



Sample preparation and extraction methods for pesticides in aquatic environments: A review

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ABSTRACT

Pesticides are the most hazardous environmental pollutants due to their stability, mobility and long-term effects on living organisms. Their existence in sources of drinking water, essential to life, is a specific danger to living organisms. In water, these compounds may metamorphose to produce substances with even greater toxicity. Pesticide levels in diverse environments are generally below the limits of detection for many analytical instruments, so it is essential to isolate and then enrich pesticides from the complex matrix to decrease the interference effects in order to obtain a reliable signal. The main objective of this review is to evaluate the new sample preparation and extraction methods for the analysis of pesticides in various water samples.

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1. Introduction

The food and agriculture organization (FAO) defines the pesticides as: "substances designed for interdicting, spoiling, enticing, repulsing or controlling any pest, including undesirable species of plants or animals, during the generation, storage, transport, distribution and processing of food, agricultural productions or animal feeds, for use as a plant growth corrector, defoliant, drying agent, fruit thinning agent or growing suppressor and substances applied to crops either before or after transport."

Pesticides have a large variety of chemical structures, with various action mechanisms and usages. Presently, over 800 active components, belonging to more than 100 classes, are available in a broad range of commercially accessible products [1]. They can be classified based on varying properties such as toxicity, usage, environmental stability, and chemical structures. In general, three major groups of pesticide classifications are the most common: (1) herbicides, substances used to manage unwanted plant growth or to kill weeds; (2) insecticides, used to destroy infesting insects; and (3) fungicides, used to control the multiplication of fungi. It should be considered that some classes might fall into more than one

major group. The utmost outspread groups of pesticides are organochlorine, organophosphorus and organonitrogen [2].

Once pesticides get into the aquatic environment, they substantially lower the quality of the surface and ground waters, which constitute sources of drinking water for a large part of the world's population. Fig. 1 illustrates the main transport pathways and the circulation of pesticides in the aquatic environment. There are many sources of pesticide discharge into the aquatic environment. They can come from both area sources (atmospheric precipitation or farmland) and point sources (effluents of diverse origins or hazardous-waste-disposal sites). They can also be transported over long distances through the air [3].

Nowadays, human beings come in contact with pesticides more often than any other pollutants. Due to the high toxicity of pesticides and their metabolites at trace levels, the monitoring and detection of them in water sources with complex matrices require sample pre-treatment and sampling steps before chemical analysis.

Sample preparation and pre-concentration methods could concentrate the pesticides, significantly lowering the limit of detection (LOD) when using conventional analytical instruments for pesticide chemical analysis. Therefore, there is an essential need to integrate sample preparation or treatment into analytical approaches for ultra-trace quantitation of pesticides in complex samples. Herein, recent tendencies are (1) automation through the coupling of sample preparation units and detection systems, (2) application of advanced sorbents, and (3) application of greener approaches, such as solvent reduced techniques. New methods and

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Abbreviations

AF-MCF	Amino modified mesostructured cellular foam	HPGF	Hierarchical pores graphene framework
ATP/Fe ₃ O ₄ /PANI	Attapulgite/Fe ₃ O ₄ /polyaniline	HPLC	High-performance liquid chromatography
CAP	Covalent aromatic polymer	ILs	Ionic liquids
CAR	Carboxen	IMS	Ion mobility spectrometry
CD	Cyclodextrin	IT-SPME	In tube solid-phase microextraction
CDD	Cyclododecane	KHA	Potassium substituted hydroxyapatite
CE	Capillary electrophoresis	LLE	Liquid-liquid extraction
Ch-MO NPs	Chitosan-metal oxide nanoparticles	LMA-HEDA	Lauryl methacrylate-co-1, 6-hexanediol ethoxylate diacrylate
Ch-Si MNP	Chitosan-siloxane magnetic nanoparticles	LOD	Limit of detection
CLF	Chloroform	LOQ	Limit of quantification
CMCD	Carboxymethyl-cyclodextrin	LPME	Liquid-phase microextraction
CNPrTEOS	Cyano propyl triethoxy silane	LR	Linear range
CNT	Carbon nanotube	LTTM	Low transition temperature mixture
Co/Cr (NO ₃ ⁻)-LDH	Cobalt/chromium-layered double hydroxide	MAED	Methacrylic acid-co-ethylene dimethacrylate
COF	Covalent organic framework	MAIDB	1-methyl-3-alkyl imidazolium bis (trifluoromethylsulfonyl) imide copolymerized with divinylbenzene
CoFe ₂ O ₄ -PGE	Cobalt ferrite embedded porous graphitic carbon	MCB	Monochlorobenzene
COOH-PAN NFsM	Carboxyl modified polyacrylonitrile nanofibers mat	MCM	Mobil composition of matter
Co/HPC	Co-doped hierarchically porous carbon	MHMS-MCNP	Mixed Hemi micelle SDS-coated magnetic chitosan nanoparticles
CTAB	Cetyltrimethylammonium ammonium bromide	MIR	Molecular imprinted resin
CTC	Carbon tetrachloride	MGO	Magnetic graphene oxide
DAD	Diode array detector	MI-ILMM	Molecularly imprinted ionic liquid magnetic microsphere
DART-MS	Direct analysis in real time mass spectrometry	MIL	Material of Institute Lavoisier
DBE	Dibromoethane	MIP	Molecular imprinted polymer
DCE	Dichloroethane	MMC	Monolithic microextraction column
DCM	Dichloromethane	MMF	Multiple monolithic fiber
DESS	Deep eutectic solvents	MMP/ZIF-8	Magnetic zinc-based zeolitic imidazolate framework
DLLME	Dispersive liquid-liquid microextraction	MMT	Montmorillonite
DLLME-SFO	Dispersive liquid-liquid microextraction with solidification of floating organic drop	MMT/PPy/N6	Montmorillonite doped polypyrrole/nylon-6
dSPE	Dispersive solid-phase extraction	M-MWCNTs	Magnetic multi-wall carbon nanotubes
DVB	Divinylbenzene	mMFP	Mesoporous melamine-formaldehyde polymer
EA-DLLME-CFO	Effervescence assisted dispersive liquid-liquid microextraction based on cohesive floating organic drop	MOF	Metal-organic framework
ECD	Electron capture detector	MS	Mass spectrometry
EDTA	Ethylene diamine tetraacetic acid	MSPE	Magnetic solid-phase extraction
FAO	Food and agriculture organization	MWCNT	Multi-walled carbon nanotube
Fe ₃ C/MnO/GC	Iron carbide/manganese oxide/graphitic carbon	NMs	Nanomaterials
Fe ₃ O ₄ @G-TEOS-MTMOS	Graphene based tetraethoxysilane-methyltrimethoxysilan sol-gel magnetic nanocomposite	NPC	Nanoporous carbons
FID	Flame ionization detector	NPD	Nitrogen phosphorus detector
FPD	Flame photometric detector	NPs	Nanoparticles
G	Graphene	OCP	Organochlorine pesticide
GC	Gas chromatography	ODS	Octadecyl silane
GO	Graphene oxide	OPP	Organophosphorus pesticide
GO-Chm	Magnetic chitosan graphene oxide	OTP	Organotin pesticide
GO@ IL@NHC-Cu	N-heterocyclic carbene copper complex supported on ionic liquid-modified graphene oxide	PA	Polyacrylate
GOQD	Graphene oxide quantum dot	PCM	Porous carbon materials
HBA	Hydrogen bond acceptor	PCP-C	Phthalocyanine containing polymer derived carbon
HBD	Hydrogen bond donor	PDA	Photodiode array
HBPE	Hyperbranched polyester	PDA	Polydopamine
HF-LPME	Hollow-fiber liquid-phase microextraction	PDMS	Poly dimethyl siloxane
HIPEs	High-internal-phase emulsions	PEA	Phenyl ethyl amine
HLB	Hydrophilic lipophilic-balanced	PEG-PPG-PEG	Poly (ethylene glycol)-poly (propylene glycol)-poly (ethylene glycol)
HPAMAM	Hyperbranched polyamidoamine	PMO	Periodic mesoporous organosilica
HP-β-CD	2-hydroxypropyl-β-cyclodextrin	POM-IL	Polyoxometalate-based ionic liquid
HPGA	Hierarchical pores graphene aerogel	PP-CMPs	Polyphenylene-based conjugated microporous polymers
		PPDA-co-Th	p-Phenylenediamine-coated-thiophene
		PPDA	Poly (p-phenylenediamine)

