ANNUAL REVIEWS

Annual Review of Analytical Chemistry

Innovative Sample Preparation Strategies for Emerging Pollutants in Environmental Samples

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Annu. Rev. Anal. Chem. 2025. 18:73–95

The *Annual Review of Analytical Chemistry* is online at anchem.annualreviews.org

https://doi.org/10.1146/annurev-anchem-071224-093709

Keywords

sample preparation, environmental matrices, cleanup, emerging devices, functional materials, organic pollutants

Abstract

Sample treatment plays a crucial role in ensuring accurate analysis of contaminants in aqueous, gaseous, and solid matrices. Emerging contaminants such as microplastics and poly- and perfluoroalkyl substances pose challenges due to their ubiquity and potential adverse effects on the environment and human health. By setting stringent guidelines, environmental protection agencies drive research and innovation in analytical methodologies. However, current reference methods are based on traditional techniques with a high use of chemicals and considerable waste generation. This review highlights the importance of advanced techniques, including solid-phase extraction and microextraction methods, enhanced by novel materials, for preparing environmental samples. Additionally, it discusses innovative formats and devices, such as drone-based systems and three-dimensional-printed devices, which are expanding the scope of environmental monitoring. This review aims to provide a comprehensive overview of trends and advances in sample preparation for environmental analysis over the past five years, offering insights into progress made and future directions.

1. INTRODUCTION

Environmental analysis is conducted to meet regulatory standards, respond to emergencies, perform routine control, and conduct scientific investigations into the fate and transport of anthropogenic and natural chemicals (1–3). Effective monitoring and mitigation of pollutants require robust, precise, and transferable analytical methods as the environmental and health implications of these compounds become increasingly evident to humans and wildlife. Among emerging contaminants, microplastics (4, 5) and poly- and perfluoroalkyl substances (PFAS) (6, 7) have garnered significant attention due to their persistence, ubiquity, and potential adverse effects on ecosystems and human health. Major environmental protection agencies, such as the United States Environmental Protection Agency (EPA), the European Environment Agency (EEA), and the International Agency for Research on Cancer (IARC), play a critical role in regulating contaminants and setting operating standards for their analysis. These organizations set stringent guidelines and regulations to safeguard environmental quality and public health. Their contributions are vital in driving research and the development of new methodologies to effectively address the challenges posed by emerging pollutants. However, the importance of environmental analysis extends beyond just identifying contaminants. It encompasses understanding the pathways by which pollutants enter and move through the environment, assessing their long-term impacts, and developing strategies for their mitigation (8–10). Given the continuous evolution of pollutants and the complexity of environmental matrices, the field of analytical chemistry must remain dynamic and innovative.

Environmental samples are complex matrices with highly varied compositions, where the presence of interferents at concentrations higher than those of the targeted analytes raises the potential for bias in the results. Furthermore, analyte concentrations can range from percentages to low part-per-trillion levels, with the latter scenario requiring an efficient preconcentration step. The complexity of environmental monitoring also drives developments in (*a*) sampling techniques, particularly passive methods; (*b*) the design of new samplers for remote testing of areas that are not easily accessible by operators [such as industries with hydrogen sulfide (H_2S) residues or the ocean depths]; and (*c*) approaches to preserve homogeneity in very large amounts or volumes of diverse samples. For all of these reasons, sample preparation is crucial in environmental analysis because it directly influences the accuracy, reliability, and sensitivity of the results. Advances in this field have led to the development of innovative techniques such as solid-phase extraction (SPE), solid-phase microextraction (SPME), dispersive liquid–liquid microextraction (DLLME), and liquid-phase microextraction (LPME) (3, 11). These techniques offer miniaturized formats and convenient solutions for chemical analysis (**Figure 1**).

Examples of the main miniaturized techniques for sample preparation and analytical extractions are listed below.

- Cartridges packed with sorbent material are used in SPE (12). They offer high efficiency in isolating analytes, are compatible with automation, and allow for reproducible results. However, they generate significant waste and require relatively large sample volumes compared to other miniaturized methods.
- The spin column format is a variant of SPE to purify nucleic acids or other molecules. A solid matrix, like silica, is packed into a cartridge. The sample is added to the column, and a vacuum or centrifuge pulls the solution through the column. The desired molecules bind to the solid matrix, while other molecules pass through, allowing simultaneous sample processing and low waste generation (13, 14). Though simple and efficient, it often requires additional buffers and solvents.

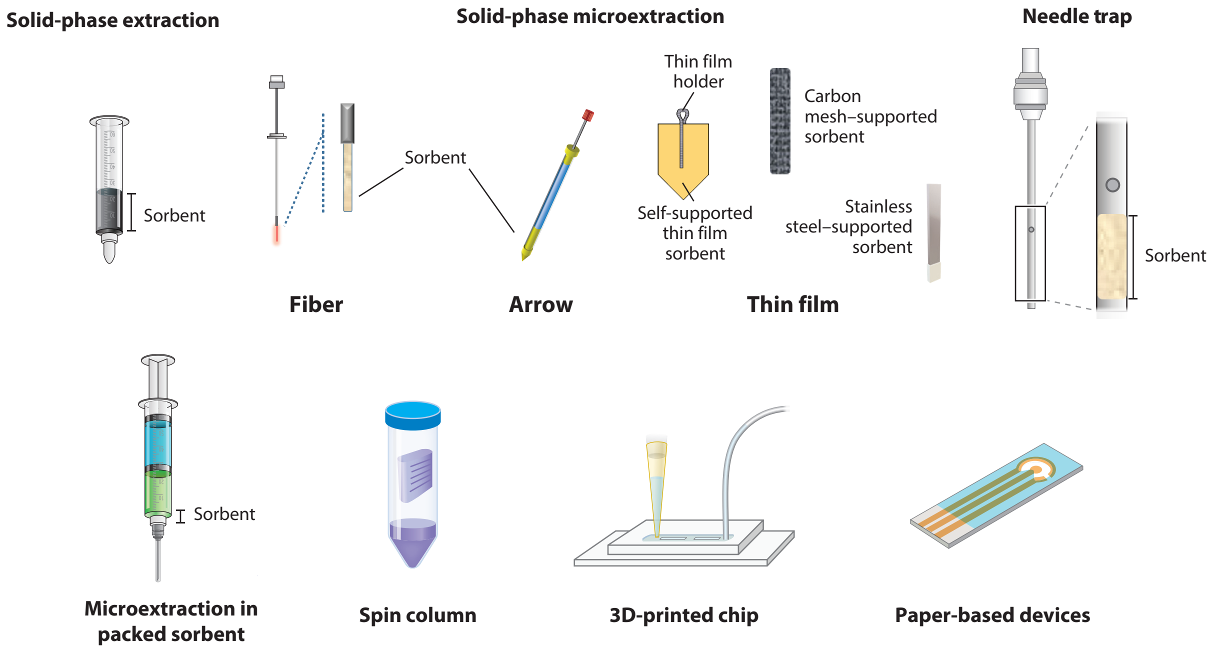

Figure 1

Examples of common solid-phase extraction and solid-phase microextraction configurations and other miniaturized sampling devices. Abbreviation: 3D, three-dimensional.

