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Review article

SPME-HPLC: A new approach to the analysis of explosives

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Abstract

Methods developed for the analysis of explosives by SPME coupled to HPLC are reviewed with special emphasis on determination and monitoring in environmental samples such as soil and water. Analysis of explosives by using SPME-HPLC as analytical technique is comparatively a new method on which a special attention is focused nowadays. It saves time, avoid use of hazardous extraction solvents, disposal costs and consequently improve the detection limits. The application of SPME is also widened for explosives by using modified 10-port interface and a C-8 refocusing unit combined with two pumps. Several parameters have been optimized to ensure quantitative results such as high concentration of salt and less acetonitrile:water ratio. CW/PDMS/DVB coatings were found to be superior over PA in terms of sensitivity.

Keywords: Explosives; SPME; HPLC; Review

Contents

Abbreviations: Am, amino; ADNT, aminodinitrotoluene; CAR, carboxen; CW, carbowax; DI, direct immersion; DNB, dinitrobenzene; DNT, dinitrotoluene; DVB, divinylbenzene; EGDN, ethyleneglycoldinitrate; GC, gas chromatography; HMX, octahydro-1,3,5,7-tetrazocine; HPLC, high performance liquid chromatography; HS, headspace; LLE, liquid–liquid extraction; MS, mass spectrometry; NaCl, sodium chloride; Na_2SO_4, sodium sulphate; NB, nitrobenzene; NG, nitroglycerin; NT, nitrotoluene; PA, polyacrylate; PDMS, polydimethylsiloxane; PETN, pentaerythritol tetranitrate; PFE, pressurized fluid extraction; RDX, haxahydro-1,3,5-trinitro-1,3,5-triazine; SC-CO_2, supercritical carbon dioxide; SE, solvent extraction; SFE, supercritical fluid extraction; SPE, solid-phase extraction; SPME, solid-phase micro-extraction; TPR, templated resin; TNB, 2,4,6-trinitrobenzene; TNT, trinitrotoluene; UV, ultraviolet

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1. Introduction

Explosives are used primarily in military purposes, industries, mining and agricultural activities. Explosives are dumped in the sea, burned or detonated in remote areas and eventually travel distances from contamination site by leaching into the soil. Explosives are toxic to some extent due to their chemical structure. These are health hazards to living things due to their carcinogenic and toxic nature [1,2]. Their presence in the environment constitutes a potentially serious contamination problem. Their toxic effects may vary from a mild headache to serious damage to internal organs. Explosive products which are gases can also be toxic [1]. The determination of specific explosives in various matrices at trace level concentration continues to be of great importance in both forensic and environment applications [3]. Due to chemical instability of explosives, HPLC is often the technique of choice to separate explosives from other interferences.

Although various extraction methods using highly efficient instruments such as SPE [4–8], column preconcentration [9,10], PFE [11,12], SE [13–15], ultrasonic, soxhlet extraction and sonication [16,17], SFE [18,19] and LLE [20,21] have been used for the preconcentration of explosives. The choice of appropriate sample preparation method greatly influences the reliable and accurate analysis. Some of the techniques given above such as LLE, SFE and SPE are being tedious and time consuming. A large volume of solvent and sample is required for these techniques which are expensive, health hazard and harmful to environment. Use of multi-step procedures for the extraction lead to loss of analytes. Because of need to detect trace concentration of explosives, it is important to concentrate on a rapid, sensitive, solvent free, less laborious and economical technique.

SPME has been proposed as a promising alternative for the sampling, isolation, enrichment of analyte and analyte introduction to a measuring apparatus in one step. It was invented in 1987–1989 [22–26]. It comprised of a holder assembly with a thin fused silica fiber coated with a sorbent. The SPME holder assembly provides protection to fiber and allows piercing of rubber septum. In case of HPLC, the cleaning of fiber is done in desorption chamber of HPLC by running mobile phase to reduce the background in chromatogram. The fiber is exposed to analytes during the operation and retraced within its holder after sampling. Distribution equilibrium is established between the sample matrix and the fiber coating. The distribution constant K_{fs} for equilibrium is given by

$$K_{fs} = \frac{C_f}{C_s}$$

where C_f is the concentration of analyte sorbed on the fiber and C_s is the concentration of analyte in the aqueous phase.