PVA	Poly (vinyl alcohol)	SPME	Solid-phase microextraction
P4VP	Poly 4-vinylpyrrolidone	SUPRAs	Supramolecular solvents
QuEChERS	Quick, easy, cheap, effective, rugged, and safe	TCE	Tetrachloroethylene
R	Recovery	TPN	Triazine-based polymeric network
RGO-ZnO	Reduced graphene oxide coated with ZnO	UiO	University of Oslo
RSD	Relative standard deviation	UV-VIS	Ultraviolet visible
SALLE	Salting-out liquid-liquid extraction	VWD	Variable wavelength detector
SBSE	Stir bar sorptive extraction	ZIFs	Zeolitic imidazolate frameworks
SDME	Single drop microextraction	ZMNIC	Zirconia/magnetite nanocomposite immobilized chitosan
SHLE	Salting-out homogeneous liquid-liquid extraction	ZnO/g-C ₃ N ₄	Zinc oxide hybridized with graphitic carbon nitride
S-MEKC	Sweeping-micellar electrokinetic chromatography	ZnS-AC	Zinc sulfide nanoparticles loaded on activated carbon
SMNM	Self-magnetic nanocomposite monolithic	β-CD/ATP	β-cyclodextrin/attapulgite composite
SPE	Solid-phase extraction		

techniques of sample preparation should not only be environmentally friendly; should they also be characterized by their simplicity of performance, as well as the low cost and time efficiency of the entire performance [4–6]. It is well known that adsorbents play an extremely important role in solid-phase sorption-based extraction methods because their adsorption performance towards target analytes directly affects the selectivity and sensitivity of the method. Nanomaterials (NMs) have been used as sorbents for the pre-concentration and determination of trace levels of pesticides from complex samples with low-volume. The selectivity and sensitivity of analytical techniques have been significantly improved by using NMs as sorbents because of their advantageous properties such as large surface area and specific affinity towards ultra-trace-level target analytes [6]. Currently, a wide variety of NMs like metallic and metal oxides, polymer-based nanocomposites, and silicon- and carbon-based NMs have been integrated into various sample preparation techniques [7].

In this review, pre-concentration and extraction methods of pesticides in environmental samples, with regard to sorbent type/extraction solvent, have been compiled since 2015 and discussed according to the most widely used extraction methods.

2. Extraction methods of pesticides

The extraction of pesticides prior to introduction to the instrument is an important step of the analysis. This process will decrease

the effect of interferences in measurement and simultaneously enrich the analyte concentration for analysis. There are several extraction methods ranging from traditional such as liquid-liquid extraction (LLE) and quick, easy, cheap, effective, rugged, and safe (QuEChERS) to more advanced techniques with minimal solvent usage. These techniques include solid-phase extraction (SPE), dispersive solid-phase extraction (dSPE), stir bar sorptive extraction (SBSE), magnetic solid-phase extraction (MSPE), solid-phase microextraction (SPME), liquid-phase microextraction (LPME) and dispersive liquid-liquid microextraction (DLLME). LLE uses large volumes of solvent that may further impose environmental issues, whereas new techniques such as SPME are solvent-less extraction techniques. Types of pre-concentration and extraction methods of pesticides along with the type of adsorbent/extraction solvent since 2015 have been summarized in Table 1.

