

SOLID-PHASE EXTRACTION (SPE): PRINCIPLES AND APPLICATIONS IN FOOD SAMPLES

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ABSTRACT

Solid-Phase Extraction (SPE) is a sample preparation method that is practised on numerous application fields due to its many advantages compared to other traditional methods. SPE was invented as an alternative to liquid/liquid extraction and eliminated multiple disadvantages, such as usage of large amount of solvent, extended operation time/procedure steps, potential sources of error, and high cost. Moreover, SPE can be applied to the samples combined with other analytical methods and sample preparation techniques optionally. SPE technique is a useful tool for many purposes through its versatility. Isolation, concentration, purification and clean-up are the main approaches in the practices of this method. Food structures represent a complicated matrix and can be formed into different physical stages, such as solid, viscous or liquid. Therefore, sample preparation step particularly has an important role for the determination of specific compounds in foods. SPE offers many opportunities not only for analysis of a large diversity of food samples but also for optimization and advances. This review aims to provide a comprehensive overview on basic principles of SPE and its applications for many analytes in food matrix.

Key words: solid-phase extraction (SPE), applications, food, sample preparation

INTRODUCTION

Food structure is a complex and non-homogeneous matrix, and its physical form can vary from solid to liquid with a natural origin or being processed. Despite advanced techniques, isolation of desired food components from inside of a complicated food matrix, which is constituted of enormous amount of chemical substances and elimination of possible errors during the determination of analyte, is still a difficult goal for researchers.

Sample preparation is a vital procedure in food analysis. After taking sample, sample preparation step before detection and quantification steps for analytes is the main part that takes up most of the total analysis time and usually has an important contribution to total

cost. Complete dissolution of the sample, removal of potential interferences, separation of desired analytes from sample, and concentrate them because of the detection limits are the basic principles of a successful preparation procedure. But also, reducing the analysis time, decreasing of the organic solvent amount used for the extraction and preventing the contamination of sample are the other important challenges in food analysis.

Liquid extraction is a traditional method for sample preparation procedures but, several disadvantages may emerge during its applications such as: incomplete phase separation, emulsion formation with aqueous samples which makes the extraction more difficult,

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decline in quantitative recoveries and use of breakable glasswares. And also, disposal problem of large quantities of organic solvents and difficulties in adaptation of the automation created a need for new methods to overcome all these application problems (Majewska et al., 2008; Thurman and Mills, 1998). Solid-phase extraction (SPE) technique was developed as an alternative practice in 1970s to liquid-liquid extraction (Thurman and Mills, 1998). Now, SPE is one of the widely used sample preparation methods for trapping analytes and separating them from sample matrix.

Solid-Phase Extraction procedure, disposable cartridges or columns which filled with the proper sorbent are used as solid surface and the desirable compound distribution between the liquid sample and solid phase until an equilibrium occurs. High recovery level, reduction in analysis time by decreasing the procedure steps and easy automation, adaptability with chromatographic analysis and a decrease in the organic solvent amount are the main advantages of SPE method (Thurman and Mills, 1998).

SOLID-PHASE EXTRACTION: AN OVERVIEW

Solid-Phase Extraction is based on the partition of the analyte between a solid phase that is usually a sorbent held in a column and a liquid phase that is a sample matrix or a solvent with analytes (Ridgway et al., 2008). As a result of the distribution by adsorption or penetration of molecules on solid surface, an equilibrium is set up and the analyte retains on the solid surface in compliance with the distribution coefficient. Collection of analyte from the SPE system can be carried out through supplying a more desirable environment to the analyte than solid-phase. Desorption of compounds with favoured liquid or other advanced techniques, such as stream of supercritical gas, can be performed via passing of the analytes into liquid phase and exit from the SPE system. After this elution step or between the retention and elution stages, one or more washing/rinsing steps can be applied in order to remove undesirable compounds which retained together with the analytes on the solid phase or eluted phase (Simpson and Wells, 2000; Thurman and Mills, 1998).