- Paper sorptive phases involve using paper as the sorbent substrate (15, 16). This format is cost-effective, environmentally friendly, and easily accessible for low-resource laboratories. It generates minimal waste and is ideal for small-scale analyses. The limitation lies in its lower extraction capacity and the need for precise optimization to achieve high sensitivity.
- Pipette tip SPE (PT-SPE) packs sorbent in a tip normally through polymerization, allowing for increased throughput with semiautomatic pipettes (17). A key benefit is sorbent reusability, which enhances cost-efficiency and reduces waste. However, the process may require multiple extraction cycles to achieve the desired analyte recovery.
- Microextraction by packed sorbent (MEPS) involves placing the sorbent directly in a syringe, which enables low-volume sampling and quick processing, as reported in several bioanalytical applications (18). Although it offers fast results and opportunities for automation, the method is limited by the sorbent choice and setup.
- SPME devices consist of sorbent-coated supports that extract analytes directly from the sample or its headspace (19). They offer simplicity and minimal solvent use and can be used for in situ sampling. SPME geometries include, but are not limited to, fibers, arrows, and thin films (**Figure 1**). SPME fibers are widely used and commercialized and are compatible with commercially available autosamplers. Thin-film SPME (20) devices are gaining attention, as they offer greater extraction capacity than fibers; moreover, the higher surface area to volume ratio allows for fast extraction times.

Additionally, drone-based systems and three-dimensional (3D)-printed devices and sorbents are emerging formats and devices that enable innovative sampling strategies (21–23).

The design and synthesis of new chemistries for solid sorbents and solvents have significantly improved the efficiency of extraction techniques by improving extraction efficiency and selectivity.

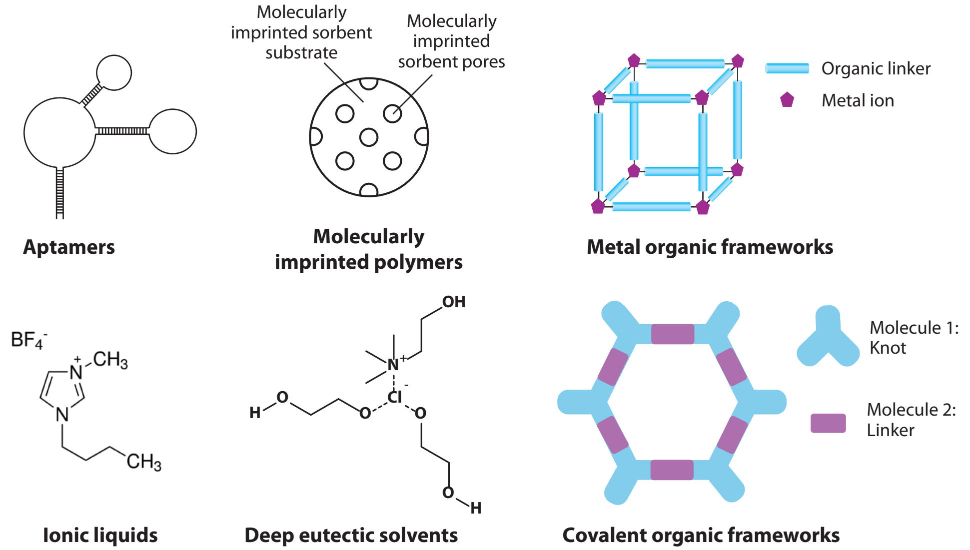

Figure 2

Examples of alternative sorbents used for sample preparation: aptamers, molecularly imprinted polymers, metal-organic frameworks, covalent organic frameworks, ionic liquids, and deep eutectic solvents.

Molecularly imprinted polymers (MIPs), metal-organic frameworks (MOFs), covalent organic frameworks (COFs), aptamers, ionic liquids, and deep eutectic solvents are increasingly applied to the field of sample preparation (**Figure 2**), pushing the boundaries of what is achievable in separation chemistry (24, 25).

The detection of contaminants using techniques with reduced environmental impact is of great importance to the scientific community. The analytical chemistry community also aims to improve the sustainability of research practices. To achieve this goal, various principles have been formulated in the field of analytical chemistry broadly (26) and in sample preparation specifically (27). In this context, the greenness of methods can be calculated and compared using various metrics, which are described in the literature (28, 29). Among all the methods, AGREE (Analytical GREEnness Metric Approach and Software) is a tool designed to quantify the environmental impact of electronic systems, focusing on energy efficiency and sustainability throughout their lifecycle. It provides a graphical color-coded summary ranging from red (worst) to green (best) from a comprehensive assessment of energy consumption, material use, and environmental trade-offs in design and production (30).

This review aims to provide an in-depth analysis of current trends and advances in environmental analysis and sample preparation over the last five years. By exploring the latest techniques and materials used in sample preparation and analytical extractions, we highlight progress made in enhancing analytical performance and ongoing efforts to monitor persistent pollutants. We also discuss methods to address the different phases of environmental samples: gaseous, liquid, and solid. Finally, the implications of these advances are pointed out, highlighting examples and areas for future research.

2. ENVIRONMENTAL SAMPLES

The accuracy and reliability of environmentally relevant data largely depend on the initial sampling process, which involves collecting representative samples from matrices such as air, water, soil, and biota (31, 32). These samples often exhibit considerable spatial and temporal variability, complicating the task of obtaining representative sampling. Moreover, various steps involved in sample preparation, such as filtration, extraction, and preconcentration, can potentially introduce contamination, loss of analytes, and matrix effects during the analytical workflow. Addressing these challenges requires meticulous planning, adherence to standardized protocols, and the development of advanced techniques to ensure the integrity and reproducibility of environmental analysis. **Table 1** summarizes the most recent (2019–2024) contributions to sample preparation for environmental analysis, grouped by sample type.

2.1. Gaseous Samples: Air Sampling (Passive/Active) Robotics/Drones and Needle Trap Devices

Analysis of gaseous samples is essential for monitoring air quality and understanding atmospheric processes, yet it presents significant challenges in sampling and sample preparation (33). Accurately capturing and analyzing gaseous pollutants such as volatile organic compounds (VOCs), PFAS, greenhouse gases, and particulate matter require adequate sampling techniques and sample treatment to ensure sample representativeness and avoid cross-contamination. Validating the accuracy and precision of the method is also crucial for the reliability of the results. Hence, the correct sample preparation technique and analytical instrumentation must be carefully selected. Furthermore, variability in ambient conditions, such as temperature, humidity, and light exposure, is a key factor that must be monitored because it can potentially affect the stability and concentration of target analytes (33).

No single air sampling method is suitable for every monitoring scenario. To address specific applications, various sampling strategies have been developed, most of which utilize some form of sorbent tube or trap regardless of the type of sampling used (passive or active). Notable examples include pumped (active) or diffusive (passive) sampling onto sorbent tubes to selectively concentrate vapors from online air streams or whole-air containers, such as canisters or bags (34). Each method has pros and cons. Passive filters offer a low-maintenance and cost-effective approach for long-term monitoring of airborne contaminants but are susceptible to variable environmental conditions. In contrast, active filters use mechanical pumps to draw air through the filter media, providing precise control over sampling conditions and enabling the collection of samples within shorter time frames, but they require power sources and regular maintenance.

More recently, these active filters have been used with cutting-edge advancements such as 3D-printed technology and drone capabilities. Leal et al. (35) developed a 3D-printed micropump for the assessment of air quality near a wastewater plant. This lab-on-a-drone (**Figure 3*a***) technology is lightweight (300 g) and cost-efficient (US$50 per device) and can detect 9 μg L^{-1} of H_2S in 10 min of sampling time. The analytical method applied was based on the reaction between fluorescein mercuric acetate and H_2S, which led to fluorescence quenching. The sample collection was done by using a mini air pump at 50 mL min^{-1} directly to fluorescein mercuric acetate for 20 min. The authors recorded data at three different times of day (7 AM, 12 PM, and 7:30 PM) and at different altitudes, including ground level and 10 m and 20 m above sea level. All data collection was performed via Bluetooth, encouraging the use of this green technology (with an AGREE score of 0.85) in remote, dangerous, or low-resource locations. More traditional air sampling devices are still useful for modern challenges. However, Zhou et al. (36) collected legacy and emerging PFAS near fluoropolymer manufacturing facilities with $PM_{2.5}$ filters (designed to remove from air fine particulate matter that is 2.5 μm or smaller in diameter). Twelve emerging and ten legacy PFAS compounds were detected in 6-day integrated filter samples collected over six months. Thirteen had higher concentrations than the regional background, and the maximum concentration exceeded 1 pg m^{-3} for ten.