This means, the extracted amount is constant within the limits of experimental error after the establishment of equilibrium. In cases having finite volume of the sample can be expressed as

$$n = \frac{K_{fs} V_f V_s C_0}{(K_{fs} V_f + V_s)}$$

where V_f is the volume of fiber coating, V_s the sample volume, and C_0 is the initial concentration of given analyte in the sample.

In cases having very large distribution constant, $K_{fs} \gg V_s$, the equation can be written as

$$n = V_s C_0$$

This indicates the amount of analyte adsorbed is directly related to volume of sample and initial concentration of analyte with finite sample volume.

In the field sampling, sample volume is very large and $V_s \gg K_{fs} V_f$. Therefore, n can be written as

$$n = K_{fs} V_f C_0$$

This indicates the amount extracted on fiber is directly related to volume of fiber coating and concentration of analyte and is independent of sample volume. So, SPME can be used for field sampling [22,23].

Analytes sorbed on to the surface of coated silica fiber are desorbed in the desorption chamber of SPME-HPLC interface. The interface consists of a six port injector with a special fiber desorption chamber, installed in place of sample loop. Desorption is carried by the use of organic solvent or mobile phase. Analytes are moved into the column for separation followed by detection in the detector.

In this article, potential of SPME is discussed for the analysis of explosives. The review provides general aspects of SPME-HPLC technique, sampling and an overview of optimization of conditions for the analysis of explosives (Table 1).

2. Sampling of explosives

Sampling is based on the transfer/adsorption of analytes to thin film of stationary polymeric phase coated on SPME fiber. Adsorption of analytes depends upon its partition between sample and stationary phase. SPME can be performed manually or by an autosampler. It can be used to collect the samples from field and then is taken back for analysis in lab. The needle opening of SPME device can be sealed by using a piece of septum/or by cooling the needle to prevent the loss of analytes during transport. A data is available on the recovery of explosives from post-explosion debris [27], soil [18], groundwater [18] and seawater [28]. Furton et al. [27] reported that small quantities of explosives were used for detonations and samples were collected from resulting craters. Soil sample collected before the detonations was used as blank. Seawater was collected 0.5 m below the surface from a location near the Hawaiian Islands and transported by placing on ice in a commercial cooler [28]. The other method for obtaining samples of groundwater and soil is by means of drilled wells [18,29]. Phytoextraction is also an alternative for soil sampling. It involves the sampling of plants grown in contaminated soil [30].

3. Extraction of explosives

The sample is placed in a vial, which is sealed with septum type cap. The fiber should be cleaned before analyzing any