Particle size of SPE sorbents usually ranges between 10–60 μm and their formats can be a syringe-barrel, cartridges, discs or pipette types (Buldinia

et al., 2002; Żwir-Ferenc and Biziuk, 2006). Many types of sorbent are available and the selection can be made according to the food matrix, the analytes interested in and interferent substances. Different mechanisms which are based on different interactions, such as van der Waals, hydrogen-bonding, dipole-dipole, or electrostatic (ion exchange) play an important role during the extraction of related analytes from samples (Ridgway et al., 2008).

Normal-phase SPE involves a polar stationary phase and retention arising from interaction between polar functional groups of the analyte and polar groups on the sorbent surface. Reversed phase involves a non-polar stationary phase with a polar or moderately polar mobile phase and generally used for aqueous samples which main interaction force are van der Waals forces. Ion exchange SPE can be used for compounds which are charged when in a solution. Finally in mixed-mode SPE, two different functional groups on the same sorbent are used. Isolation involved in both reversed phase and cation exchange is a general example for this type of SPE (Majewska et al., 2008; Thurman and Mills, 1998; Żwir-Ferenc and Biziuk, 2006).

Among the sorbent types (etc. alumina, magnesium silicate or graphitised carbon), silica is the most common one because of its suitability for modification and stability (Buldinia et al., 2002). Typical examples for the sorbents are silica bonded with nonpolar chains such as C_{18} (octadecyl), C_8 (octyl), cyclohexyl groups or polar chains such as hydroxyl, cyano and diol groups. And also, polymeric resins, Florisil, polar sorbents (alumina, charcoal) and carboxylic acid or amino groups which bonded to silica for ionic functional groups can be given as examples for the other type of support materials (Ridgway et al., 2008; Thurman and Mills, 1998). Due to certain requirements for increasing specificity, some improved extraction media, such as restricted access media (RAM), or affinity columns or molecularly imprinted polymers (MIPs) are available to trap specific compounds concerned (Ridgway et al., 2008).

Solid-Phase Extraction is a useful technique for isolation and separation applications. But also, concentration is one of the goals in its application field. The analyte leaves the pretreated column or cartridge which filled with required sorbent in a small volume of solvent. Relatively small surface of solid phase, large

volume of sample which may provide a complete retention and the use of volatile organic solvents are the main factors for utilizing SPE method for the concentration approach. In addition, SPE can be a beneficial tool for clean-up that is necessary for removal interfering compounds and taking clearly identifiable signals in chromatographic analysis (Thurman and Mills, 1998).

SPE APPLICATION IN FOODS

Food products consist of wide variety components, such as carbohydrates, proteins, fats, vitamins and minerals. While the analysis of a specific compound in food matrix, homogenization, extraction and removal interferences are the essential practices. Solid-phase extraction method is widely used for the analysis of different compounds in numerous food matrixes (Table 1). The variety of sorbents type makes SPE technique one of the best selections for specific sample preparation needs (Simpson and Wells, 2000).

Pesticides

Pesticides are usually detected by liquid chromatography, gas chromatography or capillary electrophoresis depending on such characteristic properties as polarity. Sample preparation step is a necessity due to the diluted contaminants in a very complex matrix. SPE method takes place of liquid/liquid extraction through its simplicity, possibility of polar pesticide extraction, decreasing application time and reduction in the amount of organic solvents (Raoul et al., 1997). Doong and Lee (1999) have reported comparative results for four different SPE procedures (C_{18} cartridge, alumina cartridge, Florisil cartridge and Tandem column) which were used for the clean-up step of fourteen organochlorine pesticide residues in nine kinds of fatty food samples and two nonfatty samples. A Florisil cartridge eluted with petroleum ether–ethyl ether had the most efficient clean-up procedure for fatty samples inside SPE procedures. Schenck and Donoghue (2000) have studied the characterization of pesticide residues in egg samples. The study was performed by using graphitized carbon black (GCB), aminopropyl and Florisil SPE columns for the clean-up step and followed by gas chromatography for the determination of certain organochlorine and organophosphorus pesticide residues. Both polar