Table 1 Recent contributions of sample treatment techniques in environmental analysis

Analytes (number)	Sorbent	Method	Sample	Recoveries (%)	LOD (μg L^{-1})	Reference
Gaseous samples						
PAHs (17), plasticizers (18), and vulcanization agents (4)	Commercial Tenax and SPME fiber	SPE-SPME-GC–MS/MS	Outdoor air	80–110	0.01–4.6 ng m^{-3}	80
PAHs (16)	Supercritical CO_2	SFE-HPLC-MS/MS	Urban dust and diesel particulate matter	22–107	0.16–3.33 ng	81
Microplastics (3)	Air bubbles	μSEP-μRaman and TED-GC–MS	Deionized water, soil, and air	42–77	NA	82
VOCs (5)	Electrospun PAN nanofibers	ITEX-GC–MS	Outdoor air	NA	(7.5–36)·10^{-6}	83
VOCs (11)	MCM-41	ITEX and SPME-GC–MS	Outdoor air	NA	0.0002–9.8 μg m^{-3}	84
Alkanes (C4-C18)	DVB/Car/PDMS	SPME-GC–MS	Air from cooking	31–77	NA	85
Semi-VOCs (140)	Filter + Oasis HLB	Dehumidifier-SPE-GC-TOF-MS	Indoor air	NA	NA	86
VOCs (10)	DVB/Car/PDMS	Drone-SPME-GC–MS	Combustion emissions	NA	0.039–0.136	40
VOCS (347)	PDMS/DVB and MCM-41	Drone-SPME-GC–MS	Outdoor air	65.6–103.4	(1–6,000)·10^{-6}	87
Nitrogenated compounds (20)	Phosphate-modified MCM-41	Drone-ITEX-GC–MS	Outdoor air (boreal forest)	63–95	NA	88
VOCs (83)	Tenax and Carboxen	Quadruped robotic sampler-NTD-MS	Fire fields	NA	0.37–6.99	37
Nitrogenated compounds (11)	Pseudomorphic SiO_2/ZnO	ITEX-GC–MS	Indoor air, cigarette smoke, and outdoor air	NA	0.007	89
Siloxanes (3)	Silica-coated paper	SPE-PS-MS	Outdoor air	NA	NA	90
PAHs (8)	1-Octanol and graphene oxide	VA-DLLME-μSPE-GC–MS/MS	Ambient air particulate matter $PM_{2.5}$	57–88	0.013–0.135	91
PAHs (5)	TMC-BD-COF	NTD-GC-FID	Indoor air	109–120	0.021–0.027	39
SMPs and APFs	Ultrapure water and cold-pressed sunflower oil	LLE-μFTIR	Urban aerosols (summer and winter)	96	NA	92
H_2S	3D-printed micropump	Light-emitting diode and digital light detector	Ambient air close to wastewater plant	NA	9	35
PFAS (16)	C18 SPE	Online SPE-LC–HRMS	$PM_{2.5}$	93–109	(8–50)·10^{5}	93
PFAS (8)	Polyurethane foam, CH_3OH, and carbon-based phase	Filtration, Soxhlet extraction, and SPE-UPLC-MS/MS	Outdoor air (317 positions)	NA	5.2–200 pg per filter	94
PFAS (51)	Filter with activated charcoal	SPE-LPE-GC-MS/MS	Ambient air	0–120	0.002–10	95

(*Continued*)

Table 1 (*Continued*)

Analytes (number)	Sorbent	Method	Sample	Recoveries (%)	LOD (µg L^{-1})	Reference
PFAS (10)	$PM_{2.5}$ filters	SPE-LC-MS/MS	Ambient air close to fluoropolymer manufacturing	70–120	0.0011–0.028 pg m^{-3}	36
PFAS (117)	Cryogenic air sampler	SPE-UPLC–HRMS	Particulate matter and ambient air	50–140	NA	96
Solid and semisolid samples						
Cadmium (CdI_4^{2-})	PANI-ES@paper (1 cm^2)	TFME-FAAS	Tap water, well water, river water, sewage water, seawater, and soil	96–105	0.06	97
Benzydamine hydrochloride	DES@MIP@m-UiO-66	d-SPE-UV-Vis	Sewage water, tap water, river water, and soil	91–108	NA	98
Poly(methyl methacrylate) micro/nanoplastics	DVB/Car/PDMS-coated fiber	CA-SPME-GC-MS	Drinking water, seawater, soil, ocean sediment	76–99	0.014–0.28 µg	58
PAHs (5)	SBA-15-NH_2	CA-HS-MEPS-HPLC-UV	Soil	75–113	0.042–0.25 ng g^{-1}	48
Unsymmetrical dimethylhydrazine products (5)	Car/PDMS	VA-HS-SPME	Sand	84–97	0.12–12 ng g^{-1}	50
PAHs (6) and BTEX (4)	COF/MOF (2DTP/MIL-101-Cr)	HS-SPME-GC-FID	Soil	80–108	0.07–5 ng g^{-1}	49
BTEX (4)	GO-APTES	UA-PR-SPME-GC-FID	Soil	82–120	0.1–0.4 ng g^{-1}	47
Neonicotinoids (5)	AuNPs@NH_2(C_4)-ZIF-8	SPE-HPLC-DAD	Syrup, soil, and river water	80–110	0.019–0.041	99
Triazine pesticides (5)	HLB	UE-SPE-LC-PDA	Soil and sediment	70–100	1–2 µg kg^{-1}	100
Microplastics (6)	Flotation and aeration	ATR-FTIR	Soil and sediments	78–100	NA	53
Microplastics (3)	Methanol-THF and calcined silica gel	PLE-pyrolysis-GC-MS	Soil and sewage sludge	77–131	2.1 µg g^{-1}	57
PFAS (3)	Methanol 0.5% acetic acid (v/v)	LPE-HR-CS-GFMAS	Soil	85–90	3.09 µg kg^{-1}	101
Phthalates (7)	Fe-BTC MOF	MSPD-UPLC-MS/MS	Soil	70–120	0.14–2.7 µg kg^{-1}	102
Plastic additives and contaminants (10)	Florisil and sea sand	MSPD-HPLC-DAD	Mussels	80–100	0.075–4.86 µg kg^{-1}	103
PBDEs (8)	Florisil and alumina	MSPD-GC-MS	Dust from indoor air	76–119	2.1–480 pg µL^{-1}	104
PAHs (8)	Florisil and PDMS fiber	MSPD-CA-SPME-GC-MS	Soil	73–115	4.2–8.5 ng g^{-1}	105
Nitrotoluene pollutants (5)	DES and carbon nitride	MSPD-LC-UV	Soil	78–96	0.12–0.33 µg g^{-1}	106

(*Continued*)

Table 1 (*Continued*)