Table 1
Summary of application of SPME-HPLC to explosives

Analyte	Matrix	Extraction conditions	Fiber condition	Column/ temperature	Desorption conditions	Mobile phase/conditions	Wavelength	LoD	References
Nitro explosives (2-NT, 3-NT, 4-NT, NB, 1,3-DNB, 2,4-DNT, 2,4,6-TNT, 4-Am-2,6-DNT, 2-Am-4,6-NNT, 6 DNT, RDX, NG, EGDN, PETN, HMX, 1,3,5-TNB, RDX, 1,3-DNB)	Post explosion debris	30 min, 1000 rpm, 25% NaCl	CW/TPR, CW/DVB	Combination of Res-Elut CN column (3 cm × 4.6 mm × 5 μm) and a Bodensil C-18 column (25 cm × 4.6 mm × 5 μm)	Methanol:water (1:1), static, 2 min	Isocratic acetonitrile:water (1:199)	(a) 220 nm EGDN, NG, PETN; (b) 254 nm other explosives	(a) 5–16 ng/mL (water), 10–40 μg/kg (soil)	[27]
RDX, HMX, TNT	Water, soil and plant tissue	20 min	85 μg PDMS	Supelcosil C8 column (25 cm × 4.6 mm × 5 μm) at 35 °C	Acetonitrile (5 mL)	Water/2-propanol (82:18), 1.0 mL/min	254 nm		[18]
HMX, RDX, 1,3-DNB, 3,4-DNT, TNT, 4-Am-2,6-DNT, 2,4-DNT	Sea water	30 min, 0.75 g NaCl, 500 rpm	CW/TPR (50 μm), PDMS/DVB (60 μm), PA (85 μm), DI, 30 min	C18 column (25 cm × 4.6 mm × 5 μm) at 35 °C	Static, 50 μL of 1:1 (v/v) water/acetonitrile, 1–10 min	Isocratic 50% methanol/water, 0.75 mL/min	254 nm	1–10 μg/L	[28]
Explosives	Water	27% NaCl, pH 9.6	PDMS/DVB (65 μm)	3 μm Supel cosil LC8 column (15 cm × 4.6 mm × 5 μm) at 35 °C	–	18% propan-2-ol, 1.5 mL/min	254 nm		[33,34]

sample to prevent high background in chromatogram due to contamination. SPME needle is used to pierce the septum and fiber is withdrawn from the needle of SPME sampling device and exposed to sample. Depending upon the matrix and analyte of interest, there are two modes of extraction: headspace and direct immersion sampling mode.

3.1. Headspace mode

In this mode, the fiber is exposed in the vapor phase above the gaseous, liquid and solid phase. In this case, only volatile analytes can be sampled. It protects damage of fiber coating from high molecular weight and other non-volatile contaminants present in sample matrix, as the fiber is not in direct contact with the sample. Analytes are adsorbed on fiber coating by crossing the air barrier present between extraction phase and sample surface. Muller et al. [31] and Kirkbride et al. [32] reported excellent recovery of explosives by using HS-SPME for the extraction. It was observed that HS-SPME can be used for the extraction of non-volatile explosives such as PETN, RDX and TNT but at elevated temperature up to 100 °C with extraction for longer time.

3.2. Direct immersion mode

DI involves the immersion of fiber coating directly into the liquid sample. The analytes are adsorbed directly on the fiber coating as it is in direct contact with sample matrix. Furton et al. [27] and Rivera et al. [28] extracted samples by direct immersion of fiber into the sample matrix.

It is concluded that DI is better than HS mode for the extraction of explosives [33]. These analytes exhibit high affinity for aqueous solutions and are less volatile due to their polar nature. Therefore, vapor pressure is low and it requires higher temperature for sorption by HS-SPME. In such cases, a loss of information may occur due to the decomposition of analytes at very high temperature used for HS-SPME.

4. Optimization of extraction conditions

Equilibrium is set up between the analytes in extraction phase and sample matrix. Sample agitation, internal cooling, addition of salt, change in pH and derivatization can be used to enhance the equilibrium. Proper selection of fiber is also important factor to obtain the efficient extraction of explosives from the matrix.

4.1. Sample agitation

Magnetic stirring, sonication, and intrusive stirring are used for agitation of sample. Magnetic stirring is widely used for agitation in both HS and DI SPME. It accelerates the transfer of analytes from the sample matrix to coating of fiber. Rivera et al. [28] studied stirring rates with different fibers for the analysis of explosives. A small effect of stirring was observed for carbowax fiber. On the other hand, rate of adsorption increases on PDMS/DVB fiber with increase in stirring rate (Fig. 1). It means stirring of matrix sample increases the distribution of analytes between aqueous phase and extraction phase to reach the thermodynamic equilibrium.