and nonpolar pesticides could be efficiently recovered from eggs with a range between 86–108% and 61–149%. Also, Soleas et al. (2000) utilized from a C_{18} bonded porous silica cartridge for SPE application and combined it with GC-MS method for the quantitation of 17 pesticides in wine as, dicloran, dimethoate, diazinon, chlorpyrifos-methyl, vinclozolin, carbaryl, methiocarb, dichlofluanid, parathion-ethyl, triadimefon, procymidone, myclobutanil, iprodione, imidan, dicofol, phosalone and azinphos-methyl. Jiménez et al. (2001) used solid-phase extraction through ethyl acetate eluted cartridges coupling with a gas chromatographic determination. Different types of detectors (nitrogen–phosphorus detection (NPD) and electron-capture detection (ECD)) were used separately for 37 pesticides in wine samples. Two different cartridges (LiChrolut EN cartridges and Oasis cartridges) were examined and results compared with octadecylsilane (ODS) cartridge which was mainly used in wine samples. The results have showed that polymeric phase like Oasis could be a good alternative for the detection of pesticides in wine samples.

SPE columns are widely preferred for pesticide determination in fresh fruits and vegetables. A water miscible solvent is generally used for the extraction and further clean-up step for the organic solvents is needed with SPE columns prior to GC analysis. According to Schenck et al. (2002), aminopropyl and primary secondary amine (PSA) columns could provide the best clean-up results in pesticide analysis of fresh fruit and vegetable extracts comparing with graphitized carbon black (GCB), octadecylsilyl (C_{18}) and strong anion exchange (SAX) SPE columns. Štajnbaher and Zupancic-Kralj (2003) have practiced a solid-phase extraction with highly cross-linked polystyrene divinylbenzene column (LiChrolut EN) after optimization study for clean-up and pre-concentration steps to determine 90 pesticides. Also, further clean-up step was performed with diethylaminopropyl (DEA) modified silica column if it is necessary. According to gas chromatography results, except for the most polar pesticides (methamidophos, acephate, omethoate), recoveries of the pesticides for various fruits and vegetables were higher than 80%. In addition, Barrek et al. (2003) have developed an analysis method for pesticides residues that were extracted from citrus essential oil by using SPE methods. A mixed Florisil C_{18} cartridge was

Table 1. Examples for SPE applications in various food matrixes

Analytes	Sample	SPE	Detection method	Procedure step	References
1	2	3	4	5	6
Nonpolar organochlorine and polar organophosphorus pesticide residues	Egg	GCB, aminopropyl and Florisil SPE columns	GC-ECD and GC-FPD	Clean-up	Schenck and Donoghue (2000)
Pesticides	Wine	ODS, Oasis, LiChrolut EN, Florisil cartridges	GC-ECD and GC-NPD	Extraction and clean-up	Jiménez et al. (2001)
Pesticides	Wine	C ₁₈ bonded porous silica cartridges	GC-MS	Extraction	Soleas et al. (2000)
Organochlorine pesticide residues	Shellfish, fish, meats and cereals	C ₁₈ cartridge, alumina cartridge, Florisil cartridge and Tandem column (C ₁₈ +Florisil)	GC-ECD	Clean-up	Doong and Lee (1999)
Pesticides	Fruits and vegetables	Different sorbent materials (highly cross-linked PS-DVB, graphitised carbon black, C ₁₈ , PSA, NH ₂ graphitic carbon Hypercarb and ENV+)	GC-MS	Clean-up and pre-concentration	Štajnbaher and Zupancic-Kralj (2003)
Pesticide residues	Fruits and vegetables	Graphitized carbon black (GCB), C ₁₈ , strong anion exchange (SAX), aminopropyl (-NH ₂), and primary secondary amine (PSA) columns	GC/ECD, GC/MS, GC/FPD	Clean-up	Schenck et al. (2002)
Pesticide residues	Citrus essential oil	Mixed Florisil-C ₁₈ cartridge	GC-MS, HPLC-MS	Extraction	Barrek et al. (2003)
Mutagenic amines	Beef extract	Coupling of diatomaceous earth, propylsulphonyl silica gel, and octadecylsilane cartridges	HPLC-ED, HPLC-fluorescence detection	Extraction and pre-concentration	Galceran et al. (1996)
Biogenic amines	Fish tissues	Strata X cartridge	LC-FD, LC-MS/MS	Clean-up	Sagratiini et al. (2012)
N-nitrosamines, aromatic amines, and melamine	Milk and dairy products	LiChrolut EN	GC-MS	Clean-up	Jurado-Sánchez et al. (2011)

