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Review article

Sample Pretreatment, Extraction, and Analysis of Organic Contaminants in Surface-Water Sediments: Green Approaches and Public Health Relevance-A Review

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ABSTRACT

Sediments that are exposed to surface water might take up organic contaminants from natural sources and humans, thus bringing about environmental pollution and creating a risk to human health. Hence, this gives enough reason to indicate the need for conducting proper analysis of pollutants. The literature on sediment pre-treatment, extraction of contaminants, cleaning, concentration, and instrumental analysis is covered in the review. Conventional and advanced methods include Soxhlet extraction, ultrasonic extraction, pressurized extraction, microwave extraction, and supercritical fluid extraction. These methods may differ in terms of efficiency, solvent requirements, duration, and analytical capabilities. Chromatographic and mass-spectrometric techniques, such as gas chromatography, high-performance liquid chromatography, and mass-spectrometric methods, are most commonly used today to determine and identify a range of environmental contaminants. Special emphasis has been placed on solvent-free and green methods, as well as sustainable analytical criteria for monitoring ecological performance. This study provides an in-depth look into sediment pretreatment, extraction procedures used, and analytical methods as well as environmentally friendly analytical methods and potential implications for public health, thereby providing a broader overview than reviews that focus on a specific method of extraction or category of contaminants. Effective sample preparation and recognized analytical methods are key for reliable assessment of organic pollutants contained in sediments.

Keywords: Chromatographic analysis; Organic pollutants; Public health; Sample pretreatment, Surface water, Sustainable extraction methods.

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1. INTRODUCTION

The top layers of marine and land ecosystems, where sediment layers meet surface water, serve as natural reservoirs of organic substances of both natural and human origin, such as hydrocarbons, pesticides, polymers, phenols, lipids, and many other volatile or stable compounds. The detection and quantification of these compounds in the sediments is important to assess environmental pollution, to identify contamination sources and to understand the biogeochemical dynamics [1]. The sediment matrix is highly diverse, which hinders the collection of representative field samples and their preparation

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for chemical analysis. The samples are subjected to drying, homogenization, sifting, solvent extraction and matrix cleanup prior to instrumental detection and purification procedures or concentration of desired analytes. The choice of pretreatment has a great influence on the accuracy, sensitivity and repeatability of the subsequent analytical techniques such as gas chromatography (GC), high-performance liquid chromatography (HPLC), mass spectrometry (MS), and GC-MS [1, 2].

A careful focus on the processes of sample collection, preservation, and preparation is crucial for consistent organic contamination assessment in SCS. Enhanced pretreatment not only produces more accurate results but also offers superior understanding of pollutant transport and fate mechanisms [3].

Organic contaminants accumulated in sediments can persist in aquatic environments and may become available to aquatic organisms, contributing to their transfer through food webs. Consequently, sediment contamination is relevant not only to environmental monitoring but also to biological and public-health assessment. Reliable pretreatment and analytical methods are therefore important for identifying contaminants and evaluating potential exposure through aquatic resources [4]

Appropriate quality assurance and quality control (QA/QC) and method validation, including the use of surrogate and internal standards, method blanks, matrix spikes, replicate analyses and certified reference materials, are essential for obtaining reliable and reproducible results in sediment-contaminant analysis (see the section on Quality Assurance, Quality Control, and Method Validation) [5].

This study reports on effective methods for the gathering of materials from surface water and the preparation and handling of sediments for the purpose of identifying organic materials by means of different chemical methods of analysis. A primary concern here is making improvements in the fields of sampling, separation, and purification techniques for the purpose of minimizing sample contamination as well as enhancing analyte recovery, thus increasing the accuracy and specificity of identification and quantification.

Earlier studies were primarily focused on single extraction processes, limited to certain types of pollutants and overall environmental sample preparation. However, the present study provides an all-encompassing review of sediment sampling and its pre-treatment, older as well as new extraction, purification and concentration processes, assessment of analytical methods, green chemistry methods, and the possible impact of organic pollutants from sediments that come into contact with surface waters on public health. A lot of attention is paid to the evaluation of extraction methods and their effectiveness, solvent consumption, and practical issues in order to formulate a comprehensive vision for environmental labs as well as analytical labs [6].

Consequently, sediment analysis is pertinent not only to analytical and environmental chemistry but also to biological and public health evaluations, as contaminant levels in sediments can yield significant insights into potential exposure and ecological hazards [7].

Organic pollutants accumulating in sediments could incur grave environmental health risk whereby sediments then act as fish traps where these pollutants persist in aquatic environments. Pollutants can enter aquatic food webs, eventually impacting people through contaminated water supply, fish, and other seafood. In this context, reliable monitoring of sediments for organic pollutants is of utmost importance for environmental assessment, identification of potential exposure risks, and advancement of public health protections. Reliable sample preparation, extraction, and determination will make an indispensable contribution towards the identification of pollutants at trace levels and data of significant value in environmental and public health evaluations [1, 8].