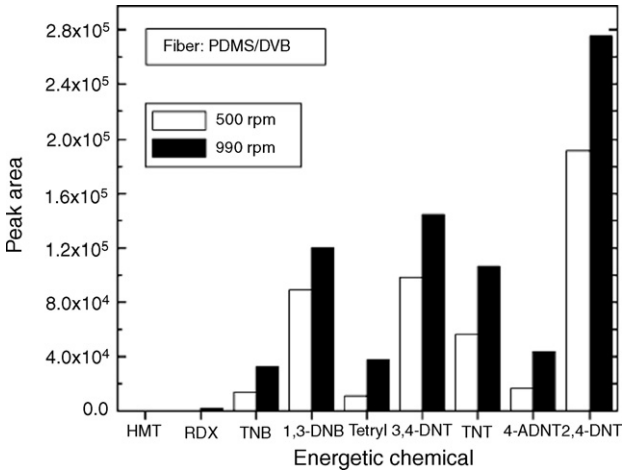

Fig. 1. Effect of stirring rate on the extraction of energetic compounds by SPME/HPLC using the PDMS/DVB fiber. Extraction vials contained NaCl (30% (w/v)), water (35 mL), energetic compounds (3.5 μg in 350 μL of acetonitrile). Fiber was immersed for 30 min in the solution stirred at either 500 or 990 rpm (reproduced with the permission from Ref. [28]).

4.2. Addition of salts

SPME fiber coatings are prone to damage during agitation in DI-SPME. To prevent this damage, extraction can be improved by adding soluble salts in sample to attain super saturation. Addition of salts enhances the extraction of analyte due to salting out effect. Generally, NaCl or Na_2SO_4 are used for improving extraction [27,28] of explosives. The enhancement in extraction of explosives was observed only at salt concentration exceeding 10% (w/v) [27,28]. At lower concentration (0–3%), increase in extraction efficiency was not noticed [34]. Therefore, extraction efficiency depends upon the concentration of salt in the medium (Fig. 2).

4.3. Selection of fibers used for explosives

The choice of fiber has a very significant impact on extraction of analytes. The efficiency of extraction process depends upon properties of fiber coating and its selectivity towards the analyte versus other matrix components. Therefore, optimization of type and thickness of fiber is required for the analysis. An increase in film thickness improves sensitivity but lengthens sampling time. There are seven different fibers available in the market namely, PDMS, PDMS/DVB, stableflex PDMS/DVB, PA, CAR/PDMS, CW/TPR and stableflex DVB/CAR/PDMS. Among these fibers, PDMS [18], CW/DVB [27], CW/TPR [27,28], PDMS/DVB [28,35,36] and PA [28] were used for the extraction of explosives. PA fiber gave lowest HPLC response and large errors for all explosives. Decomposition of stationary phase of the PA under increased pressure is reported by Rivera et al. [28] PDMS/DVB is more selective with respect to nitroaromatic compounds. The recovery of RDX or HMX like nitramine on CW/TPR was found to be very less [28].

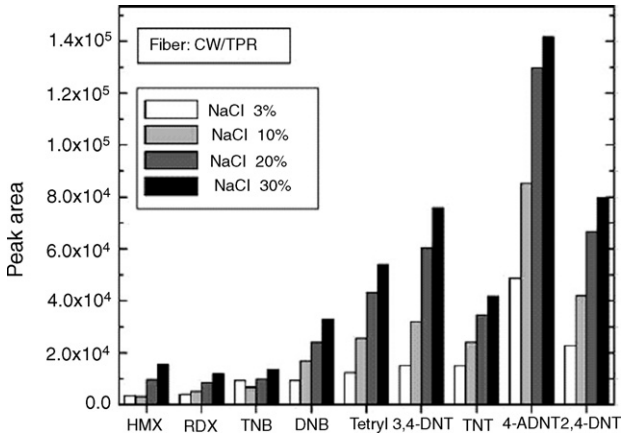

Fig. 2. Effect of medium salinity on the extraction of energetic compounds by using SPME/HPLC. Extraction vials contained various amounts of NaCl (3, 10, 20, or 30% (w/v)), water (25 mL), energetic compounds (2.5 μg in 250 μL of acetonitrile). Fiber was immersed for 30 min in the solution stirred at 500 rpm (TNB = 1,3,5-TNB; DNB = 1,3-DNB) (reproduced with the permission from Ref. [28]).