Table 1 – cont.

1	2	3	4	5	6
Acrylamide	Coffee	Oasis HLB (hydrophilic/lipophilic copolymer sorbent), Bond Elut-Accucat (cation and anion exchange sorbent)	LC-MS/MS	Clean-up	Andrzejewski et al. (2004)
Acrylamide	Cereal-based foods	Oasis HLB cartridges	LC-MS/MS	Clean-up	Zhang et al. (2005)
Ochratoxin A	Alcoholic beverages	LiChroCART (C ₁₈)	On-line SPE-LC-ESI-MS/MS	Extraction	Bacaloni et al. (2005)
Ochratoxin A	Roasted coffee	NH ₂ column	HPLC	Clean-up	Sibandaa et al. (2002)
Patulin	Apple juice concentrate	C ₁₈ cartridge	HPLC-UV	Extraction	Li et al. (2007)
Anthocyanins	Fruits and vegetables	Oasis® MCX sorbent, C ₁₈ , HLB and LH-20	HPLC-PDA, HPLC-MS and Fourier transform infrared (FT-IR) spectroscopy	Fractionation	He et al. (2011)
Anthocyanins	Berry species	Amberlite XAD7	–	Purification	Denev et al. (2010)
Folic acid	Fortified beverages	C ₁₈ Plus cartridges	HPLC-DAD	Purification and concentration	Pérez Prieto et al. (2006)
Folates	Different food matrix	Silica-based sorbent materials (SAX (strong anion-exchange), phenyl (PH), phenyl end-capped (PH EC), and cyclohexyl endcapped (CHEC))	HPLC-DAD	Purification	Nilsson et al. (2004)
Tetracyclines residues (oxytetracycline (OTC), tetracycline (TC) and chlortetracycline (CTC))	Honey	C ₁₈ SPE cartridges	HPLC-FD	Extraction and clean-up	Peres et al. (2010)
Tetracyclines	Milk samples	Strata SCX, Strata C18-E, Strata CN and Strata Phenyl	Reverse phase-LC and amperometric detector	Extraction and pre-concentration	Casella and Picerno (2009)

used in the procedure and results obtained with GC-MS and HPLC-MS techniques as a simple and rapid detection method.

The analysis of pesticides for several types of water samples requires certain preliminary sample preparation step. SPE techniques have an extensive use as being one of the best practices besides several multiresidue methods in the literature for pesticides, including triazines and degradation products (Font et al., 1993; Rodrigues et al., 2007; Sabik et al., 2000).

Amines

Aliphatic, aromatic, heterocyclic and heterocyclic aromatic amines are the different amine groups that are found at various food matrixes but the determination of these trace level compounds is a problematic issue because of their high volatility and polarity structures, high solubility in water and relative basic character (Molins-Legua and Campins-Falco, 2005). Especially protein-rich heated foods, such as cooked meat and fish and fermented products (cheese, wine or beer) are potential sources for amines which are mutagenic/genotoxic substances and increase cancer risk in humans (Toribio et al., 2000).