This evaluation was carried out on the basis of narrative literature research that included keywords about sediments, organic pollutants, sample preparation, extraction, microextraction, and testing techniques. The peer-reviewed studies reviewed were based on extraction parameters, use of the solvent, time spent on extraction, recovery, detection limits, reproducibility, and restrictions. The results were grouped based on extraction and analytical methods to make the comparison of traditional and modern extraction methods easier.

2. REVIEW METHODOLOGY

This assessment was performed by evaluation of the literature related to the sampling, pretreatment, extraction, purification, and analytical identification of organic pollutants in sediments interacting with surface water. Pertinent literature was located via searches in Scopus, Web of Science, PubMed, and Google Scholar employing various combinations of keywords associated with sediment, surface water, organic pollutants, sample preparation, extraction, microextraction, purification, gas chromatography, liquid chromatography, mass spectrometry, and environmentally friendly analytical techniques. The study in question was selected because of its relevance to sediment preparation, extraction effectiveness, analytical evaluation, ecological significance, and possible consequences for public health. The authors emphasized those works that contained the information concerning extraction parameters, solvents, extraction time, recovery rates, detection limits, and methodology limitations. Those studies that did not have relevant information regarding organic contamination in sediment analysis or did not provide valuable methodological information were excluded from the analysis. The articles were organized according to the pre-treatment process for sediment samples, extraction and purification techniques, analytical methods, and green analysis methods.

3. TYPICAL SEDIMENT PREPARATION PROCEDURES

Sediments typically require pretreatment to extract the target substances from the matrix when being contacted with surface water before examination. Samples are usually dried and transformed to a homogeneous particle size and sieved to eliminate big particles [9] .

Then organic compounds are extracted by Soxhlet processing Ultrasound Aided Extraction Process (UAE), Pressurizing Liquid Extraction (PLE) or Assistant Microwave Extraction (MAE) depending on analyte and matrix type. The coextracted impurities are removed by standard solid-phase extraction (SPE) or column chromatography, and the extract is concentrated by rotational evaporation or nitrogen blowdown. The extract is filtered and stored in a way that ensures durability and conformity with GC, HPLC or GC-MS [1].

3.1. Drying and homogenization: ambient air or oven drying at 40 °C. The samples are ground in a traditional mechanical grinder or a standard mortar and pestle to obtain a

homogenous substance with a consistent particle size which enables for reliable extraction [10, 11].

3.2. Sieving and Grinding: Sediment samples are often passed through sieves to remove larger particulates and obtain a more uniform particle-size distribution. The reported sieve size varies in studies; thus the range of 63–500 μm refers to different particle sizes rather than a single standard sieve size. The particle size affects analyte recovery, since smaller particles have a bigger surface area and could also be better adsorbents of organic impurities. Therefore, the sieve size used should be compatible with the properties of sediment and the target analytes [11, 12].

3.3. Filtering Process and Standard Storage Methods: Before instrumental analysis, extracts are filtered to eliminate particles. Labile compounds have to be preserved at low temperatures according to inert atmosphere to prevent breakdown [13].

3.4. Choice of analytical techniques: The pretreatment process must be compatible with the detection method chosen (GC, HPLC, LC-MS or GC-MS). Appropriate pretreatment improves sensitivity, reproducibility, and accuracy [14].

4. SOLVENT-BASED SEDIMENT PRETREATMENT AND EXTRACTION TECHNIQUES.

Solvent extraction methods are chosen based on the chemical attributes of the target compounds and the qualities of the sediment matrix. Traditional Soxhlet extraction guarantees the effective extraction of the samples, but it requires large volumes of solvents and long extraction times. Ultrasound-Assisted Extraction, Pressurized Liquid Extraction and Microwave-Assisted Extraction can reduce the time and volume of solvents used in the extraction process and improve the effectiveness of the extraction. Following extraction, suitable purification and concentration may be required to remove co-extracted matrix materials and to provide an extract suitable for chromatography or mass spectroscopy examination.

4.1 Soxhlet Extraction: Soxhlet extraction has been the most extensively used method for separating organic chemicals from naturally occurring sediments for several decades. A specified quantity of dried and uniform sediment is positioned in a cellulose or glass-fiber thimble and subjected to continuous extraction with an appropriate solvent under reflux conditions. The solvent circulates through the sample and reverts to the boiling flask, facilitating ongoing extraction. Based on the polarity characteristics of the chosen analytes, common options involve dichloromethane, acetone, hexane, or a 1:1 (v/v) hexane-acetone mixture. The solvent constantly percolates through the sediment dissolving the adsorbed organics and transfers them to the boiling flask; exhaustive extraction typically requires 6–24 h in total (many reflux cycles). The extract is concentrated by rotary evaporator and refined through silica gel and/or alumina column chromatography in order to eliminate coextracted lipids, pigments and other interferences. The purified extract can be analyzed by GC–MS or HPLC, depending on the properties of the target analytes. The primary disadvantages are the long extraction time and large solvent consumption. [15, 16].