2.1. Solid-phase extraction

Solid-phase extraction (SPE) was first introduced in the mid-the 1970s and became commercially available in 1978 [227]. SPE is presently one of the most widespread extraction methods for the pretreatment of environmental samples [8–13].

Compared to traditional LLE techniques, SPE is simple, comfortable, and easy to automate. In addition, SPE has several distinguished advantages, including (1) lower solvent volume in the extraction process than that of traditional LLE; (2) simple manipulations that make it possible for field treatment; (3) high enhancement factors proportional to the volume of water passed through the SPE cartridge. It is well known that adsorbents act significantly in the SPE method due to their adsorption performance to the pesticides and also its effects on the selectivity and sensitivity of the method [228]. Novel adsorbents have been constructed to raise the performance of solid-phase sorption-based extraction methods and satisfy the requirements of highly sensitive and selective green analytical chemistry. Many novel nanomaterials, such as, carbon nanotube (CNT) [91,142], multi-walled carbon nanotube (MWCNT) [12,31,66], graphene (G) [29,86,133] and graphene oxide (GO) [41, 59–61], metal-organic frameworks (MOFs) [30,51,52] and molecular imprinted polymers (MIPs) [11, 13, 14, 18–22], have been used as sorbents in the extraction. Table 1 shows a partial list of pesticides being extracted from environmental samples before analysis. Column 2 lists the most common adsorbents reported since 2015 including MOF, MIP, G, GO and MWCNTs.

Burakham et al. [27] announced automated modification and application of zeolite NaY as a sorbent for SPE. Zeolites are crystalline aluminosilicate of alkali and alkaline land which are combined with SiO₄ and AlO₄ in a framework skeleton [229]. Zeolite Y has large channels, great surface area, high cation exchange capacity, and Si/Al ratio [230]. Zeolites are impressive support

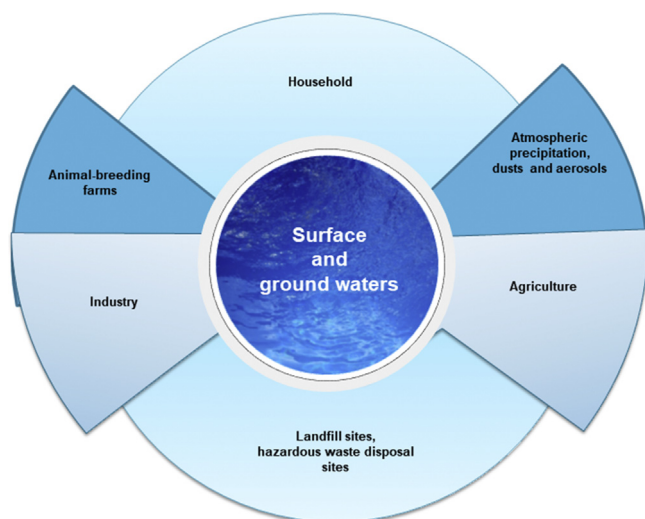


Fig. 1. Sources and fates of pesticides in the aquatic environment.

Table 1

Overview of extraction methods for the analysis of pesticides in environmental waters.

