2019 2nd Scientific Workshop on Chemical and Agricultural Sciences (SWCAS 2019)

Application of Solid Phase Extraction Technology in Environmental Chemical Analysis

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Keywords: environmental chemical analysis; solid phase extraction; application

Abstract: Solid phase extraction technology is widely used in environmental chemical analysis. The technology is convenient, fast and efficient, which brings great benefits to environmental chemical analysis. The article is an overview of solid phase extraction technology, including the basic principles, its classification and separation mode, the operation method of solid phase extraction technology, and fully expounds the application of solid phase extraction technology in environmental chemical analysis.

1. Introduction

At present, China's social and economic level and science and technology have greatly improved and improved, but along with it, environmental problems have emerged. Although people's economic level and quality of life have greatly improved, but the chemical industry, automobiles Multiple factors such as exhaust gas have adversely affected the natural environment, and its pollution level is increasing. Environmental problems are an important issue that cannot be ignored in the process of social development. To effectively improve environmental problems, efficient and rapid analytical and analytical techniques are essential. . Solid phase extraction technology is a kind of sample pretreatment technology developed in recent years. Its analysis results have high precision, short time and high efficiency, which greatly improves the accuracy of pre-processing basic data of environmental samples.

2. Overview of solid phase extraction

2.1 Principle of solid phase extraction technology

Liquid chromatography is the basic technical principle of solid phase extraction technology. Its separation mode mainly includes reverse phase, normal phase, ion exchange and adsorption. The adsorbent used in the solid phase extraction technique is the same as the stationary phase commonly used in liquid chromatography, and only has a certain difference in the shape and particle size of the filler. The particle size distribution of the solid phase extraction packing is wider than that of the stationary phase for liquid chromatography, and the solid phase extraction cartridge is a one-time consumable. The solid phase extraction technique mainly uses a solid substance as an extractant to extract certain components from a sample, and generally takes a column separation, and the stationary phase is a part of the solid phase extraction column that functions as a separation. Solid phase extraction technology is an adsorption extractant, which is mainly used for the treatment of tax components. When the water is measured by the solid phase extraction column with suitable adsorption, the sample to be tested does not exceed the sample to be tested. The adsorption capacity is completely retained and then eluted by the selective solvent of the flask. Therefore, the solid phase extraction technique is an effective way to simultaneously extract and concentrate.

DOI: 10.25236/swcas.2019.021

2.2 Classification and separation mode of solid phase extraction technology

2.2.1 Classification of solid phase extraction technology

According to the basic principle of solid phase extraction (SPE), it is mainly divided into normal phase SPE (NP-SPE), reversed phase SPE (RP-SPE) and ion exchange SPE (IE-SPE). The adsorbents used in normal phase SPE are all polar, interacting with the polarities of the polar functional groups on the surface of the adsorbent by polar functional groups (hydrogen bond, $\pi - \pi$ bond interactions, dipole-dipole interactions,dipole-induced dipole interaction and other polarities - polarity)effects to retain polar compounds dissolved in non-polar cut-offs. The adsorbent and target compound used in the reverse phase SPE are generally non-polar or weakly polar, and the target is retained mainly by the carbon-hydrogen bond of the target and the functional group on the surface of the silica to generate a non-polar van der Waals force or dispersion force.The ion exchange SPE is generally adsorbed by electrostatic attraction between a charged group of the target and a charged group on the bonded silica.

2.2.2 Solid phase extraction technology separation mode

The separation mode of the solid phase extraction technique is closely related to the type of adsorbent in the stationary phase and the nature of the eluent. There are many types of SPE adsorbents, and the adsorption type includes activated carbon, silica gel, diatomite, magnesium silicate, and alumina. In the chemically bonded phase silica gel, The positive phase has an amino group, a nitrile group, a glycol group, etc.The reversed ones have C_1, C_2, C_6, C_8, C_{18}, a nitrile group, a cyclohexyl group, a phenyl group, etc.; the ion exchange has a quaternary amine, an amino group, a diamino group, a benzenesulfonic acid group, a carboxyl group, and the like. Further, a polymer, a porous styrene-divinylbenzene copolymer or the like is also required. The polymer is applicable to the pH range relative to the bonded phase filler and, therefore, is used in a wider range of applications.