Galceran et al. (1996) have examined a sample preparation and clean-up procedure with three stages of SPE for the determination of twelve heterocyclic amines in beef extract. A diatomaceous earth cartridge, propylsulphonic cartridge and a C₁₈ cartridge were used coupling with each other and the results were gained by HPLC with electrochemical and fluorescence detection. Twelve heterocyclic amines were determined and also, indolpyridine and imidazopyridine derivatives were detected successfully. Rivera et al. (1996) used multiple solid-phase extraction steps for charcoal-grilled meat. Determination of polycyclic aromatic hydrocarbons (PAHs) was carried out with diatomaceous earth column coupled to a propylsulfonic column (PRS) and the HPLC-UV analysis. Then, C₁₈ column coupled to PRS was used for the separation of nitrogen-containing polycyclic aromatic hydrocarbons (PANHs) and heterocyclic aromatic amines (HAAs). Biogenic amines on fish tissue were also determined by the extraction with trichloroacetic acid and clean-up procedure with SPE (Sagratini et al., 2012).

Jurado-Sánchez et al. (2011) have studied solid-phase extraction in the clean-up and pre-concentration

steps for the determination of toxic substances like as N-nitrosamines (NAms), aromatic amines (AAs), and melamine in milk and dairy products. Furthermore, utilization of SPE technique for the extraction and concentration instead of vacuum distillation and liquid-liquid extraction also revealed successful results for the analysis of volatile N-nitrosamines in foods (Raoul et al., 1997).

Acrylamides

SPE application for the acrylamide detection in food samples is well-documented topic in the literature (Ahn et al., 2002; Granby and Fagt, 2004; Mastovska and Lehotay, 2006). Especially for the clean-up procedure, SPE are often used by many researchers and by the U.S. Food and Drug Administration (FDA)'s survey for acrylamide analysis (Acrylamide, 2014). Roach et al. (2003) and Andrzejewski et al. (2004) have studied the analysis of acrylamide which based on an aqueous extraction of acrylamide and a clean-up procedure firstly with Oasis HLB and secondly an Accucat column. Results of the studies were obtained by LC-MS-MS analysis for various kinds of food samples and for coffee samples respectively. The limit of quantitation of the procedures was 10 ppb (µg/kg) for the first and second had 10 ng/g for ground and instant coffees and 1.0 ng/mL for brewed coffee. Zhang et al. (2005) have practiced another study for cereal based foods. For enhancing the level of recovery, different cartridge types were evaluated (as non-polar stationary phase (Elut-C18, 1 cc/100 mg or 3 cc/500 mg) and hydrophilic-lipophilic balanced (Oasis HLB, 3 cc/60 mg or 6 cc/200 mg)). Detection was performed by liquid chromatography coupled with electrospray ionization tandem mass spectrometry (LC-MS/MS). As regards to the report results, Oasis HLB (6 cc/200 mg) showed a good performance in recovery.

Ochratoxin A and Patulin

Ochratoxin A which can be produced by *Penicillium* and *Aspergillus* species contaminates a large variety of foods as cereals, beans, spices, dried fruits, coffee, beer, and wine, and causes an important human health hazard (Monaci and Palmisano, 2003). Solid-phase extraction is a practical alternative for Ochratoxin A (OTA) detection instead of solvent

extraction which uses large volume of organic solvent or immunoaffinity columns which has low recovery levels in wine samples. Limited shelf-life and characteristic features of immunoaffinity columns which are sensitive against environmental conditions are the other factors that restrict the application field (Jornet et al., 2000). Leitner et al. (2002) have used different analytical methods which contained solid-phase extraction (SPE) with immunoaffinity or RP-18 sorbent materials for clean-up step. This report indicated that SPE combined with LC-MS-MS technique is a good alternative to HPLC-FL protocols already established in the literature (Varelis et al., 2006; Lattanzio et al., 2011). Sibandaa et al. (2002) have examined ochratoxin A in roasted coffee. Interfering compounds were separated with by the aminopropyl (NH₂) column and further clean-up was occurred with an immunoaffinity column (IACs). Recovery range obtained was from 72 to 84% and the limit of detection was 1 ng/g. Bacaloni et al. (2005) have developed an online SPE-liquid chromatography-electrospray tandem mass spectrometry (SPE-LC-ESI-MS/MS) method for the analysis of ochratoxin A for wine and beer samples. Fabiani et al. (2010) compared three different clean-up methods as SPE column, immunoaffinity column (IAC) and the liquid-liquid extraction (LLE) for the detection of Ochratoxin A in wines. According to linearity and recovery values obtained, SPE and immunoaffinity columns had better results than LLE method.