Extraction method	Adsorbent/extraction solvent	Matrix	Analyte	Analytical method	LOD (μg L ⁻¹)	LOQ (μg L ⁻¹)	LR (μg L ⁻¹)	R (%)	Ref.
SPE	MCM	Environmental water	OPPs, carbamates and triazoles	HPLC-MS	0.01	0.05	0.10–75.55	65–126	[8]
	Mesoporous silica-based on the sol-gel material CNPrTEOS	Environmental water	Polar and non-polar OPPs	HPLC-UV	0.072–0.091	0.24–0.30	0.8–100	80.1–108	[9]
	HPGA	Environmental water	Pyrethroids	GC-MS	0.012–0.11	–	0.2–50	65.7–105.9	[10]
	MIP	Environmental water	Triazines	HPLC-MS/MS	0.007–0.068	0.022–0.227	0.25–50	88–100	[11]
	MWCNTs	Environmental water	Carbamate insecticides	HPLC-MS	0.01–0.05	0.08–0.2	5–1000	92.2–103.9	[12]
	MIP	Environmental water	Chloroacetamide herbicides	HPLC-DAD	0.03–0.06	0.09–0.15	0.1–200	82.1–102.9	[13]
	HLB	Environmental water	Triazines	HPLC-UV	0.05–0.1	–	0.1–25	–	[14]
		Field water	Neonicotinoids, OP and OC insecticides	GC-MS	0.0021–0.0042	–	–	63–124	[15]
	COF	Environmental water, juice, fruit and vegetables	Pesticide	HPLC-UV	0.02–0.050	0.06–0.150	0.1–160.0	87–103.3	[16]
	ZMNIC	Water and juice	OPPs	GC-MS	0.001–0.004	0.003–0.012	0.01–500	91.9–104.4	[17]
	MIP	Maize, water, and soil	Triazine herbicides	HPLC-UV	0.0009–0.0012	–	0.1–16 μmol L ⁻¹	82–99	[18]
		River water	Imidazole fungicides	HPLC-UV	0.023–0.031	–	0.1–20	84.2–95.0	[19]
		Waste water and plant	OP insecticides	GC-FPD	294	980	0.9992	94–104	[20]
		Water and food	Carbendazim	HPLC-DAD	7	23	50–5000	84.32–99.14	[21]
		Environmental water	Atrazine	HPLC-DAD	0.20	0.60	–	83–89	[22]
	Oasis HLB	Water	Pesticides	HPLC-MS/MS and GC-MS/MS	–	0.01–0.1	LC-MS/MS: 3.5–600 GC-MS/MS: 5–50	LC-MS/MS: 51.9–144 GC-MS/MS: 45–134	[23]
	C18, PS2, Oasis HLB, AC2, and PLS–3	Water	Pesticides	HPLC-MS	0.008–0.08	–	–	More than 50%	[24]
	G-coated cotton fiber	Water	Pesticides	GC-MS	–	0.02–0.09	0.02–10	83–107	[25]
		Water	Pesticides	GC-MS/MS and HPLC-MS/MS	0.006	0.02	0.99	70–117.3	[26]
	CTAB modified zeolite NaY	Water	Pesticides	HPLC-UV	0.005–140	0.02–600	0.9963–0.9991	77–111	[27]
	Surfactant	Water	Pesticides	HPLC-MS/MS	–	0.027–0.090	0.1–40	83–93	[28]
	G- modified TiO ₂	Water	Carbamate pesticide	HPLC-UV	2.27–3.26	–	5–150	83.9–108.8	[29]
	MOF	Water	OCPs	GC-MS	0.0025–0.016	0.010–0.074	0.05–50	87.6–98.6	[30]
	MWCNTs	Surface water	Pesticides	HPLC-MS/MS	0.0003	0.0016–0.0452	0.99	100	[31]
	COOH-PAN NFsM	Surface water	Atrazine	HPLC-DAD	–0.00095	0.3	0.3–40	81.35–120.32	[32]
		River water and vegetable juices	Bio pesticides	HPLC-MS	0.09–0.12	3.73–16.51	5–500	88.6–95.8	[33]
	PLRP-s cartridge	Drinking, ground and surface water	Acidic and polar pesticides	HPLC-MS/MS	0.0012–0.018	Below 0.1	0.05–2	70–120	[34]
	β-CD polymer@Fe ₃ O ₄	Honey, tomato, and environmental water	Benzoylurea insecticides	HPLC-DAD	0.02–0.05	–	0.1–80.0	87.3–112.5	[35]
	C18	Surface waters	OCPs	GC-MS	–	0.00001–0.00037	–	82–117	[36]
		Environmental water	Herbicides	HPLC-MS/MS	–	–	–	100	[37]
		Surface water	OCPs	GC-MS/MS	0.00008	–	0.9981–0.9998	70.3–115.1	[38]
		River water	Pesticide	HPLC-UV	–0.00038	0.0095–0.0244	10–200	88–102	[39]
					0.0028–0.0073				
	Surface water	OCPs	GC-ECD	0.0003–0.0054	–	0.99	95–106	[40]	

dSPE	GO	Water and celery	Phenylurea herbicides	HPLC-DAD	0.01–0.02	0.03–0.08	0.05–40	86–112	[41]
	Oasis HLB	Drinking water	Neonicotinoid pesticides	HPLC-MS/MS	0.001–0.004	1–200	–	–	[42]