In addition to the above-mentioned fillers similar to those used in HPLC, a trapping medium with the same principle as that of molecular exclusion chromatography and a molecularly imprinted adsorbent similar in principle to immunoaffinity chromatography are new types of solid phase extraction. Stationary phase, these are effective means of pre-analytical treatment of body fluid samples. When operating in a specific solid phase extraction technique, it is necessary to select a suitable adsorbent depending on the nature of the target. As shown in Table 1, it is the type of common adsorbent and its associated separation mechanism, the nature of the eluent, and the nature of the component to be tested.

Table 1 Different types of adsorbents used in SPE

Adsorbent	Separation mechanism	Elution solvent	Analyte properties
Bonded silicone C_8 , C_{18}	Inversion	Organic solvents	Non-polar - weak polarity
Porous Styrene-divinylbenzene copolymer	Inversion	Organic solvents	Non-polar - medium polarity
Porous graphite carbon	Inversion	Organic solvents	Non-polar - quite polar
Alanine bonded silica gel	Positive phase	Organic solvents	Polar compound
Magnesium silicate	Positive phase	Positive phase	Polar compound
Ion exchange resin	Ion exchange	A certain pH aqueous solution	Anion-cationic organic matter
Antibody binding adsorbent	Immunoaffinity reaction	Methanol/water solution	Specific pollutant

2.3 Solid phase extraction technology operation method

The operation methods of the solid phase extraction technology mainly include activating, loading, washing the adsorbent, and eluting the analyte (as shown in FIG. 1).

(1) Activation. Rinse the cartridge with a column-filled eluent or rinse the filter with 5-10 ml of eluent before extraction. First, the adsorbent can be washed with a water-soluble organic eluent such as methanol. The methanol can wet the surface of the adsorbent and then penetrate into the non-polar silica-bonded phase, making the silica gel more easily wetted by water, and then adding water. Or buffer rinse. Before the sample is added, it is kept moist by using the SPE adsorbent. If the adsorbent is dry, the sample retention value is easily lowered. If the dryness of the small column is different, the reproducibility of the recovery rate is affected.

(2) Adding samples. Select a relatively weak solvent to dissolve the sample. After the liquid sample is added to the SPE cartridge, the non-retained or weakly retained components flow out with the solvent, and the fraction to be tested and the other gun-retaining component are on the adsorbent. The flow rate of the loading or eluent is too fast, which may result in incomplete retention or elution, which has a great influence on the extraction effect and recovery rate; the flow rate is too slow, the working time is elongated, and the efficiency is lowered. The solid phase extraction effect and recovery rate can be guaranteed under the flow rate of the adaptation range. Under normal circumstances, the flow rate is lower than 5mL.min, and the ion exchange extraction is lower than 2mL.min, general “column chromatography”, such as D101. Macroporous resin, polyamide, alumina, etc., if it is at natural flow rate, it can be unregulated. If the flow rate is too fast, it needs to be adjusted with a column with a piston; when the adsorbent is fine and the natural flow rate is too slow, it needs An external force is applied, for example, pressurization, decompression, and centrifugation.

(3) Cleaning the adsorbent. The cleaning solution of the reverse phase SPE is generally water or a buffer, and a small amount of organic eluent, inorganic salt or pH can be added to the clear liquid. The cleaning solution added to the cartridge should be in the solvent range of a small column, while the SPE filter is 5-10 ml.