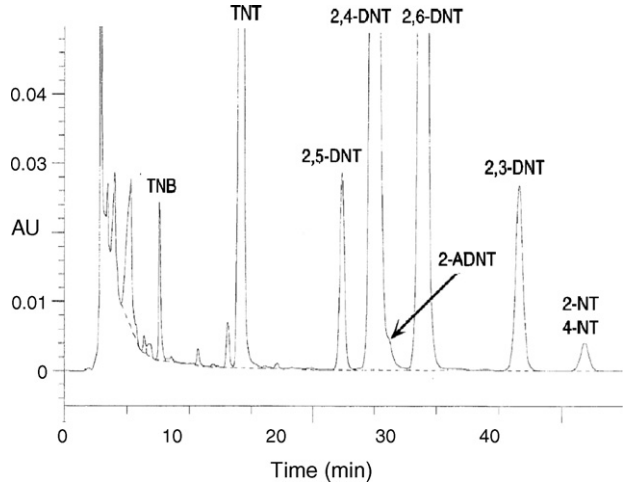

Fig. 3. Analysis of TNT and its derivatives by HPLC-UV in the aqueous phase of a soil sample obtained from a former TNT manufacturing plant (reproduced with the permission from Ref. [18]).

5. Desorption of explosives

After the exposition to a sample, the fiber is withdrawn into the needle of SPME holder. It is introduced into desorption chamber of HPLC–SPME interface which is a specially designed six port injector with a desorption chamber installed in place of sample loop. Analytes are desorbed from the fiber into the mobile phase. There are two desorption modes: dynamic and static desorption mode.

5.1. Dynamic desorption

In dynamic mode, analytes are diffused from the coating into the stream of carrier fluid. It is used for desorption of weakly adsorbed analytes.

5.2. Static desorption

It is used for the strongly adsorbed analytes. It involves the soaking of fiber into the solvent for a specified time for the complete desorption of analytes. Rivera et al. [28] optimized the desorption time to 5 min for the desorption of explosives.

6. Applications

Halasz et al. [18] developed a method for the extraction of explosives and their degradation products from water, soil and plant tissue samples. Acetonitrile, SPME and SC-CO_2 were used for the extraction of analytes. The extracted samples were analyzed by using HPLC-UV (Fig. 3), CE-UV and GC–MS. Results obtained by using SPME-GC–MS and SPME-HPLC-UV was compared by analyzing the water and soil from a TNT manufacturing plant. A correlation factor in 90–100% was obtained.

Furton et al. [27] optimized the conditions for the recovery of explosives by using modified SPME-HPLC interface. By using optimized desorption and injection variables, improved chromatographic resolution and sensitivity were obtained. The optimum conditions for extracting explosives are at low acetonitrile to water ratios and high NaCl salting concentrations (Fig. 4). The proposed method was applied to the analysis of real post-explosion debris. The technique can be utilized for analyzing explosives after field sampling.

Rivera et al. [28] extended the potential of technique for the preconcentration of nine explosives by using SPME-HPLC-UV. The optimized technique was applied to the analysis of seawater and groundwater samples. Excellent agreement was observed between the results of SPE-HPLC-UV and SPME-HPLC-UV analysis of explosives.

Wu et al. [35] modified SPME/HPLC interface by using a 10-port valve and a C-8 refocusing unit (Fig. 5). This eliminated the potential problem of significant extra column desorption caused by a large sample volume due to large volume of desorption