		Seawater	Triazine herbicides	HPLC-MS/MS	0.008–0.02	0.023–0.657	0.025–0.5	80.3–99.8	[43]
		Surface and groundwater	Pesticides	HPLC-MS/MS	0.001–0.005	0.003–0.01	–	Higher than 70	[44]
		Urban wastewater	Pesticides	HPLC-MS/MS	–	0.001–0.015	0.0011–1.6	75–115	[45]
		Surface water	Pesticides	GC x GC-MS	1.48	–	–	–	[46]
			Pesticides	HPLC-MS	–	0.0005–0.1	0.1–1000	35–89	[47]
		Environmental water	Neonicotinoids	HPLC-MS/MS	0.01119	0.00005–0.0005	0.00005–0.1	58.9–109.9	[48]
	Seawater	Pesticides	HPLC-MS/MS	0.00002–0.00371	0.00005–0.027	0.0000025–0.4	80–120	–	[49]
	Poly-Sery HLB	Surface water and sediment	Neonicotinoids insecticides	HPLC-MS/MS	–	0.00001–0.00005	0.99	83–106	[50]
	MOF	Cabbage and water	Carbamate pesticides	HPLC-UV	0.01–0.02	–	0.05–20	94.4–99.6	[51]
	MOF	Mixed juice, orange juice, and tap water	Phenoxyacetic acid herbicides	HPLC-UV	0.1–0.5	0.2–2	0.2–250	77.1–109.3	[52]
	SPE cartridge	Drinking water	Pesticide	GC-MS	0.00105	0.00319–0.14624	0.001–0.25	55.54–121.21	[53]
				HPLC-MS/MS	–0.04821	–	–	–	
	Oasis HLB	Surface water	Pesticide	HPLC-MS/MS	0.00008	–	0.2–200	50–150	[54]
	Oasis WAX				–0.04433	–	–	–	
	Sep-Pak Plus AC 2								
	–	Whole water	Pesticide	HPLC-MS/MS	0.0001–0.1	0.0002–0.005	0.998	63–113	[55]
	Mesoporous silica doped with Ti	Environmental water	OPPs	GC-NPD	0.4–3.1	0.5–4.4	–	81–104.5	[56]
	Magnetic hollow zein nanoparticles	Water and soil	Chlorpyrifos	HPLC-UV	25	–	50000–2000000	94.3–96.2	[57]
	PCP-Cs	Water and vegetable	Phenylurea herbicides	HPLC-UV	0.01–0.02	0.03–0.06	0.1–80.0	97.5–112.8	[58]
	GO@PA	Environmental Water and leaf lettuces	Phenylurea herbicides and phenylurea insecticides	HPLC-UV	0.10–0.25	0.3–0.75	2.0–100	85.5–106.0	[59]
	GO-Chm	Rice and river Kabul water	Phenylurea herbicides	HPLC-UV	2.5–3.6	8.6–19	50–6000	90.32–94.09	[60]
	GO/HIPes	Farmland water	Triazine herbicides	HPLC-DAD	2.5–5.6	–	25.0–500.0	–	[61]
	NH ₂ @COF	Environmental water	Carboxylic acid pesticides		0.010–0.060	0.04–0.20	0.2–100	89.6–102.4	[62]
	HLB	Water	Pesticides	HPLC-MS	0.000001	0.000004–0.16508	0.9906	76–97	[63]
					–0.04127	–	–	–	
	SDB-RPS and C18	Surface water	Pesticides	GC-MS	0.03–0.172	0.017–0.099	–	75.33–104.99	[64]
				HPLC-MS					
	Mesoporous silica material	Environmental waters	Pesticides	HPLC-MS/MS	–	0.01	1–100	70.1–113.5	[65]
	MWCNT	River water	OPPs	HPLC-MS	0.00002–0.001	0.00007–0.333	0.001–0.2	88.3–124.9	[66]
	PMO	Wastewater	Phenoxy acid herbicides	CE	700–1500	2200–5000	2000–30000	78.3–107.5	[67]
	HBPE	Environmental water	Benzoylurea insecticides	HPLC-UV	0.024–0.068	0.082–0.230	1–25000	85.5–96.12	[68]
	MMT	Fruit juice and natural surface water	Neonicotinoid Insecticide	HPLC-DAD	0.005–0.065	0.008–0.263	0.5–1000	8–176	[69]
	PPPD/PA	Drinking water and lemon and orange juice	OPPs	HPLC-DAD	0.15	0.5	0.5–500	76–102	[70]
	Glass fiber	Water	Pesticide	HPLC-MS/MS	0.0001–0.01	0.0005–0.025	0.999	77–117	[71]
	β-CD/ATP	Environmental water	Pyrethroids	HPLC-UV	0.15–1.03	0.5–3.43	2.5–500	76.8–86.5	[72]
	ATP/Fe ₃ O ₄ /PANI	Environmental water	Benzoylurea insecticides	HPLC-DAD	0.02–0.43	–	0.9985–0.9997	77.37–103.69	[73]
	M-MWCNT	Environmental water	OCPs	GC-μECD	0.00004	0.0001–0.089	0.0002–2	83–115	[74]
					–0.00027	–	–	–	
	G	Environmental water	Triazine and Neonicotine Pesticides	GC-MS and HPLC-MS/MS	0.03–0.4	0.10–1.3	0.5–100	83.0–108.9	[75]