Analytes (number)	Sorbent	Method	Sample	Recoveries (%)	LOD (µg L^{-1})	Reference
Pharmaceuticals, PCPs, and biocides (59)	CH_3OH	VA-MSPD-LC-MS/MS	Marine sediments	60–140	0.13 ng g^{-1}	45
Microplastics (1)	Hydrolization, C18 sorbent, and methanol	SPE-LC-UV	Marine sediments	98	17.2 µg kg^{-1}	107
Microplastics (1)	Oil-based extraction and Fenton's reagent	LLE-microscope and FTIR	Sewage sludge and organic-rich substrates	40–99	NA	54
Extractable organic matter	Sieving and fumigation	LPE-GC-MS	Soil	8–80	NA	108
Aqueous samples						
Antibiotics (14)	Paper-based polymeric ionic liquid (13 mm i.d.)	TFME-HPLC–MS	Sewage water	79–127	0.44–54.4	109
NSAIDs (3)	Ferrofluid-DES	VALLME-HPLC-DAD	Lake water, river water, and tap water	90–108	1.68–2.05	110
PFAS (4)	SPME (fiber and blade) HLB-WAX/PAN	SPME-DART-MS	Ultrapure water	80–120	0.0033–0.0167	76
Fungicides (17)	Cork by-products	SPE-GC–MS/MS	River water, tap water, rainwater, and spring water	70–118	0.0003–0.1^1	111
Cetirizine dihydrochloride	Lignin-based spherical particles (20 mg)	DSPE-UV	Sewage water	90	NA	112
PAHs (10)	Commercial SBA-15/C8	D-µSPE-GC–MS	Groundwater	92–112	0.15–3.03	113
Bisphenol A	DES@MIP column (10 × 60 mm)	Fixed-bed column-HPLC-UV	Rain and river water	91–108	NA	114
Fe(III)	*N*, *N*-dimethyl-*n*-octylamine solvent + NaOH	SS-LPME-FAAS	Tap water, river water, seawater, and sewage water	NA	0.015	115
Methylene blue	3D-printed support with an obround cross-section (2.5 × 2.5 mm)	µEME-SIA-UV-Vis	Wastewater	81–87	1300	116
Phenolic compounds (7)	NiCr@NiCr LDHs-SO_4 NSAs fiber	SPME-HPLC-UV	River water, rain, and domestic sewage water	87–108	0.015–0.156	117
Cd(II)	CNF-TMHM	DSPE-ICP-MS	Wastewater	92–94	4.17	118
Phenols (5)	UiO-66-NH_2@coated frits	SPE-HPLC-DAD	Tap water and bottled drinking water	NA	0.04–0.67	119
OCs (3)	MIL-100(Fe)	3D-printed stirrer-HPLC-DAD	Biodigester and wastewater samples	92–109	0.3–1.7	70

(*Continued*)

Table 1 (*Continued*)

Analytes (number)	Sorbent	Method	Sample	Recoveries (%)	LOD (μg L^{-1})	Reference
Disinfection by-products (6)	HLB	Drone-TFME-portable-GC-MS	Hot tub water	NA	5	72
Neonicotinoids (5)	MIL53(Al)@paper	SPE-HPLC-DAD	Tap water, spring water, river water, marsh water, and irrigation water	86–114	1–1.6	16
PFAS (6)	MWCNTs@Fe_3O_4@C	m-SPE-UHPLC–MS/MS	Tap water, barreled water, and melted snow	71–104	(3–9)·10^5	120
PFAS (4)	HLB-WAX/PAN	SPME-LC–MS/MS	Tap water, lake water, bottled water, and river water	70–130	0.001–0.0025	78
PFAS (15)	HLB-WAX/PAN	SPME-LC–MS/MS	Seawater, melted snow, and human plasma	70–130	0.0025–0.01	77
PFAS (13)	C18 and aminopropyl silica	µSPE-LC-MS/MS	Surface water	86–111	2.9·10^4	75
PFAS (53)	WAX	SPE-LC-MS/MS	River water, wetland water, lake water, drinking water, and wastewater	20–138	0.0028–0.018	121
Microplastics (6)	Hydrophobic iron NPs	m-SPE-stereomicroscope	Seawater, freshwater, and sediments	78–93	NA	122
OCs and ICs (29)	HLB/PDMS and PPy-CO_2	UA-DSPE-ICP-MS or TFME-GC-MS	Produced water	90	NA	123

Abbreviations: APFs, additives, plasticizers, and fibers; ATR, attenuated total reflectance; AuNPs, gold nanoparticles; BTEX, benzene, toluene, ethylbenzene, and xylenes; CA-SPME, cooling-assisted solid-phase microextraction; CNF-TMHM, cellulose nanofibers coated with 2-(2-(di(thiophen-2-yl)methylene)hydrazineyl)-4-methylphenol; COF, covalent organic framework; DART, direct analysis in real time; DES, deep eutectic solvent; DSPE, dispersive solid-phase extraction; D-µSPE; dispersive micro solid-phase extraction; DVB/Car/PDMS, divinylbenzene/carboxen/polydimethylsiloxane; FAAS, flame atomic absorption spectrometry; GC, gas chromatography; GC-FID, GC with flame ionization detector; GO-APTES, graphene oxide/3-aminopropyltriethoxysilane; HLB, hydrophilic–lipophilic balance; HLB-WAX/PAN, hydrophilic-lipophilic balance–weak anion-exchange/polyacrylonitrile; HPLC, high-performance liquid chromatography; HRMS, high-resolution mass spectrometry; HS-SPME, headspace-assisted SPME; ICs, inorganic compounds; ICP, inductively coupled plasma; ITEX, dynamic in-tube extraction; LLE, liquid–liquid extraction; LOD, limit of detection; LPE, liquid-phase extraction; LPE-HR-CS-GFMAS, liquid-phase extraction high resolution-continuum source-graphite furnace molecular absorption spectrometry; MCM-41, Mobil Composition of Matter No. 41; MEPS, microextraction by packed sorbent; MIP, molecularly imprinted polymer; MOF, metal-organic framework; MS, mass spectrometry; MSPD, matrix solid-phase dispersion; mSPE, magnetic solid-phase extraction; MWCNTs, multiwalled carbon nanotubes; NA, not applicable; NiCr@NiCr LDHs-SO_4 NSAs, NiCr LDHs nanosheet arrays with intercalated sulfate ions on nichrome; NPs, nanoparticles; NSAIDs, nonsteroidal anti-inflammatory drugs; NTD, needle trap device; OCs, organic compounds; PAHs, polycyclic aromatic hydrocarbons; PAN, polyacrylonitrile; PANI-ES, polyaniline emeraldine salt; PBDEs, polybrominated diphenyl ethers; PCPs, personal care products; PDA/DAD, photo-diode array detector; PDMS, polydimethylsiloxane; PFAS, perfluoroalkyl and polyfluoroalkyl substances; PLE, pressurized liquid extraction; PM, particulate matter; PPy-CO_2, poly(pyrrole-1-carboxylic acid); PS, paper spray; SFE, supercritical fluid extraction; SIA, sequential injection analysis; SMPs, airborne small microplastics; SPE, solid-phase extraction; SPME, solid-phase microextraction; SS-LPME, switchable solvent-based liquid-phase microextraction; TED-GC–MS, thermal extraction-desorption GC–MS; TFME, thin-film microextraction; THF, tetrahydrofuran; TMC-BD-COF, covalent-organic framework based on trimesoyl chloride and benzidine; TOF, time-of-flight; UA-PR, ultrasound-assisted pressure-regulated; UE, ultrasonic extraction; UPLC, ultra-performance liquid chromatography; UV, ultraviolet; VA-DLLME-µSPE, vortex-assisted dispersive liquid–liquid micro solid-phase extraction; VALLME, vortex-assisted liquid–liquid microextraction; Vis, visible; VOCs, volatile organic compounds; µEME, nonsupported microelectromembrane extraction; µSEP, microplastic separator (hydrophobicity-water/air-based enrichment cell); µSPE, micro solid-phase extraction.