Also, SPE technique may be used for detection of patulin which is other mycotoxin produced by *Penicillium*, *Aspergillus* and *Byssoschylamy* species. Boonzaaijer et al. (2005) have developed a method for apple products (apple juice, apple puree and apples) with using two clean-up steps and HPLC-DAD analysis. A C₁₈ SPE column for the first clean-up step and Romer #224 column for the second step were used in this research and results indicated that this new method could be an alternative to conventional liquid-liquid extraction via the application of two stages SPE purification. Li et al. (2007) have revealed another simple procedure for patulin in apple juice samples using a C₁₈ cartridge for SPE, and HPLC analysis with UV detector. Also, a comparison with liquid-liquid extraction procedure was performed. The minimum detectable amount obtained from this procedure was 0.005 mg/kg.

Sterols

Sterol analysis in food samples requires a multiple stage procedure which includes saponification and purification processes and gas-chromatography (GC) or high-performance liquid chromatography (HPLC) as a final stage. But SPE method, especially for cholesterol analysis, can represent an alternative way for more simple and rapid application. Russo et al. (2005) have developed a SPE-GC method for cholesterol analysis in animal fats. Results of the research showed that fat samples, principally contained at least 0.8 µg·mg⁻¹ of cholesterol, could be analyzed with this method effectively and represented an alternative to saponification. In addition, SPE technique coupled with supercritical CO₂ extraction could be an alternative to traditional soxhlet method for the determination of free cholesterol in some egg-containing food samples (Boselli et al., 2001).

Anthocyanins

Anthocyanins are compounds which belong to polyphenols and they are responsible for the red, blue, and purple colors exhibited by most fruits, and vegetables. Due to the protective effects of these compounds against chronic diseases as cancer, the importance of anthocyanin applications in food or nutraceutical fields increases dramatically. Degree of purity of anthocyanins is an important concern for many researchers because of the validity of the studies. Current extraction methods are non-selective and contain non-phenolic impurities such as sugars, organic acids, other phenolic compounds, amino acids and so on. Polarity of organic acids and anthocyanins differ from each other widely. An ion exchange column can be ideal for polar tartaric, malic, and fumaric acids. On the other hand, a reversed-phase mechanism can retain non-polar anthocyanins (Thurman and Mills, 1998). However, SPE method gains a great attention as a purification tool in the anthocyanin extraction procedures (Huopalahti et al., 2000; Khoo et al., 2012). Denev et al. (2010) obtained anthocyanin-rich extracts which 94.4% of the sugars and more than 88.5% of the acids were separated through SPE methods. At the end of the process, the dried extract that contained 9.6%–24.6% anthocyanins showed high antioxidant efficiency. He and Giusti (2011) have applied a novel approach with using a cation-exchange/reversed-phase combination SPE

and compared this method with three commonly used SPE procedures as C₁₈, HLB, and LH-20 cartridges. The results obtained from research showed that this new method had a high anthocyanin purity and the capacity of removal impurities was extensive.