(continued on next page)

Table 1 (continued)

Extraction method	Adsorbent/extraction solvent	Matrix	Analyte	Analytical method	LOD ($\mu\text{g L}^{-1}$)	LOQ ($\mu\text{g L}^{-1}$)	LR ($\mu\text{g L}^{-1}$)	R (%)	Ref.
MSPE	RGO-ZnO	Apple, cucumber and water	OPPs	GC-MS	0.00001–0.00005	0.00005–0.00018	0.0005–0.2	75–104	[76]
	N-octylated magnetic NPs	Drinking and surface water	OPPs	GC-FPD	0.02–0.1	0.05–0.33	0.5–800	82–110	[77]
	Zinc-based MOF	Water and fruits juice	OPPs	GC-FID	0.03–0.21	–	0.1–100	91.9–99.5	[78]
	MOF	Water	Neonicotinoid insecticides	GC-MS	0.02–0.4	0.05–1.0	10–500	73.7–119.0	[79]
	Ch-Si MNP	Water	Pesticides	HPLC-VWD	2–46	6–154	12.5–150	100	[80]
	GO@ IL@NHC–Cu	Environmental water	Triazole fungicides	IMS	0.18	0.6	0.994	92–94	[81]
	LMA-HEDA	Environmental water	Phenylurea herbicides	HPLC-UV	0.027–0.053	–	0.1–4.0	80.1–97.9	[82]
	PP-CMP	Environmental water	Herbicides	HPLC-MS	0.00055–0.00384	0.00183–0.0126	0.05–10	86.9–101.3	[83]
	AF-MCF	Environmental water	Atrazine	HPLC-UV	0.5	–	2–200	94.6–107.2	[84]
	mMFP	Tap and river water	Chlorinated herbicides	HPLC-UV	0.04–0.18	0.13–0.60	2–200	87.4–109.5	[85]
	IL-mG	Surface water	Triazine herbicides	HPLC-UV	0.09–0.15	0.31–0.51	0.55–500	97–100.8	[86]
	Fe ₃ C/MnO/GC	Water, grape, and potato	Herbicides	HPLC-UV	0.01–0.10	0.00004–0.00033	0.1–200	81–112	[87]
	MWCNTs	Environmental water	Sulfonylurea herbicides	HPLC-DAD	0.01–0.04	0.03–0.13	0.05–5.0	76.7–106.9	[88]
	MOF	Environmental water	Pyrazole/pyrrole pesticides	HPLC-DAD	0.3–1.5	1.0–5.0	2.0–200.0	81.8–107.5	[89]
	IL	Environmental water	Pesticides	HPLC-UV	0.5	1.5	2–250	58.9–85.8	[90]
	CNT	Environmental water	Pesticides	GC-MS	0.51–2.29	1.19–5.35	0.9040–0.9733	79.9–111.6	[91]
	Fe ₃ O ₄ @MAIDB	Environmental water	Triazole fungicides	HPLC-DAD	0.0050–0.0078	0.017–0.026	0.05–200.0	75.1–120	[92]
	Fe ₃ O ₄ -EDTA@Zr (IV)	Environmental water	OPPs	GC-MS	0.1–10.30	0.33–34.33	100–20000	86.95–112.60	[93]
	G@SiO ₂ @Fe ₃ O ₄	Environmental water	OPPs	GC-FPD	16–33	50–100	50–5000	90.2–102.9	[94]
	Fe ₃ O ₄ @SiO ₂ -G	Environmental water	OCPs	GC- μ ECD	0.00012–0.00028	–	0.001–0.100	80.8–106.3	[95]
	MI-ILMM	Environmental water	OCPs	GC- μ ECD	0.12–0.26	0.40–0.87	1.0–100	86.2–100.4	[96]
	Co/HPC	Environment water and white gourd	Trizine herbicides	HPLC-DAD	0.02	–	0.2–100	80.3–120.6	[97]
	MOF	Drinking and environmental water	OCPs	GC-MS	0.00039–0.00070	0.00145–0.002	0.002–0.500	79.4–98.3	[98]
	Fe ₃ O ₄ @G-TEOS-MTMOS	Water	Polar and non-polar OPPs	GC- μ ECD	0.0014–0.0237	0.0047–0.0789	0.1–1	83.09–110.53	[99]
	Fe ₃ O ₄ /SiO ₂ /CMCD	Water	Quaternary ammonium herbicides	CE-DAD	0.8–0.9	–	5–500	70.2–100.0	[100]
	Fe ₃ O ₄ -SiO ₂ -P4VP	Water	Phenoxy acid herbicide	CE-UV	3–10	10–30	10–500	80–85	[101]
	Magnetoliposom	Surface water of the Songhua River	OCPs	GC-MS/MS	0.00004–0.00035	0.00014–0.00059	0.0002–0.125	80–109	[102]
	Fe ₃ O ₄ @SiO ₂ @GO-PEA	Fruit, vegetable, and water	OPPs	GC-NPD	0.02–0.1	0.06–0.3	0.06–200	94.6–104.2	[103]
	IL	Water and fruit juices	OPPs	GC-FID	0.02–0.06	–	80–300000	70–89.2	[104]
	ZMNIC	Water and juice	OPPs	GC-MS	0.031–0.034	0.0105–0.112	0.1–500	86–106	[105]
	PPDA-co-Th@Fe ₃ O ₄	Environmental water and fruits juice	OPPs	GC-FID	0.1–0.3	–	0.3–500	88.1–99.2	[106]
	KHA/Fe ₃ O ₄	Environmental water and fruits juice	OPPs	GC-FID	0.03–0.22	–	0.1–200	89.0–99.7	[107]
	RGO/Fe ₃ O ₄ @Au	Seawater	OCPs	GC-ECD	0.0004–0.0041	0.0013–0.0136	0.05–500	69–114	[108]
	CoFe ₂ O ₄ -PGC	Environmental water	Pyrethroids	HPLC-DAD	–	–	–	80.2–110.9	[109]
	Fe ₃ O ₄ -NH ₂ @MIL-101(Cr)	Environmental water	–	GC-ECD	0.005–0.009	0.015–0.025	0.020–2	72.1–106.8	[110]
	M-MWCNTs	Environmental water	Triazole fungicides	HPLC-MS/MS	0.011–0.067	0.037–0.224	0.04–500.0	91.2–108.4	[111]
	Carbon nanosphere @ Fe ₃ O ₄	Environmental water	Triazole fungicides	HPLC-MS/MS	–	0.00056–0.00695	0.95	77.8–93.5	[112]
	MMP/ZIF-8	Environmental water	Triazole fungicides	GC-MS/MS	0.08–0.27	–	1–400	83.4–96.9	[113]