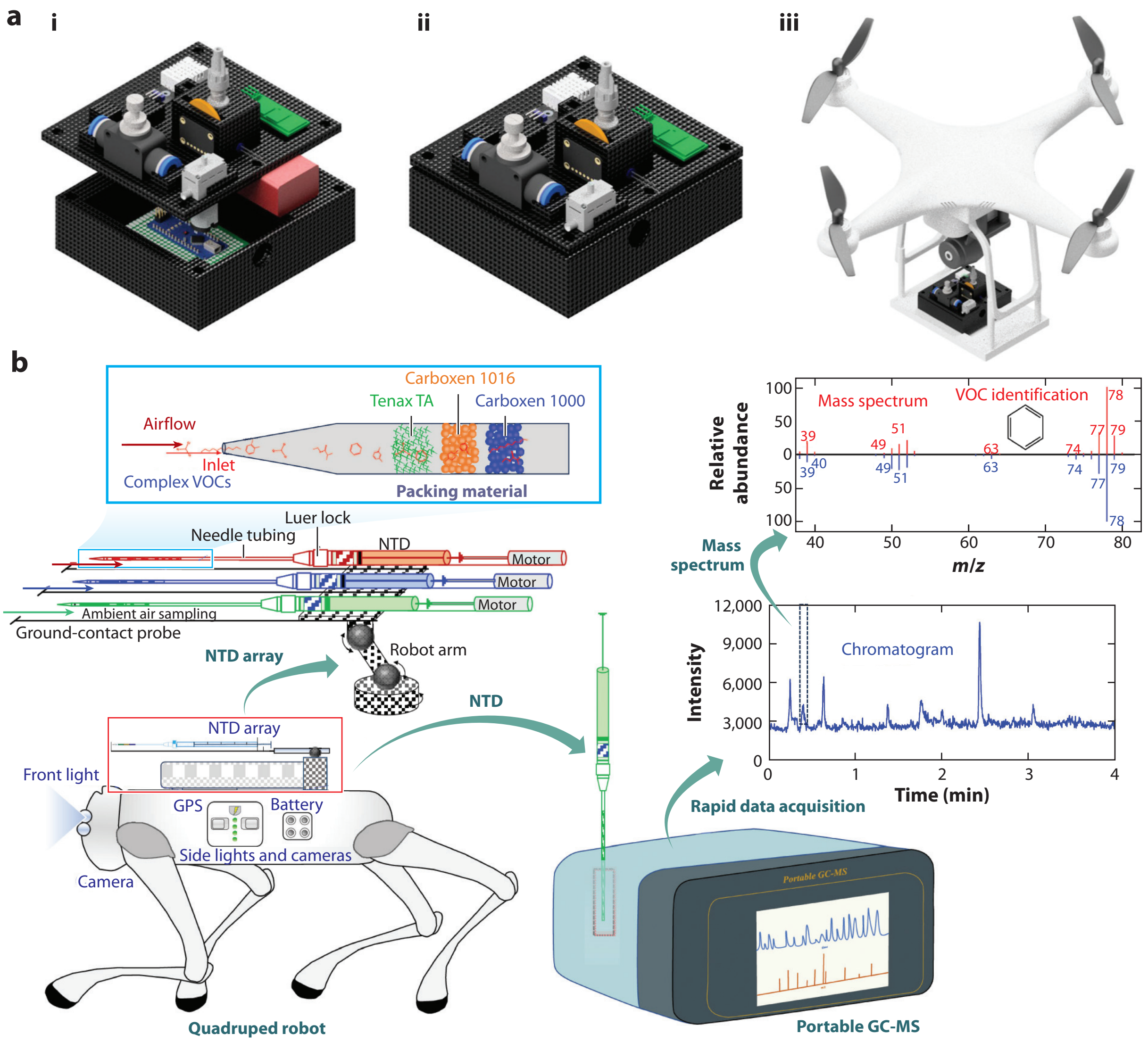

Figure 3

(*a*) 3D model of a lab-on-a-drone platform for air quality monitoring using a 3D-printed micropump: (*i*) unmounted sampler, (*ii*) mounted sampler, and (*iii*) complete lab-on-a-drone setup. (*b*) Robot-MS system for on-site detection of hazardous VOCs. Panel *a* adapted with permission from Reference 35; copyright 2023 American Chemical Society. Panel *b* adapted with permission from Reference 37; copyright 2024 American Chemical Society. Abbreviations: GC-MS, gas chromatography-mass spectrometry; GPS, global positioning system; *m/z*, mass-to-charge; NTD, needle trap device; VOC, volatile organic compound.

Another good option in air sampling is the use of needle trap devices (NTDs), which consist of a stainless steel needle packed with a sorbent material, through which fluid can be actively drawn in and out by a gas-tight syringe or pump, or into which analytes can be introduced passively by diffusion (38) (**Figure 1**). The advantages of NTDs include their portability, minimal sample preparation, and the ability to provide rapid and accurate results. Potential sorbent saturation is the most important challenge of this approach. To overcome this problem, Khoubi et al. (39) packed the needle device with an imine-based covalent-organic framework [trimesoyl chloride-benzidine-COF (TMC-BD-COF)] able to absorb a high quantity of polycyclic aromatic hydrocarbons (PAHs) owing to its large specific surface area. This device, coupled with a gas chromatography–flame ionization detector (GC-FID), obtained good figures of merit, including recoveries of 109–120%, limits of detection (LODs) as low as 0.021–0.027 μg L^{-1}, and an estimated precision of 8.51–10.34%.

Special attention should be given to the combination of robotics and NTDs for hazardous VOC sampling for use in inaccessible environments and situations that pose health risks. Liu et al. (37) examined 83 typical hazardous VOCs with a robot sampler coupled with an NTD array (**Figure 3*b***). The method was tested for on-site detection of emissions in a fire field with using a portable gas chromatography–mass spectrometry (GC-MS) device. The presented robot-MS system achieved low LODs (sub-ng mL^{-1} levels), good reproducibility [relative standard deviation (RSD) <20%, $n = 6$], excellent quantitative accuracy ($R^2 > 0.99$), and good detection speeds (within minutes). However, it is important to note that the system encounters significant challenges when handling nonvolatile analytes or when operating in rainy or snowy weather.

A third option for gas sampling commonly seen in the literature is SPME (**Figure 1**). This innovative solvent-free sampling method utilizes a fiber (or another format such as a thin film, blade, arrow, or magnet) coated with an extractive phase to selectively capture and concentrate volatile and semivolatile organic compounds from the air. SPME offers several advantages, including simplicity, rapid sample preparation, and the ability to preconcentrate trace levels of analytes. Despite its benefits, SPME is not free from drawbacks, such as the need for precise control of extraction conditions and potential issues with fiber saturation for highly concentrated gaseous samples. Continued advances in SPME technology are essential for enhancing its application in environmental gas analysis. One example by Chen et al. (40) described on-site identification and spatial distribution of air pollutants from combustion areas using a drone-based SPME coupled with a portable GC-MS instrument. Good analytical performance, in terms of sensitivity, reproducibility, and linear dynamic response, was achieved. Compared to coupling SPME with a benchtop instrument, drone-based SPME sampling and subsequent analysis with a portable GC-MS offers miniaturization, straightforward maintenance, and easy operation.