4.2 Ultrasound-Assisted Extraction (UAE; also known as sonication extraction)**:** this is rapid and efficient for extracting organic compounds from sediment in contact with surface water. The method employs ultrasonic frequencies (20-40 kHz) on a combination of solvent and sediment to produce cavitation bubbles. When they collapse, they create localized high pressure and temperature that disturbs the sediment matrix and favors solvent penetration and faster analyte dissolution and normal desorption. A typical method is to combine an air-dried homogenized sediment sample with a conventional organic solvent including dichloromethane, acetone, hexane or a mixture thereof in a glass jar and sonicate for 15 to 30 minutes using an ultrasonic bath or probe. The extract undergoes filtering or centrifuging, concentrating, and purification, if necessary, by a conventional silica gel or even alumina column and is assessed by GC or HPLC, as appropriate. The UAE approach is more effective than the Soxhlet technique, using a smaller quantity of solvent and fewer extraction cycles. It is a rapid, inexpensive and more environmentally acceptable method to filter organic pollutants in sediments exposed to surface water [17-20].

4.3 Pressurized Liquid Extracting (PLE): This method is also known as accelerating solvent extraction (ASE). It extracts organic molecules from complex solid matrix compounds by using elevated temperatures (50-200 °C) along with pressure (100-300 psi) to maintain solvents in liquid state over their typical boiling points, which increases the solubility of analytes and improves mass transfer. Homogenized sediment specimen is added to the extraction cell containing an inert dispersion agent (e.g. diatomaceous earth, or sand) to avoid channeling and subsequently taken out with hexane, dichloromethane, acetone or mixes thereof for several cycles under regulated conditions. The obtained extract is highly concentrated and purified using SPE or column-based chromatography prior examination by GC-MS or HPLC. PLE has shorter assessment time, lower solvent consumption, a possibility for automation and greater reproducibility compared to Soxhlet extraction [21-23].

4.4 Microwave-Assisted Extraction (MAE): MAE is a technique that uses microwave energy to enhance the extraction of organic molecules from solid matrices, including sediments that interact with surface waters. The sediment is suspended in a microwave transparent tank with an appropriate solvent, often polar or polar/non-polar including methanol, methyl- tert-butyl ether or water/organic solvent combinations. Microwave radiation heats the solvent and matrix quickly, allowing the solvent to penetrate, and the analyte to dissolve and desorb. The technique is performed at controlled temperatures (50-200 °C) and pressure. The extract is obtained, cooled and reconstituted by a rotary evaporation or the nitrogen stream and then purify it by SPE or column-based chromatography prior to GC-MS along with HPLC analysis. MAE has quicker extraction time, reduced solvent use, higher efficiency and improved reproducibility compared with conventional techniques [24-26].

4.5 Supercritical Fluid Extraction (SFE): a supercritical fluid serves as the extractant. Near its critical temperature and pressure, its solvating power of the fluid can be tuned by

adjusting operating conditions. SFE is most commonly applied to solid matrices and is used for both analytical sample preparation and large-scale processing (e.g., decaffeination). Target compounds partition into the supercritical mobile phase and are carried out of the matrix.

SFE provides fast extraction with minimal use of traditional solvents and is suitable for pollutants like polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), pesticides, and petroleum hydrocarbons. Nonetheless, its application can be constrained by the necessity for high-pressure apparatus and the imperative for optimization, especially concerning polar analytes [27-30].

5. ALTERNATIVE AND MINIATURIZED EXTRACTION TECHNOLOGIES

Besides conventional solvent extraction methods, there are several alternative and micro-scale techniques available to extract and pre-concentrate organic contaminants from sediment samples. Liquid-liquid extraction (LLE) and solid-phase extraction (SPE) generally require liquid extracts taken from solid sediment samples after initial extraction. On the other hand, solid-phase microextraction (SPME) and stir-bar sorptive extraction (SBSE) often permit the possible omission of formal extraction. These methods can be performed on sediment samples directly, in their vapor phase and even in the water phase associated with the sediment. Thanks to significantly lower consumption of solvents, these methods have a great potential to replace existing methods of trace organic contaminant examination

5.1. LLE separates components of a liquid mixture by contacting it with an immiscible solvent that preferentially dissolves one or more target components. Analyte distribution between the two phases depends on their relative affinities for each phase [27].