(4) Elution of the analyte. Select 5-10mo ionic strength is weak, but wash the eluent of the test substance, in order to keep the target drunk limit when adsorbing, the elution is as much as possible to elute it cleanly, can effectively improve the recovery rate, if the recovery rate is too Low, it can increase the amount of adsorption and choose other adsorbents that have stronger adsorption capacity to the target, and can also replace the eluent.

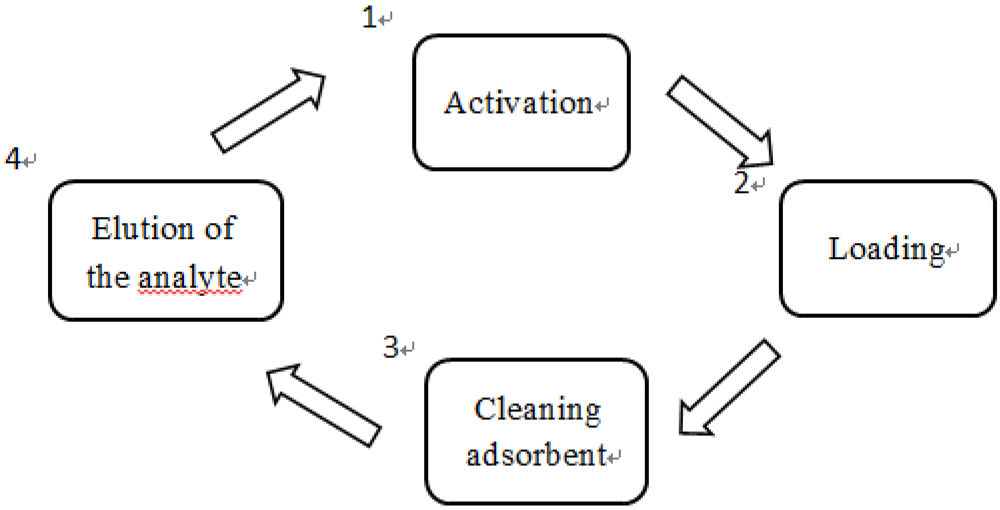

Figure 1 Solid phase extraction technology operation method

3. Application of solid phase extraction technology in environmental chemical analysis

3.1 Polycyclic aromatic hydrocarbons

The analysis of polycyclic aromatic hydrocarbons is an important monitoring part of environmental pollutants. The sanitary standards for drinking water in China stipulate that the content of 3,4-benzopyrene in water should be less than$0.01 \mu g / L$.The key to the analysis of benzopyrene in water is the separation of benzopyrene from other organic matter in the enriched water sample. Some of the compounds in polycyclic aromatic hydrocarbons have strong teratogenic, carcinogenic or mutagenic effects. Compared with classical liquid-liquid extraction, solid phase extraction takes less time, uses less solvent, and is easier to emulsify. For example, water quality testing such as Songhua River and Huangpu River in China was widely applied to solid phase extraction technology as early as the 1980s. Hou Xuewen [1] and other scholars used solid phase extraction as a sample pretreatment method. The results showed that under optimized experimental conditions, The spiked recovery of polycyclic aromatic hydrocarbons is $78\%^{1}30\%$, the relative standard deviation is $0.8\%^{5}.0\%$. The limit of quantification is $0.0007^{0}0.0199 \mu g / L$, This method is fast and simple, and can meet the analytical detection requirements of polycyclic aromatic hydrocarbons in water.