2.2. Solids and Semisolids: Matrix Solid-Phase Dispersion and Solid-Phase Microextraction

The analysis of solid and semisolid environmental samples presents a complex but essential field of study. Analyzing solid samples allows for the detection of persistent pollutants that might be undetectable in water or air as a result of dilution or dispersion. The main types of solid and semisolid samples comprise soil, sediments, and particulate matter from marine and river systems. Although some noninvasive approaches have been reported (41), the heterogeneity of these samples can lead to inconsistent results, as contaminants may be unevenly distributed. For this reason, homogenization is usually conducted to ensure representativeness, although extraction of a large amount of sample is another option. In this context, the physical properties of these samples, such as varying moisture content and particle size, can complicate sampling procedures and require specialized equipment that is usually not portable, increasing the risk of contamination during sampling and transport. Other labor-intensive and time-consuming sample preparation processes may be needed, such as heating, grinding, and sieving, which can impede direct and efficient analysis (42). On the positive side, solid samples require less preservation and can be stored for extended periods without significant degradation. The ability to perform in-depth, localized analysis on solid matrices also supports detailed spatial mapping of contamination, aiding in targeted remediation efforts.

Traditional sample preparation techniques for soil analysis, such as Soxhlet extraction, digestion, or calcination, are preferred by regulatory agencies for the determination of illicit substances and trace contaminants. However, these techniques often require large sample sizes, generate significant amounts of toxic waste, and involve complex logistics. To address these drawbacks, new methodologies such as microextraction techniques (SPME, LPME), matrix solid-phase dispersion (MSPD), and others have been developed. These innovative methods reduce sample size, minimize waste, and enhance efficiency and sensitivity, better aligning with both operational needs and environmental concerns.

One of the most prominent sample preparation techniques for solid environmental samples is MSPD, which can simultaneously extract and clean up the sample. In this process, the sample is broken down in the presence of solid support, leading to complete disruption of the sample and interactions between the sample matrix and the sorbent. The disruption creates a new phase that is then transferred to a column for the elution of the analytes (43, 44). Soares et al. (45) developed a quick and cost-effective multiresidue analysis of 59 analytes, including pharmaceuticals, personal care products, and antifouling booster biocides in marine sediments from Brazil utilizing vortex-assisted MSPD coupled with liquid chromatography–tandem mass spectrometry (LC-MS/MS). The validated method achieved a low LOD down to 0.13 ng g^{-1}, good linearity, satisfactory accuracy (60–140%), and good precision (RSD $< 20\%$), while only requiring 2 g of sample and 5 mL of methanol.

Other important techniques for analyzing volatile and semivolatile species in solid samples are based on different modalities of SPME, including ultrasound-assisted, heating-assisted, cooling-assisted (CA-), and vacuum-assisted (VA-) SPME or combinations of these (46–48). Generally, the main advantages of these techniques, compared to conventional methods, are their shorter extraction time and increased efficiency. Furthermore, additional sample treatment steps, such as digestion and filtering, can be avoided. Maleki et al. (49) reported a portable in-syringe vacuum-assisted headspace (VA-HS-)-SPME method coupled to a GC-FID for simultaneous analysis of volatile and semivolatile compounds in soil. Interestingly, the authors developed a laboratory-made fiber coating made from a hybrid of COFs and MOFs to increase the sorbent affinity for the analytes. Furthermore, the use of external pumps to create the vacuum was avoided, which made the process smooth and straightforward.

Zhakupbekova et al. (50) isolated trace transformation products of rocket fuel, such as unsymmetrical dimethylhydrazine, in sand samples using VA-HS-SPME (without the addition of water as a dispersing media) followed by GC-MS. By only using 1 g of sample and setting 20 s as the air evacuation time at 23°C, they achieved lower LODs, better accuracy, and better precision at a similar or lower cost of sample preparation compared to existing HS-SPME methods.

Finally, it is worth highlighting the analysis of microplastics in solid environmental samples (51, 52). Microplastics are small plastic particles ($\leq$5 mm) that are currently one of the most concerning environmental problems. Their indiscriminate release via direct introduction into the environment through products such as cosmetics and cleaning products, or by the degradation of large plastic debris, has increased their global occurrence. Concerns about their low biodegradability and negative effects on ecosystems have prompted the development of different methods to determine, characterize and, if possible, remediate their presence in the environment.

Direct and noninvasive methods have been reported in the literature, especially those based on rapid extraction using density or affinity, followed by analysis with Fourier-transform infrared (FTIR) spectroscopy and microscopy (53–56). Despite the simplicity of these approaches, they are useful for fast detection, characterization, and remediation. Furthermore, the necessary equipment is not sophisticated, and untrained personnel can perform the analysis, although the information can be biased because the techniques only conduct a surface analysis.

In contrast to direct and noninvasive methods, destructive techniques including pyrolysis provide a wealth of information about the total composition of the sample, possible additives introduced in plastic manufacturing, organic pollutants adsorbed into the surface, and more (57). Combining modern approaches based on CA-SPME with a pyrolysis step for the analysis of microplastics offers the unique advantage of integrating the thermal decomposition of micro- and nanoplastics and the enrichment of signature compounds into one step. As an example, Xu et al. (58) reported a quantitative determination of poly(methylmethacrylate) micro- and nanoplastics in soil samples using CA-SPME and GC-MS. They were able to detect amounts as low as 0.28 μg using pyrolysis at 350°C with an estimated incubation time of 13 min without any further pretreatment or need for use of organic solvents.

Finally, current research being performed by Ren et al. (59–61) devoted to the separation of microplastics from heterogeneous solid mixtures should be highlighted. This group has been working with magnetic levitation to detect not only organic pollutants on microplastics, but also sequential separation of microplastics by density and size. Experiments are usually carried out in flow cells sandwiched between two ring magnets and connected to programmable pumps for flow control in the presence of $MnCl_2$ (a paramagnetic salt). Briefly, the first separation dimension relies on progressively increasing the Mn^{2+} concentration, which allows for the magnetic levitation of microplastics with specific densities. In the second dimension, flow rate gradients are increased while keeping Mn^{2+} concentrations constant (**Figure 4*a***). This step separates the magnetically levitating microplastics by their particle sizes. Consequently, microplastics are collected based on their increasing density, and within each density, the particles are fractionated from smaller to larger sizes (59, 60). To analyze contaminants adsorbed onto microplastics, position-dependent microplastic trapping in a biphasic medium was applied. This procedure involves a paramagnetic aqueous donor phase containing mixed microplastics and a diamagnetic organic acceptor phase. By adjusting the height of the sample container within a magnetic field, selective density-dependent trapping of microplastics is achieved (**Figure 4*b***). Simultaneously, organic pollutants adsorbed onto the microplastics are desorbed into the organic acceptor phase, which can be