5.2. SPE is used for isolation, enrichment, and purification of target compounds from aqueous solutions. The aqueous sample passes through a solid sorbent that retains the analytes, which are then eluted with a small volume of solvent. Relative to LLE, SPE requires less solvent, avoids emulsion problems, and is more amenable to automation.

5.3. Solid-Phase Microextraction (SPME) is a reduced-solvent extraction method that integrates sampling, extraction, and preconcentration into one process. A coated fiber is subjected directly to the sediment sample, its headspace, or a related aqueous phase, contingent upon the analytical methodology and target substances. Organic pollutants are enclosed by the fiber coating and then released for their necessary study, usually by means of GC or LC. The method of SPME allows for the reduction of solvent spent and time of extraction, so it is especially feasible in the case of the identification of volatile and semivolatile organic pollutants.

5.4. Stir-Bar Sorptive Extraction (SBSE) is an additional reduced-solvent method that relies on the sorptive extraction of organic substances onto a magnetically coated stir bar. The stir bar may be utilized with an aqueous phase, sediment-associated extract, or headspace, contingent upon the analytical setup. Following extraction, the preserved analytes may undergo heat or solvent desorption before chromatographic evaluation. SBSE offers significant extraction capability and can be advantageous for the preconcentration of trace organic pollutants while considerably minimizing the consumption of organic solvents.

6. COMPARISON OF EXTRACTION METHODS FOR SEDIMENTS IN CLOSE CONTACT WITH SURFACE WATER

Table 1 outlines the main extraction methods used for preliminary treatment of sediments in contact with surface water. Soxhlet extraction is comprehensive but demanding in time and solvent. The UAE is faster, uses less solvent, and is hence more environmentally sustainable. Both PLE and MAE are fast, efficient, and reproducible, but they require special equipment and are expensive to set up. The new methods give significant improvements in efficiency, solvent reduction and analytical performance in comparison with the Soxhlet extraction [20, 23, 28, 29] (Table 1). Table 1 outlines the key extraction methodologies examined in this segment, comprising Soxhlet extraction, ultrasound-assisted extraction (UAE), pressurized liquid extraction (PLE/ASE), microwave-assisted extraction (MAE), and supercritical fluid extraction (SFE). The methodologies vary in their extraction mechanisms, solvent requirements, operational parameters, suitability for various contamination categories, equipment demands, and possible constraints.

Consequently, Microextraction techniques such as Solid Phase Microextraction and Stir Bar Sorptive Extraction can be directly applied on relevant sample phases or on liquid extracts obtained from sediment samples. For this reason, these techniques must be distinguished from conventional solid–liquid extraction methods and cannot be considered as the same [[20, 23, 28, 29].

Numerous extraction techniques examined in this analysis are also reflected in established standard methodologies. The US EPA SW-846 series encompasses methodologies for Soxhlet extraction (3540C), pressurized fluid extraction (3545A), microwave extraction (3546), ultrasonic extraction (3550C), and supercritical fluid extraction (3560–3562), along with associated techniques for identifying particular organic pollutants. These standardized protocols offer defined structures for the production and examination of organic pollutants in solid matrices, such as soils and sediments. Determinative methods commonly paired with these extractions include US EPA Methods 8270 (semivolatile organic compounds by GC-MS), 8081 (organochlorine pesticides by GC), 8082 (polychlorinated biphenyls by GC), 8141 (organophosphorus compounds by GC) and 8310 (polycyclic aromatic hydrocarbons by HPLC). Comparable ISO soil-quality standards, for example ISO 18287 (polycyclic aromatic hydrocarbons by GC-MS) and ISO 10382 (organochlorine pesticides and polychlorinated biphenyls by GC with electron-capture detection), are often adapted for sediments [31, 32]. As these methodologies illustrate, the choice of an extraction method must take into account both the specific contaminant of interest and the relevant regulatory or laboratory guidelines.

Table 1 A comprehensive analysis of extraction techniques employed for the pretreatment of sediments interacting with surface water [30, 33-35].