SPME	MIL-101(Fe) @PDA@Fe ₃ O ₄	Environmental water and vegetable Genuine water	Sulfonylurea herbicides	HPLC-DAD	0.12–0.34	–	1–150	87.1–108.9	[114]
	Fe ₃ O ₄ @HPAMAM		Benzoylurea insecticides		0.39–0.72	2.5	2.5–500.0	75.1–111.4	[115]
	MIL-101-NH ₂ @Fe ₃ O ₄ –COOH	Environmental water	Fungicides	HPLC-UV	0.04–0.4	–	0.2–100	71.1–99.1	[116]
	MHMS-MCNP	Surface, ground environmental and wastewater	OPPs	HPLC-UV	0.07	0.23	0.2–1000	96–99	[117]
	Fe ₃ O ₄ -NH ₂ -CAP	Water and soil	Phenylurea herbicides	HPLC-UV	0.01–0.03	0.03–0.09	0.10–40	6.0–103.0	[118]
	3D Co/Ni@C	Water	Pyrethroids	HPLC-UV	0.0038–0.0067	0.013–0.022	0.1–100	85.6–106.9	[119]
	Fe ₃ O ₄ @SiO ₂ @KIT-6	Environmental water		HPLC-UV	0.005–0.01	0.018–0.04	0.02–1200	86.58–98.80	[120]
	TPN/Fe ₃ O ₄ NPs/GO	Water and food	Acidic and basic pesticides	HPLC-UV	0.17–1.7	0.5–5.0	0.5–500	92.7–95.3	[121]
	Fe ₃ O ₄ @SiO ₂ -GO-MOF	Environmental water	Benzenoid-containing insecticide	HPLC-UV	0.30–1.58	1.0–5.2	20.0–1000	81.2–113.1	[122]
	Fe ₃ O ₄ -NH ₂ @MOF-235	Honey, fruit juice and tap water	Benzoylurea insecticides	HPLC-UV	0.25–0.5	–	1.0–300.0	84.02–99.62	[123]
	Fe ₃ O ₄ @PDA-DES	Environment and drinking water	Sulfonylurea herbicides	HPLC-PDA	0.0098–0.0110		5.0–200	72.5–108.6	[124]
	Fe ₃ O ₄ @ CoFe ₂ O ₄ @ PPy	Soil and water	Chlorophenoxy herbicides	HPLC-MS/MS	0.03–3.36	–	0.1–200	80–117	[125]
	Carbonized PDA	Environmental water	OCPs	GC-MS	0.0014–0.015	–	0.05–50	72–109	[126]
	AuNPs-based SPME coating	Environmental water	OCPs	GC-ECD	0.13–0.24	0.44–0.81	–	85.0–97.1	[127]
	MIP	Environmental water	Triazines	HPLC-DAD	0.024–0.030 2.0–5.3	–	–	78–104	[128]
	Fe ₃ O ₄ @MIL-100(Fe) Biosorbent-based fiber	Environmental water	Triazine Herbicides			6.1–15.7	0.0061–70	97.5–103	[129]
		Water	OCPs	GC-ECD	0.00019 –0.00071	0.00065–0.00238	0.005–0.100	60–113	[130]
	Cork fiber	Water	OCPs	GC-ECD	0.0003–0.003	0.001–0.01	0.01–0.075	60.3–112.7	[131]
	C18	Water	OCPs	GC-MS	0.000059 –0.000151	0.000197–0.000503	0.002–0.5	70.2–119	[132]
	Hierarchical G	Water	OCPs	GC-MS	0.00008 –0.00080	0.00025–0.0027	0.01–30	82.8–113	[133]
	Mesoporous TiO ₂ NP	Water	OCPs	GC-MS	0.00008 –0.00060	0.00027–0.002	0.005–0.500	81.2–118.1	[134]
	Sol-gel/nanoclay	Water	OPPs	GC-MS	0.003–0.012	0.01–0.02	0.01–2.0	86–104	[135]
	Mesoporous silica	Water	Triazole pesticide	GC-MS	0.05–0.09	–	0.1–2000	–	[136]
	–	Water	Pesticide carbaryl	UV-VIS	0.033	0.11	100–1200	97–112	[137]
	PDMS/DVB	Surface and ground water	Pesticides	GC-MS	0.05–1	–	0.05–20	–	[138]
	CAR-PDMS	Waste water	Hydrazine	GC-MS/MS	0.002	0.007	0.9986	–	[139]
	MMF-MAED	Water and juice	Benzoylurea insecticides	HPLC-DAD	0.026–0.075	0.084–0.25	0.1–200	65.1–118	[140]
	rGOQD@Fe	Fruit juice and real water	OPPs	GC-MS	0.04–0.07	0.11–0.21	0.14–540	82.9–113.2	[141]
	CNT@ SiO ₂	Vegetables, fruits and water		GC-IMS	0.005–0.020	0.010–0.050	0.01–3	79–99	[142]
	NH ₂ -MIL-53(Al)	Tap and river water	OCPs	GC-MS	0.000051 –0.00097	0.083–3.23	0.001–1	77.4–117	[143]
	ZnO/g-C ₃ N ₄	Cucumber, pear, Green tea and Minjiang water (local river)	Pesticides	GC-MS	0.001–0.0025	0.003–0.005	0.003–5.0	79.1–103.5	[144]
	PA	River, fountain, reservoir and wellspring	Fungicides	GC-MS/MS	lower than 0.001	–	0.001–2	92–104	[145]
	Co/Cr (NO ₃) ⁻ -LDH	Environmental water	Acidic pesticides	HPLC-UV	0.05–0.08		0.1–500	90–110	[146]

(continued on next page)

Table 1 (continued)