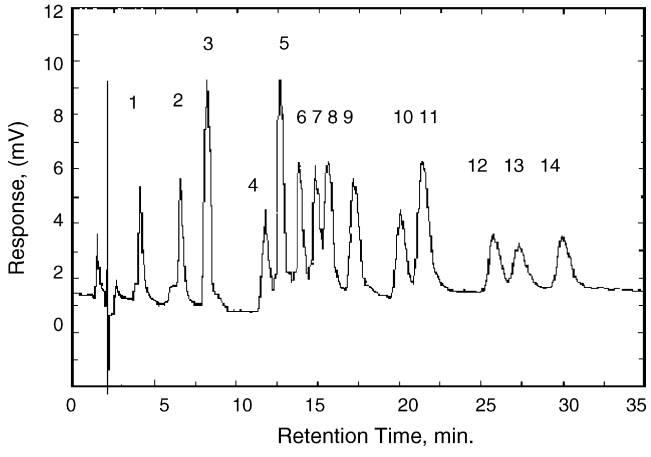

Fig. 4. Chromatogram of SPME-HPLC-UV of EPA 8330 mixture standard (20 ng mL, each in 25 % NaCl aqueous solution of CH_3CN:H_2O ratio of 1:199. Peak 1 = HMX; Peak 2 = RDX; Peak 3 = 1,3,5-TNB; Peak 4 = Tetryl; Peak 5 = 1,3-DNB; Peak 6 = TNT; Peak 7 = 4-A-2,6-DNT; Peak 8 = NB; Peak 9 = 2-A-4,6-DNT; Peak 10 = 2,6-DNT; Peak 11 = 2,4-DNT; Peak 12 = 2-NT; Peak 13 = 4-NT; Peak 14 = 3-NT (Reproduced with the permission from Ref. [27]).

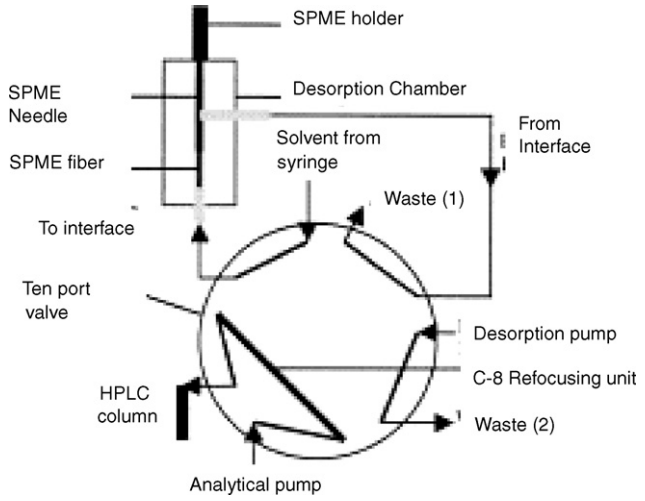

Fig. 5. Ten-port valve at "static desorption" position (reproduced with the permission from Ref. [33]).

chamber and liquid laminar flow behavior. It was combined with an analytical pump and desorption pump. The use of separate desorption and separation improved the stability of chromatogram baseline. This combination also improved the sensitivity and reproducibility of method.

Haag et al. [36] reported the use of SPME-HPLC for the determination of 14 explosives in water by using PDMS/DVB fiber. This application is also reported by Shirey et al. [37].

7. Conclusions

SPME is regarded as a very promising extraction method for chromatographic determination of explosives up to the ppb levels in environmental samples. Explosives with low volatility can be easily estimated using hyphenation of SPME with HPLC. The extraction step is same for both hyphenations of SPME with GC as well as HPLC. But desorption of the analytes from the fiber is carried out in a specially designed interface hyphenated with HPLC using the mobile phase. The hyphenation is very simple and quick for the thermally unstable analytes in which SPME-GC fails to provide good results. Further modifications in the interface having 10-port valves are reported resolving the problems of extra column desorption caused be large sample volume. A wide spectrum of explosives can be analyzed with excellent retention time reproducibility and sensitivity. Thus, SPME can be considered as an extraction technique for the future. Advances in this area will greatly extend the application of SPME. As the hyphenation of SPME with HPLC is relatively new and very limited literature is available therefore more work is needed to be done in this area so that this environmental friendly technique could be encouraged for analytical studies in future.

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