Technique	Principle	Advantages	Typical Applications	Limitations	Cost Effectiveness	Relevant standard method
Soxhlet Extraction	Endless continuous hot solvent reflux through the sediment for many cycles, with analytes solubilising up to saturation	Exhaustive extraction, well-established, simple setup.	Semi-volatile and non-volatile organic compounds in soils, sediments	Solvent consumption, time-consuming (6–24 h), energy intensive	Moderate; low-cost equipment but high solvent and energy costs	EPA SW-846 3540C
Ultrasound-Assisted Extraction (UAE)	Ultrasonic waves produce microscopic boiling in solvent–sample mixture that improves mass transfer	Short reaction times, reduced solvent consumption and simple operation under mild conditions.	Polar and semi-polar organics: pesticides PAHs in sediments and soils	Solvent penetration may be limited in dense or heterogeneous matrices; this can be mitigated by optimizing solvent, sonication time and power	Cheap ultrasonic bath, less solvent used; consumes less energy.	EPA SW-846 3550C
Pressurized Liquid Extraction (PLE/ASE)	By keeping the solvent liquid above its boiling point, solubility and mass transfer may be increased by high temperature and pressure	Fast, reproducible, high recovery possible with minimal solvent usage; automation friendly.	Sediments and soils contaminated with PAHs, PCBs, pesticides and other hydrophobic pollutants	Needs non-standard equipment; high capital investment; less suitable for thermolabile compounds	Moderate to low; entire equipment is costly, but cost of operation using less solvent	EPA SW-846 3545A
Microwave-Assisted Extraction (MAE)	Microwave energy rapidly heats the solvent and sample matrix, enhancing analyte desorption and mass transfer.	Rapid extraction; reduced solvent consumption; efficient heating; simultaneous processing of multiple samples is possible.	PAHs, pesticides, phenolic compounds, and other organic contaminants in sediments and soils.	Requires microwave-compatible extraction vessels and controlled operating conditions; non-uniform heating may occur depending on matrix composition.	Moderate; Equipment costs are higher than for conventional UAE, but reduced extraction time and solvent consumption may lower operating costs.	EPA SW-846 3546
Supercritical Fluid Extraction (SFE)	Supercritical fluids, commonly CO_2, penetrate the solid matrix and dissolve target analytes under controlled temperature and pressure conditions.	Low solvent residue; reduced use of conventional organic solvents; tunable extraction properties; suitable for thermally sensitive compounds.	Non-polar and moderately polar organic contaminants, including hydrophobic environmental pollutants.	Requires high-pressure equipment and careful control of temperature and pressure; extraction of polar compounds may require modifiers.	Relatively high initial equipment cost; potentially lower solvent use and waste generation.	EPA SW-846 3560–3562

7. COMPARISON OF DETECTION METHODS

The choice of chromatographic detectors depends on analyte volatility, polarity, and required detection sensitivity. GC-based methods are preferred for hydrophobic, semi-volatile organics, while LC methods are better suited to polar and thermally labile compounds. Table 2 presents a concise comparison of the principal detection approaches [27, 36-39].

Table 2: Comparison of chromatographic and mass spectrometric detection methods for organic contaminants in surface water-contact sediments.

Technique	Typical detectors	Best-suited analytes and strengths
Gas chromatography-mass spectrometry (GC-MS)	Quadrupole mass spectrometry, selected ion monitoring or full scan	Used primarily for PAHs, PCBs and some pesticides and other semivolatile compounds; identification conferred by specificity/ library correlation.
GC with selective detectors (ECD, FID and FPD)	Electron Capture Detector (ECD) for halogenated compounds; Flame Ionization Detector (FID) for hydrocarbons; Flame Photometric Detector (FPD) for sulfur- and phosphorus-containing compounds.	ECD is very sensitive to organochlorines, PCBs and chlorinated pesticides. FPD for organophosphates in standard validated USGS methods
High-performance liquid chromatography (HPLC)	Diode-array detector (DAD) and other optical detectors; hyphenated LC-MS	Ionic-liquid-based extraction combined with HPLC-DAD was applied to marine sediments and is useful for polar organic compounds and specific phenols.
Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS)	High-throughput assays using triple quadrupole or high-resolution mass spectrometry	Critical to pharmaceuticals, polar agrochemicals, and many new toxicants; observed in assessments supporting hyphenated methods
Comprehensive GC×GC-TOFMS	Two-dimensional gas chromatography time-of-flight mass spectrometry	Suitable for complex mixtures, biomarker characterization and the resolution of co-eluting compounds in port and harbour sediments
High-resolution MS for organic matter	FT-ICR or Orbitrap instruments	Mainly used for total organic matter characterization and non-targeted molecular profiling, since not suitable for standard quantification

8. COMPOUND-SPECIFIC PROTOCOLS

Different analyte classes require tailored protocols for sampling, extraction, cleanup, and detection. Table 3 summarizes the recommended procedures for the major classes of organic contaminants found in sediments in contact with surface water [24, 27, 29, 40].

Table 3: Protocols used to collect and treat class-specific samples of major classes of organic contaminants found in sediments in contact with surface water, PAHs (polycyclic aromatic hydrocarbons), PCBs (polychlorinated biphenyl). Organochlorine and organophosphate pesticides; oil hydrocarbons; phenolic molecules; volatile/semi-volatile organics/emerging contaminants [1, 2, 22, 25, 41].