Folates

Folates are a group of vitamin cofactors which belong to B vitamins. In recent years, many positive healthy effects of these substances as prevention against cardiovascular disease, cancer and neural tube defects have been revealed in various studies (Vahteristo and Finglas, 2000). The main sources of folates are green leafy vegetables, grains, egg yolk and folate fortified food products (Arcot and Shrestha, 2005). Folic acid is the form of folates which is used for a dietary supplement or for enrichment of foods. Because of the healthy effects, the determination of folates in food samples becomes an important research area nowadays. The analysis of folates is not easy because of their multiple forms, instability, and lower concentration level in food and biologic samples (Vahteristo and Finglas, 2000). SPE is a common method for sample preparation in folate analysis. Pawlosky and Flanagan (2001) introduced a C-18 Sep-Pak cartridge for SPE process in wheat, corn and oat cereals and quantitative analysis was performed with LC-MS. Another research prepared by Breithaupt (2001) for fortified fruit juices samples through a clean-up process was performed with an anion-exchange SPE and after, the quantitative analysis was practised by ion-pair reversed phase-HPLC (RP-HPLC). Nilsson et al. (2004) have presented a comparative study for SPE cartridges in folates analysis of some kinds of food samples. Silica based sorbents (trimethylaminopropyl, phenyl, phenyl endcapped, and cyclohexyl endcapped) were tested. Using of phenyl endcapped cartridge with usual trimethylaminopropyl (SAX) cartridges gave the cleanest extract. Pérez Prieto et al. (2006) have also presented a procedure based on C-18 cartridge for purification and concentration steps and followed by a HPLC-DAD analysis for obtaining the results. The study was performed for beverages such as milks, fruit nectars, isotonic drinks and yoghurts which were fortified with folic acid.

Antibiotics

Tetracyclines (TCs) are a member of antibiotics that are used worldwide against the wide variety of organisms. Because of its possible health effects, there is a need for rapid and regular methods for food samples to detect this drug which is used as a veterinary medicine. Brandšteterová et al. (1997) have presented a comparative study for the analysis of tetracycline in meat, milk and cheese samples with using SPE and matrix solid phase dispersion (MSPD) methods. According to the results of the report, the detection limits were stated as 15–22 ng/g for SPE and 30 ng/g for MSPD. Also, Casella and Picerno (2009) have applied a SPE procedure with reverse phase liquid chromatography for the determination of six tetracyclines in milk samples. For the clean-up and pre-concentration steps, four different type of sorbents were tested (C₁₈–E silica-based reversed phase, CN silica-based normal phase, phenyl-silica, and polymeric Strata SCX). Good retention was obtained in phenyl-silica and polymeric Strata SCX cartridges. Tetracyclines were detected successfully and recovery level of the procedure was ranged between 70%–118%, respectively. Another research has been carried out by Peres et al. (2010) for honey samples. SPE method was used as a single step with a common C₁₈ SPE cartridges for the determination of oxytetracycline (OTC), tetracycline (TC) and chlortetracycline (CTC) residues and utilized from HPLC-FD method for getting analysis results.

CONCLUSIONS

Solid-Phase Extraction is one of the sample treatment methods based on passing a liquid sample through a sorbent. The analytes of interest or interferences in the case of clean-up retain on the sorbent through different interaction mechanisms. The variety of these interactions and several kinds of sorbent make SPE a powerful technique which offers different extraction procedures and optimization ways to researchers in worldwide.

This review shows certain specific applications of SPE technique in food analysis. There are a large number of analytes which come from food matrix and the determination procedures with SPE technique demonstrate a great diversity in the literature proportionally. It is a difficult work to summarize all of them in one

survey and this paper contains the general and most applied analytes in the food field.

Solid-Phase Extraction is still a developing field in modern analytical methods. Advances in sorbent chemistry, especially for the selectivity and sorption capacity, simplified procedures and more automation are the some of the potential future trends in this field. The main promotive factor which causes improvements in SPE methods may be excess demand for high diversity and general tendency to take advantage of more reliable, sensitive and rapid analytical methods. It seems that the level of sophistication in SPE will proceed and maybe go further from most of the other sample preparation methods which are used as an alternative to SPE currently.

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