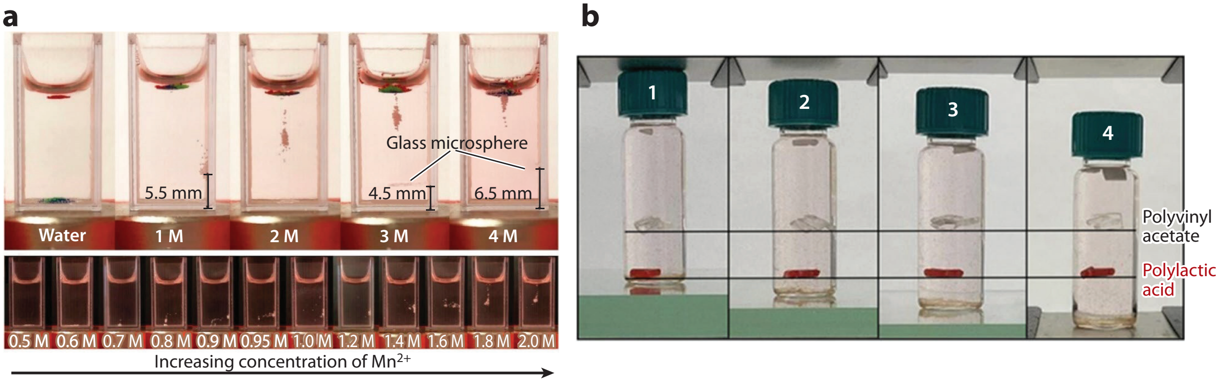

Figure 4

Separation of microplastics using magnetic levitation. (*a*) Effect of Mn^{2+} concentration microsphere levitation height (microspheres were left for 1 min to reach equilibrium positions in all instances). (*b*) Plastic particles of polyvinyl acetate (*translucent*; 1.19 g cm^{-3}) and polylactic acid particles (*red*; 1.24 g cm^{-3}) containing 2 M Mn^{2+} in solution at different heights. Height of the vial relative to the bottom magnet: (vial 1) 9 mm, (vial 2) 6 mm, (vial 3) 3 mm, and (vial 4) 0 mm. To adjust the vial height, 1-mm-thick glass slides were used. Panel *a* adapted with permission from Reference 60; copyright 2022 American Chemical Society. Panel *b* adapted with permission from Reference 61; copyright 2022 American Chemical Society.

easily separated and transferred for organic pollutant determination using high-performance liquid chromatography (HPLC) (61).

2.3. Aqueous Samples: Solid-Phase Extraction and Solid-Phase Microextraction

Analysis of aqueous environmental samples is essential for monitoring water quality and assessing the presence of contaminants in various water bodies, such as rivers, lakes, seas, and groundwater. The main challenges in water analysis include the diversity of potential pollutants, ranging from organic chemicals to heavy metals, with concentrations from low ng L^{-1} to g L^{-1}. Furthermore, environmental sample matrices can be highly complex, and their composition can interfere with separation and detection methods, leading to significant matrix effects, especially with direct injection techniques such as dilute-and-shoot (62). Despite these challenges, low LODs can be achieved by using large volumes of samples, when available. Recent advances in sample treatment for aqueous environmental samples have significantly enhanced the accuracy and efficiency of pollutant detection and analysis, particularly compared to traditional methods such as SPE and liquid–liquid extraction (LLE), which often require large volumes of reagents and generate considerable waste. Innovative strategies to miniaturize the size of devices and volume of sorbent used led to the development of micro-SPE (not to be confused with SPME) and liquid–liquid microextraction (LLME), which not only significantly reduce the environmental impact but also increase analytical efficiency. With the goal of reducing the environmental impact of sample preparation techniques, researchers have proposed natural materials (e.g., chitosan, cellulose, lignin, natural deep eutectic solvents) as SPE sorbents (63) or liquid extractive phases (64). Highly tailored materials including MIPs, carbon-based MOFs, and layered double hydroxides (LDHs) have also been reported (65–67). Although synthesizing these materials can be tedious, the resulting devices are affordable and simple to use.

Lab-in-a-bottle is an open-source technology that integrates on-site sampling and preconcentration techniques (68–70). Vargas-Muñoz et al. (70) developed a lab-in-a-bottle sampling platform incorporating 3D-printed paddle stirrers coated with an MIL-100(Fe) MOF for in situ extraction of phenolic pollutants in different wastewater samples (**Figure 5*a***). The presence of the MOF improved extraction efficiency by favoring hydrophobic interactions. After optimization, LODs ranged from 0.3 to 1.7 $\mu g\ L^{-1}$, with precisions of 4.5–6.8% RSD and accuracy in real samples of 92–109%. The field-deployable platform combines sampling and analyte extraction/preconcentration into a single step, reducing analyst work time, preserving sample integrity, and ensuring representative results. The portable stirrer, costing approximately US$100, is both inexpensive and easy to replicate using commercially available materials. It is user-friendly, with stirrer paddles that can be easily installed, removed, and replaced after their initial use.

SPME has been refined to offer higher selectivity and sensitivity, enabling the preconcentration of compounds at trace levels while minimizing reagent use and waste. Vanguard applications of this technique are the result of the availability of new portable and miniaturized technologies, streamlining the workflow. Grandy et al. (71) deployed a submarine SPME sampler to characterize biogeochemical profiles among various deep sea hydrothermal vents (**Figure 5*b***). After sampling, analysis was carried out by LC-MS/MS, identifying labile dissolved organic sulfur compounds, which are biomarkers of anammox bacteria, fungi, and lower animals. This report encouraged the use of SPME for sampling in inaccessible locations and for understanding the ecology of the deep sea and its biogeochemical cycles. Grandy et al. (72) also reported the on-site screening of environmental pollutants in water systems using drone-based thin-film microextraction (TFME) coupled with a portable GC-MS instrument. The drone was deployed above the surface of the water to be sampled, and TFME devices were released into the water for sampling. The thin films