Compound class	Sampling/pretreatment	Recommended extraction / cleanup	Detection and notes
PAHs	Composite Depositional Finer Fraction and/or Surface Collections; sift to organic fraction, dry.	Solvent extraction with Soxhlet as well as benzene/hexane or Accelerated Solvent Extraction; fractionation by silica/florisil; desulfurization if needed.	Quantification via GC-MS or traceable with a FID; better peak quality and recovery validation by fractionating the sample.
PCBs (congeners)	0–1 cm surface samples; freeze or freeze-dry and homogenize	Clean-Up Procedures: Soxhlet extraction (toluene refluxed for hours) or Accelerated Solvent Extraction; Gel Permeation Chromatography (GPC); Florisil/silica clean-up before concentration.	GC-MS or GC-ECD using isotopic/internal standards (standards labeled with 13C used to quantify)
Organochlorine pesticides	Composite surface sediments; acid pretreatment or neutral preparation as required methods	Headspace solid phase microextraction (SPME)	Extraction methods based on Solid Phase Extraction (SPE) with diverse GC ECD as well as GC MS methodologies are also available for multi OCP complexes, characterized by a detection limit at the low ng/g level.
Organophosphate pesticides	extracting free water from surface sediments	Solvent extraction (dichloromethane), followed by concentration and purification.	Identification of Selected Organophosphates by Gas Chromatography with Flame Photometric Detection (GC FPD)
Legacy pesticide mixes / OCP + PCB multi-residue	Composited fine fraction, careful chain-of-custody	Soxhlet/ASE Extraction with Multi-fractionation Silica/Alumina Desulfurization by florisil purification and selective gel-permeation chromatography	Validated multi-analyte methods have been published: GC-ECD or GC-MS confirmation, with surrogate corrections. .
Total Petroleum Hydrocarbons / TPH	Surface or estuarine sediments; determine TOC (total organic carbon)	Headspace SPME	Quantification and pattern analysis of TPH by GC FID; Interpret concentrations on an organic carbon–normalized basis.
Phenolic compounds and alkylphenols	Surface sediments frequently necessitate derivatization.	Gas chromatography employing solvent extraction with suitable polar solvents followed by purification and derivatization.	Phenolic GC–MS methodologies utilizing derivatization have been documented for bisphenol A and nonylphenol. HPLC techniques have likewise been employed for their quantification. Gas chromatography techniques can utilize either isothermal or temperature-programmed settings, contingent upon the analytes and the derivatization method.
Volatile and semi-volatile organics	Pretreatment methods (water leach, slurry and sonicated methanol)	Leachate or batched flush and trapping, or heat ionization sorbent-based traps are confirmed to give recovery efficiencies of about 75–92% depending on pretreatments.	Purge-and-trap GC-MS provides accurate quantitation of volatiles; appropriate concentration techniques can be used to maximize analyte recovery.
Pharmaceuticals and many polar emerging contaminants*	Limited data in the literature to support standardized methods of sediment administration.	Insufficient evidence	Not enough evidence to recommend any validated, sediment extraction and detection workflows from the papers presented

Pharmaceuticals and various polar emerging contaminants present analytical difficulties because of their heterogeneous characteristics and robust interactions with sediment substrates. The pH and matrix composition can influence their extraction and recovery. Consequently, enhanced extraction, purification, and method verification are necessary, with LC–MS/MS being employed for precise measurement.

Quantitative method-performance metrics offer a more insightful foundation for evaluating sediment extraction techniques compared to qualitative descriptions alone. Consequently, pertinent studies were scrutinized for documented recovery, accuracy, and detection thresholds. The values in Table 4 are presented as study-specific representative outcomes rather than universal performance ranges, due to their substantial dependence on target analytes, sediment properties, extraction conditions, cleanup methods, and detection methodologies. The data indicate that both traditional and contemporary extraction methods can yield acceptable analytical results, while their quantitative attributes differ significantly among contaminant categories and analytical setups.

Table 4. Representative quantitative analytical performance of extraction methods for organic contaminants in sediment.

Extraction method	Target contaminants	Matrix	Recovery (%)	Precision (%RSD)	LOD/MDL	Reference
Soxhlet extraction	Organochlorine pesticides	Sediment	88–110	5.1–7.9*	0.6–2.1 ng/g	[42]
Microwave-assisted extraction (MAE)	Organochlorine pesticides	Sediment	74–98	4.5–9.8*	0.6–2.1 ng/g	[43]
Ultrasound-assisted extraction (UAE)	PAHs and aliphatic hydrocarbons	Marine sediment	>90	Not numerically reported	Not reported	[44]
Ultrasound-assisted extraction	PAHs	Marine sediment	87–107	2.0–6.8	Not reported	[45]
Pressurized liquid extraction (PLE) + SPE	Industrial/waste-indicator organic compounds	Sediment	76 ± 13	Not reported	12.5–520 µg/kg	[23]
QuEChERS* + UPLC-MS/MS	Multi-class emerging contaminants	Soil/sediment	70–120	<20 in most cases	MLOQ 0.2–10 µg/kg	[2]
Microwave-assisted extraction (MAE)	14 PAHs	Certified sediment reference material	97	Not reported	Not reported	[26]

* QuEChERS: (Quick, Easy, Cheap, Effective, Rugged, and Safe); MDL, method detection limit; MLOQ, method limit of quantification.

