are expected to grow exponentially in near future.

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