3.2 Pesticide

Pesticides and herbicides that develop rapidly after the emergence of organophosphorus pesticides. These pesticides contain carbamates, and some people will be poisoned by suicide, accidental or poisoning. To treat and rescue such poisoned patients, it is necessary to analyze the carbamate data in Nangong Yao as a reference data. Solid phase extraction and high performance liquid chromatography can effectively separate and detect the body fluids of poisoned patients. The urethane pesticides are used to remove protein interference.Zhao Li [2] and other scholars established a method for solid-phase extraction-gas chromatography-tandem mass spectrometry (GC-MS-MS) detection of 9 herbicide residues in domestic water, and adjusted the pH of the water sample to 1.0-2.0.After C_{18} solid phase extraction, it was eluted with ethyl acetate-dichloromethane (1:1, v/v),The eluate was concentrated to near dryness by nitrogen blowing and determined by volumetric setting with ethyl acetate. Separation by capillary column using DB-35MS, selective ion scanning (SIM) mode detection, and external standard method for quantification. research shows,9 herbicides in $0.05-1.0 \mu g / ml$ range, have a good linear relationship, Method detection limit is $0.0001-0.0005 \mu g / L$;The average recovery was 72.30%-105.34%, and the relative standard deviation was 2.16%-6.39% (n=6). This method can be used for simultaneous detection with various herbicide residues in drinking water.

3.3 Polychlorinated organic compounds

Traditional water sample analysis requires separation, enrichment, purification, etc., but these steps take a long time, resulting in no error in the experimental time. After the solid phase extraction method is combined with other technologies, it is only necessary to separate different samples by using simple equipment, low cost and less solvent. Lin Yanping [3] believes that the solid phase extraction technology can be applied to the pretreatment of poorly soluble semi-volatile organic compounds, which can achieve rapid and efficient enrichment and concentration. Combined with the optimized conditions of GC-MS and HPLC, separation, qualitative and trace quantitative detection analysis were achieved. Among the many enrichment and concentration factors affecting such compounds, the effect of improving the polarity of water bodies is obviously better than that of small column type preference and super-span radiation. By adding acetone with a water content of 5%, the polarity of the water body can be effectively improved, and most of the water-insoluble SVOCs are effectively enriched to achieve the purpose of trace detection. Liu Jing [5] and other scholars were enriched at a flow rate of 3.0 ml/min through a large sample sampler Cleanert PS solid phase extraction column, vacuum-dried, eluted with 10.0 ml of acetone, and evaporated to eluate with 1.0 ml. The alkoxide is brought to volume and analyzed under selected chromatographic conditions. The study found that the concentration of 17 polychlorinated organic compounds is $0-100 \mu g / L$,it has a good linear relationship with the peak area of the chromatogram, and the correlation coefficient is greater than 0.999.The method detection limit is $0.0025-0.074 \mu g / L$ .The recoveries of the spiked samples were 81.0%-116.0%, and the relative standard deviations of the assays were 0.56%-6.8% (n=6).

3.4 Phenols

Among organic pollutants, phenolic compounds are a more toxic part, so it is necessary to analyze the classification by solid phase extraction. The use of UV detectors in the analysis of classified compounds in solid phase extraction water can effectively separate and detect the classified compounds in 11 and at the same time do not require derivatization. Liu Xiaowu [5] determined the phenolic compounds in water by the combination of solid phase extraction and high performance liquid chromatography. The solid phase extraction cartridge was activated by 4 ml of methanol and 4 ml of pure water before adsorption. The phenolic compound was adsorbed by the solid phase extraction cartridge, and then eluted with acetonitrile. The eluate was concentrated to 1 ml to V (water): V. (acetonitrile) 8:2 gradient elution, ending with V (water): V (acetonitrile) 2; 8, flow rate 1.0 ml / min, separation of phenolic compounds by ODS-C_{18} detection wavelength of 285 nm. The linear relationship of the standard curve is greater than 0.9990; the detection limit is less than the requirements of the sanitary standard for domestic water; the average spike recovery is between 70-90%.

4. Conclusion

In summary, solid phase extraction technology is widely used in air, water quality, polycyclic aromatic hydrocarbons, pesticides, organ chlorine compounds, etc. in the environmental analysis of European countries without the advantages of simplicity, rapidity, high precision, low solvent consumption and high recovery rate. With the deepening and expansion of research on solid phase extraction technology, it is believed that in the future, solid phase extraction technology will receive more and more attention and welcome from environmental chemistry analysis work, and provide more help for environmental chemistry analysis work in China.

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