According to numerical data, no method of extraction can ensure superior efficiency across the board in all categories of organic contaminants. Although Soxhlet extraction can produce considerable recoveries with tough-to-extract hydrophobic substances, it usually requires long extraction times and a lot of solvent. The UAE and MAE techniques can produce high recoveries with short extraction times, while the PLE method ensures effective extraction with low solvent consumption. New multiresidue techniques such as QuEChERS combined with LC-MS/MS have been developed for simultaneous determination of multiple chemicals with acceptable recoveries and sensitivity within low quantification limits. Hence, it is essential to select the appropriate extraction method based on the physical and chemical properties of the target analyte, the nature of the sediment, requirements for sensitivity, solvent consumption, and the analytical method being employed.

9. GREEN AND SUSTAINABLE EXTRACTION TECHNOLOGIES AND SOLVENT

Recent advancements in eco-friendly analytical chemistry have concentrated on minimizing solvent usage, dangerous chemicals, energy consumption, and waste production during sample preparation. Contemporary methods can diminish extraction duration and solvent usage in contrast to traditional Soxhlet extraction. Miniaturized and automated methods can diminish the usage of samples and solvents while enhancing analytical efficiency [46].

The choice of extraction solvent is essential. Water, ethanol, supercritical carbon dioxide, and deep eutectic solvents (DESs) have been researched to replace traditional solvents like hexane and dichloromethane. The label "green solvent" must not just be based on the source of the solvent; rather, other factors such as toxicity, biodegradability, volatility, energy requirements, and waste produced must also be taken into account. DESs and ionic liquids (ILs) can help lower volatility and evaporation of the solvent; however, the environmental impact depends on the chemical structure of these substances and needs to be verified and not assumed [47-50]. Ecological analytical metrics offer a numerical method for evaluating extraction techniques. The Analytical Eco-Scale assesses the dangers of solvents and reagents, quantities, energy usage, and waste, while Analytical GREEnness Metric Approach (AGREE) measures analytical methods according to the 12 tenets of green analytical chemistry on a scale from 0 to 1. AGREEprep is dedicated to the sustainability of sample-preparation methodologies.

Consequently, sustainable sediment extraction must take into account extraction efficiency alongside solvent toxicity, solvent volume, energy usage, waste production, and comprehensive analytical performance. This comprehensive evaluation is especially crucial when choosing more sustainable options for regular environmental oversight [51-53].

Figure 1 presents the overarching procedure for sediment analysis, encompassing sampling and pretreatment, followed by extraction, purification, concentration, and instrumental assessment, incorporating both traditional and environmentally friendly extraction methods.

10. QUALITY ASSURANCE, QUALITY CONTROL AND METHOD VALIDATION

Assessing the organic pollutants in sediments requires proper QA/QC procedures. In general, methods include surrogate standards, isotopically labeled internal standards, method blanks, matrix spike, duplicated, and certified reference materials. All the above methods assist to understand the extent of contamination, extraction recovery, and evaluation of accuracy and precision. Most important validation factors include extraction recoveries, precision (%RSD), limit of detection (LOD), limit of quantification (LOQ), linearity, selectivity, and matrix effects. Proper QA/QC and validation protocols are very important for getting reliable and repeatable results in sediment contaminant assessment [5].

11. PROSPECTIVE OUTLOOK

It is expected that future works on sediment preparation and evaluation will be focused on automation, green technology, and increased throughput. The application of Artificial Intelligence can improve extraction criteria and speed up the interpretation of results [54]. The application of sorbents based on nanomaterials such as graphene and magnetic nanoparticles [55] will allow an increase in the total adsorption capacity of the sorbents as well as their specificity [56]. Combining the advantages of the existing technologies with an increased use of green solvents in hybrid extraction technologies can enhance the process efficiency, and reduce solvent consumption as well as the length of the overall analysis and its environmental impact [57, 58].

