

Review

Application of negligible and full depletion micro-extraction in environmental sciences



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ABSTRACT

Negligible depletion micro-extraction (nd-ME) and full depletion micro-extraction (fd-ME) have been developed for specific purposes in environmental studies. Nd-ME is mainly designed to measure the free concentration and therefore evaluate the bioavailability of analytes in environmental matrices, and achieved by using large ratio of sample to extraction phase or extraction phase with low distribution coefficients for analytes. On the contrary, fd-ME is adopted to avoid matrix effects by determining the total amount of the analyte in the samples, and performed by using very low ratio of sample to extraction phase or extraction phase with extremely large distribution coefficients for analytes. In this review article, we highlight the basic concepts and applications of nd-ME and fd-ME in environmental sciences.

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Contents

1. Introduction	10
2. Principle and characteristics of nd-ME and fd-ME	11
3. Comparison for fd-ME and nd-ME	12
4. Application of nd-ME in environmental study	12
4.1. nd-SPME	12
4.1.1. Uptake kinetics	13
4.1.2. Rate limiting process	13
4.1.3. Coating material	14
4.1.4. Calibration method	14
4.1.5. Matrix effects	14
4.2. nd-LPME	16
5. Application of fd-ME in environmental study	17
5.1. Fd-LPME	17
5.2. Fd-SPME	17
6. Conclusions	19
Acknowledgments	19
References	19

1. Introduction

The discharge of huge and wide range of chemicals to the environments may exert adverse effects on the environment and

ecosystems. Recently, the determination of these chemicals in different environmental compartments such as air, water and soil/sediment has become critically important due to the rising concern over the deteriorating environmental quality. Nowadays various classes of environmental contaminants have been investigated which includes, among others, pesticides, polynuclear aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), polybrominated diphenylethers (PBDEs) and aromatic amines. These

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compounds are of great environmental concern due to their toxicity, carcinogenicity, or endocrine disrupting effects [1]. Environmental risk assessment of these chemicals depends on the understanding of their concentration level, fate, transformation and transportation in environmental systems. To this end, the concentrations of these chemicals in various environmental samples have to be measured by chemical analysis. Due to the trace levels of chemicals and the complex matrix of these samples, an extraction step prior to chemical analysis is generally required to isolate and enrich the trace level analytes from sample matrices [2,3].

Classical extraction procedures consume large amounts of toxic solvents which result in environmental and occupational hazards. These extraction techniques are also time consuming and often less selective. Therefore, microextraction techniques that are solvent free or consume very small amount of solvent are developed as alternatives [2–4]. Microextraction techniques are alternative sample preparation techniques which have been widely used in different fields of environmental science. It is easy to operate, environmentally friendly and utilize very small volume of extracting phase (usually a few microliter ranges) relative to the sample volume. In these techniques, analytes are extracted by a small volume of a liquid/solvent as in liquid-phase microextraction (LPME) or by a small volume of a solid or semi-solid polymeric material/adsorbent, as in solid-phase microextraction (SPME) [4]. Despite the substantial structural differences between SPME and LPME methods, both techniques share similar features as microextraction approaches and they are widely adopted as efficient sample preparation procedures in analysis of environmental samples [3,4].

Usually, most SPME and LPME procedures are nonexhaustive (i.e., conducted in the regime of incomplete extraction) in which only a small part of the target analytes were extracted from a large amount of sample into a small amount of extracting phase to obtain a high enrichment factor. These extraction modes usually result in sample matrix-dependent extraction efficiency and thereby calibration difficulty when real samples are analyzed [5,6]. According to recent reports, this matrix effect can be eliminated by performing extractions in exhaustive mode, where the analytes are completely extracted from relatively small amount of sample (a few milliliter to a few micro liter volume of extracting phase) by modifying the extraction conditions to obtain an extraction efficiency value close to 100% [7,8].

Negligible microextraction (nd-ME) and full depletion microextraction (fd-ME) involving SPME as well as LPME are among miniaturized sample preparation techniques which have attracted considerable attention in recent years for environmental applications. Both LPME and SPME have been utilized as negligible and full depletion microextraction techniques for extraction of environmental pollutants. However, so far applications of nd-ME and fd-ME in environmental analysis have seldom been reviewed. To fill this gap, the present review compiles the new developments and applications of nd-ME and fd-ME techniques for the determination of chemical pollutants in environmental matrices.