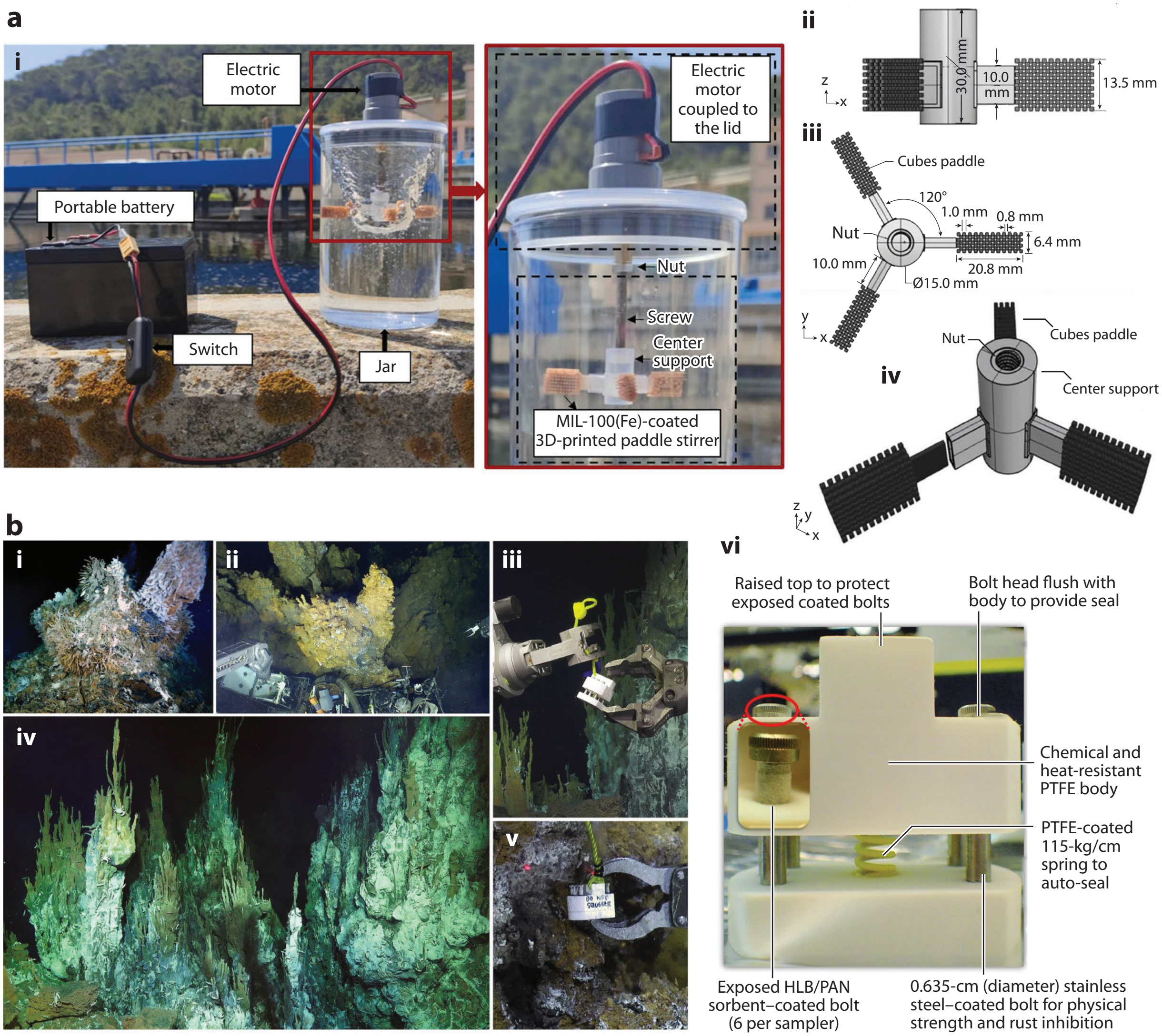

Figure 5

(*a*) Lab in a bottle. (*i*) Portable stirring device with MIL-100(Fe)-coated paddles for in situ surface water sampling. (*ii*) Front view, (*iii*) top view, and (*iv*) 3D view of the portable stirring device. (*b*) Use of SPME for submarine in situ sampling. (*i*) El Gordo chimney with tubeworms, Axial Seamount. (*ii*) Ultranochichi chimney, Urashima site, downward view of iron-oxide covered chimney. (*iii*) SPME sampler manipulation at the Alba vent. (*iv*) Overview of the Alba chimney complex, with shrimp and crabs. (*v*) Triggering of the SPME sampler at Ultranochichi. (*vi*) Details of the internal and external components of a coated bolt sampler used for sampling. Panel *a* adapted with permission from Reference 70; copyright 2023 Elsevier. Panel *b* adapted with permission from Reference 71; copyright 2020 Springer Nature. Abbreviations: 3D, three-dimensional; HLB/PAN, hydrophilic-lipophilic balanced particles embedded in polyacrylonitrile; MIL-101(Fe), Matériaux de l'Institut Lavoisier containing Fe as metal center; PTFE, polytetrafluoroethylene; SPME, solid-phase microextraction.

were coated with hydrophilic-lipophilic-balanced particles embedded in poly(dimethylsiloxane) (HLB/PDMS). Only 10 min were needed to preconcentrate organic pollutants from hot tub water.

Finally, we highlight PFAS analysis, given the rising global awareness of the persistent presence of these compounds in aquatic systems, with SPE being the gold standard sampling technique used for their capture (73, 74). Although analysis of PFAS in water has advanced with techniques such as SPME and DLLME using selective sorbents, several challenges remain, driven by the complex diversity and inert behavior of these substances. In addition, PFAS analysis in water requires ultratrace-level detection, requiring expensive, highly sensitive instruments and specialized training. Another significant issue is the potential for cross-contamination of samples, because PFAS are widespread in laboratory materials and equipment, which demands strict contamination control measures. Given that many PFAS include ionizable moieties in their structures, weak anion exchange (WAX) solid sorbents have proven useful in the isolation of legacy and emerging PFAS, as seen in several studies (75).

Olomukoro and colleagues (76–78) have applied ion exchange sorbents to SPME to isolate PFAS in complex environmental samples. In particular, an SPME device coated with HLB that included weak anion exchange moieties embedded in polyacrylonitrile (HLB-WAX/PAN) was developed and was able to preconcentrate PFAS at low part-per-trillion levels (78). The authors extended the work to 15 analytes, including short carbon chain PFAS, which are particularly challenging to extract (77). The SPME sample treatment was coupled to the LC-MS/MS device to achieve the limits of quantification (LOQs) recommended by regulatory agencies [e.g., the EPA currently regulates the concentration of several PFAS not to exceed 4 ng L^{-1} in drinking water (79)]. To speed up the screening of PFAS, the authors directly coupled SPME to the mass spectrometer, avoiding the analytical separation step and enabling a solvent-free method (76). This is possible due to the high preconcentration factor that can be achieved by SPME as well as the analytical power of MS instrumentation. Remarkably, this approach achieved a linear dynamic range from 10 to 5,000 ng L^{-1} and LOQs of 10, 25, and 50 ng L^{-1} with an analysis time of less than 20 s per sample.

3. CONCLUSION AND FUTURE PERSPECTIVES

In conclusion, environmental analysis has made significant strides over the past five years, driven by the urgent need to understand and mitigate the impacts of pollutants on ecosystems and human health. The development of robust and precise analytical methods has been instrumental in detecting and monitoring a wide range of contaminants. Sample treatment is a critical aspect of obtaining robust and reliable results in environmental analysis.

In gaseous sample treatment, capturing and concentrating trace pollutants such as VOCs and persistent organic pollutants remain significant challenges. Traditional methods such as adsorption onto solid sorbents and subsequent thermal desorption are effective but can be labor intensive and require sophisticated equipment. Furthermore, ensuring the stability and integrity of analytes during sampling and storage is crucial to obtaining accurate results. Advances in passive sampling techniques and the development of novel sorbent materials show promise in addressing these challenges by offering higher enrichment factors and selectivity.

For solid samples, such as soil and sediments, the heterogeneity of the matrix poses a significant challenge for consistent and representative sample treatment. Traditional extraction methods such as Soxhlet extraction and microwave-assisted extraction can be time consuming and require substantial solvent use. Additionally, the presence of organic matter and other interferents can complicate the extraction and analysis of target contaminants. Newer approaches, including SPME and magnetic-assisted flotation techniques, aim to enhance efficiency and reduce environmental impact but require further optimization and validation for routine use.

For aqueous samples, the diverse range of contaminants, often present at trace levels, necessitates a high preconcentration factor that improves the sensitivity of the analytical method and selective extraction strategies. Traditional techniques such as SPE and LLE require large volumes of solvents and generate substantial waste, posing environmental and economic concerns. Additionally, matrix effects from varied water sources—such as wastewater, groundwater, and seawater—can complicate analyte recovery and accuracy, necessitating more robust and adaptable treatment methods. Emerging techniques such as SPME and DLLME offer improvements by minimizing reagent use and reducing waste, but they still face challenges in scalability and standardization.

Future developments in sample treatment for environmental analysis will focus on enhancing efficiency, sensitivity, and sustainability. The integration of automated and miniaturized systems aims to streamline workflows and reduce human error. The use of advanced materials and nanocomposites continues to evolve, offering improved selectivity and sensitivity. Additionally, green chemistry principles are driving the development of solvent-free and low-waste methods, aligning with environmental sustainability goals. The combination of these innovations with robust standardization protocols will be crucial to overcome current challenges, advance the field of sample treatment for environmental analysis, and achieve substantial progress in safeguarding environmental health and promoting sustainable development initiatives worldwide.

DISCLOSURE STATEMENT

The authors are not aware of any affiliations, memberships, funding, or financial holdings that might be perceived as affecting the objectivity of this review.

ACKNOWLEDGMENTS

The authors thank the University at Buffalo (UB), The State University of New York and the UB Research Foundation for funding.

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