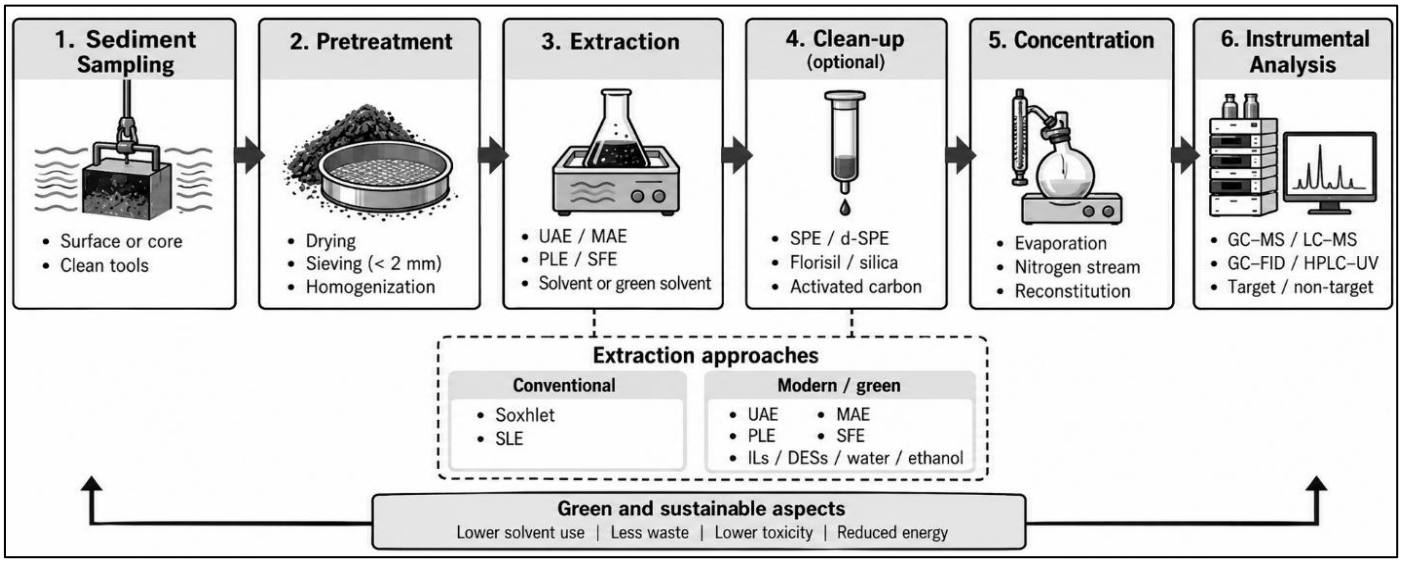


Fig 1. Diagrammatic representation of the workflow for the pretreatment, extraction, purification, concentration, and instrumental evaluation of organic pollutants in sediments, emphasizing traditional and contemporary/eco-friendly extraction methods.

12. CONCLUSION

Sediment extraction is a necessary step for achieving proper collection and measurement of organic pollutant levels from those sediments. Conventional Soxhlet extraction technique enables effective extraction of semi-volatile and non-volatile analytes but it might be a time-consuming process that requires relatively large amounts of solvent. New techniques such as Ultrasound-Assisted Extraction (UAE), Pressurized Liquid Extraction (PLE), and Microwave-Assisted Extraction (MAE) help avoid many of the limitations of traditional methods and deliver the needed results in less time using smaller quantities of solvent. UAE is an attractive technique to be used in daily practice due to its simplicity and low cost. PLE and MAE are very effective for the extraction process and can be automated. Supercritical fluid extraction (SFE) allows the use of smaller amounts of organic solvents and control of the selectivity of the process but requires technology that applies high pressures and specialization of the setup for polar analytes. New methods do not solve all issues in sediment analysis. For instance, UAE optimization may be required for heterogeneous or dense matrix materials. The PLE and MAE techniques require high-technology equipment and careful control of temperature and pressure. In addition, heat-sensitive analytes may decompose when operated in hot environments. Hence, the selection of the extraction technique should depend on the properties of the analyte, the complexity of the matrix, the required degree of analytical sensitivity, available equipment, and economic considerations. It must be noted that the choice of solvent and method adjustment are vital; nevertheless, the use of green solvents and modern extraction technologies may help to diminish environmental impact. Using advanced extraction techniques allows saving time and solvent compared to traditional techniques; however, their drawbacks should be considered when creating a practical analytical protocol for complicated sediment samples.

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Conflict of interest

The authors declare no conflicts of interest.

Ethical Approval

Not applicable. The review article does not involve any studies dealing with humans or animals performed by the author.

Author contributions

Bahaa Malik Altahir. Conceptualization; Formal analysis; Investigation; Methodology; Project administration; Data curation; and Writing – original draft; Writing – review and editing.

Keith Edward Taylor. Formal analysis; Validation; Visualization; and Writing – review and editing. Supervision; Project administration; Writing – original draft; and Writing – review and editing.

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AI declaration

The authors used Claude AI & Gemini Pro to improve the manuscript's academic tone and readability. After using this tool/service, the authors reviewed and edited the content as needed and took full responsibility for the publication's content.

Data availability

Data will be made available on request.

13. REFERENCES

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