2. Principle and characteristics of nd-ME and fd-ME

In polydimethylsiloxane (PDMS)-based SPME and hollow fiber membrane-based LPME, only the free fraction of analytes are extracted by partitioning into a small volume of extraction phase, whereas the analytes associated to matrices such as humic acids and proteins are excluded. If only negligible amount of an analyte is extracted, the free concentration in the sample will not change significantly (Fig. 1) and the extracted amount will then be proportional to the free concentration [9]. As this application causes negligible depletion of the free fraction, it has been named

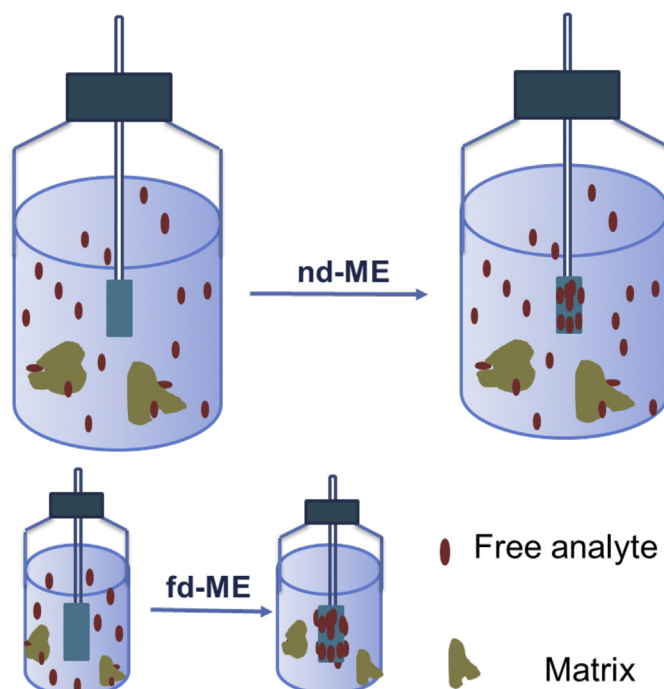


Fig. 1. Scheme of the principle of nd-ME and fd-ME.

nd-ME. However, as the depletion will never be 0%, a limit must be set until the depletion is considered negligible. Górecki and Pawliszyn [10] proposed 1% so that the amount extracted is lower than 1% of the initial amount of the analyte present in the sample. Vaes et al. [11] have set this limit at 5% and thus the depletion of the analytes less than 5% at all times was considered, while some other researchers have chosen 10% [12,13]. Therefore, depletion is considered negligible when analyte amount changes by 10% or less during extraction because this is an amount in line with experimental error [14]. Understanding of how the maximum allowed depletion affects the outcome of the experiment should precede the choice of a decent limit, and this should preferably be reported in all nd-ME publications.

On the other hand, fd-ME, also named exhaustive microextraction, is a situation where an analyte has high affinity for extracting phase and thus the analyte is almost completely removed from a sample. In this situation, the capacity of the extraction phase for the analytes extracted must be significantly larger than the amount of the analyte in the sample. Determination of whether exhaustive extraction is possible for a given microextraction may be made either experimentally or theoretically. Experimentally, determination must be made for various sample volume regarding whether the amount extracted is >90% of that originally present in the sample [14]. For instance, according to a previous report [15], for an extraction method to be considered exhaustive, more than 90% of the analyte should be removed from the sample solution after the extraction is completed. In the analysis of unknown sample using an exhaustive extraction, the initial sample concentration may be determined directly from a measure of the amount extracted, provided that the volume of the sample is known.

Nd-ME and fd-ME are commonly achieved based on SPME or LPME methods and developed for specific purposes in environmental studies. Both SPME and LPME are mainly equilibrium extraction methods rather than an exhaustive extraction technique, i.e., even after the extraction process has been completed a substantial portion of the analytes usually remain in the matrix. In these methods, a small amount of extraction phase is exposed to a

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