Extraction method	Adsorbent/extraction solvent	Matrix	Analyte	Analytical method	LOD ($\mu\text{g L}^{-1}$)	LOQ ($\mu\text{g L}^{-1}$)	LR ($\mu\text{g L}^{-1}$)	R (%)	Ref.
SBSE	MMF	Environmental water and orange juice	Carbamate pesticide	HPLC-DAD	0.017–0.29	0.057–0.96	0.1–200	80.4–117	[147]
		Tap, river and waste waters	Sulfonylurea herbicides	HPLC-DAD	0.009–0.018	0.028–0.060	0.0500–200	70.1–108	[148]
		Water and soil	Sulfonylurea herbicides	HPLC-DAD	0.018–0.17	0.061–0.44	0.1–200.0	70.6–119	[149]
	MOF	Environmental water	Fungicides	HPLC-MS	0.00134–0.0148	0.00445–0.0494	0.005–5	90.4–97.5	[150]
		Environmental water	Triazine herbicides	DART-MS	0.005–0.05	–	0.991	92.4–125.7	[151]
		Environmental water	OPPs	GC-NPD	0.005–0.008	0.015–0.025	0.015–50	88–108	[152]
	PDMS	Surface water	Pesticides	GC-MS/MS	0.001–0.458	0.039–0.732	0.05–100	75.6–137.31	[153]
	Si-GO	Ground and mineral water	Triazines	HPLC-MS/MS	0.0011–0.0029	0.0038–0.0097	0.2–4.0	–	[154]
	MMC	Environmental water	Triazoles	HPLC-DAD	0.014–0.031	0.11–0.074	0.100–200	78.9–106	[155]
	Magnetism-enhanced monolith	Environmental water	Triazines	HPLC-DAD	0.074–0.23	0.24–0.68	0.5–200	70.7–119	[156]
	POM-IL	Environmental water	Triazole pesticides	GC-MS	0.005–0.03	–	0.02–100	–	[157]
	PDMS	Water	OCPs	GC-MS	0.00012–0.00207	–	0.01–0.5	64.7–111	[158]
	MWCNTs-PDMS	Water and soil	Triazine herbicide	IMS	0.006–0.015	0.02–0.05	0.05–10	85.9–104.3	[159]
	PDMS-MIL 100-Fe	Environmental water	Triazine	HPLC-UV	0.021–0.079	–	0.2–500	98–118	[160]
	ZnS-AC	Tap, river and mineral water	Carbamate insecticide		0.03–0.05	1–1.7	2–30000	91.7–98	[161]
	MOF	Environmental water	OPPs	GC-FPD	0.043–0.085	–	0.2–100	89.3–115	[162]
	CDD	Farm field water	Endocrine disruptor pesticides	GC- μ ECD	0.004–0.0045	0.001–0.015	0.005–5	83.2–98.7	[163]
LLE	MOF	Soil and lake water	Sulfonylurea herbicides	HPLC-UV	0.04–0.84	–	10–800	68.8–98.1	[164]
	ZrO ₂ -rGO	River water and agricultural wastewater	Ethion	IMS	1.5	5.0	5.0–200	93–97	[165]
	MMT/PPy/N6	River	OPPs	GC-MS	0.05–0.3	–	0.3–1000	80.3–95.3	[166]
	ZrO ₂ /SiO ₂ composite	Environmental and Food	OCPs	GC-MS	0.03–1.3	0.1–4	0.0001–5	69.0–114.1	[167]
	SMNM	Environmental water	OP insecticides	HPLC-UV	0.07–0.89	0.23–2.94	1–1000	80.60–104.52	[168]
	Acetonitrile and DBE	Aqueous samples	Pesticides	GC-FID	0.34–5	1–16	1–10000	91–105	[169]
	DCM	Fresh water sediment	OTP	HPLC-MS	0.13–44	0.43–1.46	5–250	73.7–119.6	[170]
	DCM	Water	Benzulfuron-methyl herbicide	GC-MS	0.1	–	50–5000	74.1–94.1	[171]
	Salting-out solution (NaCl in phosphate buffer)	High salinity and biological samples	Pyrethroid insecticides	GC-MS	1.5–60.6	–	5–5000	74–110	[172]
	DCM and ethylacetate	Surface water, sediment, and fish	Pyrethroid insecticides	HPLC-PDA	0.01–0.03	0.05–0.07	0.07–0.49	97–99	[173]
LPME	n-Hexane	Surface waters	Pyrethroid and OP insecticides	GC-MS	–	0.0125–0.125	0.98	67–114	[174]
	DCM and hexane	River, pond and tube well water and river sediment	Pesticides	GC-MS/MS	–	0.01–0.08	5–250	70.76–111.52	[175]
	Nanofluids [C4MIM]PF ₆	Environmental water	Fungicides insecticides	HPLC-UV	0.13–0.19	0.44–0.64	0.996	74.94–96.11	[176]
		Water	OPPs	HPLC-UV	0.01–0.1	0.05–0.4	0.09–200	96.9–103.2	[177]
	Decanoic acid and tetrabutylammonium hydroxide	Water and fruit juice	OPPs	HPLC-UV	0.1–0.35	–	0.5–400	92.2–110.5	[178]
	1-Dodecanol/p-xylene	East lake, well and rainwater	OPPs	GC-FPD	0.12–0.56	–	0.6–100	83.7–112	[179]
	DCM and DCE	Dam lake, river and well water	Herbicides	GC-MS	0.3–2.0	6.5	5.0–100	83–121	[180]
	DLLME	Environmental water	Fungicides	HPLC-UV	0.02–0.10	–	0.5–500	70.7–105	[181]

SPE-DLLME	[P4448] [Br] and Na [N(CN) ₂]	Environmental water	Pyrethroid pesticides	HPLC-UV	0.16–0.21	–	1–100	80.20–117.31	[182]
	[BeMIM][Tf ₂ N]	Environmental water	OPPs	HPLC-UV	0.01–1.0	–	0.9994–0.9998	82.7–118.3	[183]
	Acetic acid-containing CLF	Environmental water	OPPs	GC-FPD	0.004–0.03	–	0.03–300	75.8–104.2	[184]
	1-Octanol	Environmental water	Multiresidue pesticides	HPLC-DAD	0.8–3.3	2.5–11.0	8.6–800	81–121	[185]
	TCE	Water	Pesticide	GC-MS	0.0032–0.0174	0.0096–0.052	0.0096–100	84–108	[186]
	[(C ₄ MIm) Cl] and (Li [NTf ₂])	Water	OPPs	GC-MS	0.005–0.16	0.017–0.054	0.005–0.016	97–113	[187]
	M-β-CD/ATP	Water	Fungicides	HPLC-DAD	0.02–0.04	–	1.00–500	98–115	[188]
	[P ₄₄₄₁₂][PF ₆]	Water	Pyrethroid insecticides	HPLC-UV	0.71–1.54	–	1–500	87.1–101.7	[189]
	Decanoic acid	Water		HPLC-UV	0.24–0.68	–	1–500	84.7–95.3	[190]
	1-Octanol	Water	Pesticide	HPLC-MS/MS	–	0.0125–1.25	0.9849–0.9962	60–120	[191]
	LiNTf ₂	Water	Triazole fungicide	HPLC-UV	0.74–1.44	–	5–250	70.1–115	[192]
	1-Undecanol	Water and grape juice	Herbicides and fungicides	HPLC-MS	0.0027–0.0097	0.009–0.0323	0.05–10	72.4–101.5	[193]
	[N8881] PF ₆	Water and tea beverage	Benzoylurea insecticides	HPLC-UV	0.29–0.59	0.97–1.97	2–500	85.93–90.52	[194]
	DBE	River water and fruit juice	OPPs	GC-FID	0.82–2.72	2.60–7.36	3.1–1000	64–83	[195]
	1-Dodecanol	Mineral water	Triazine and triazoles	HPLC-MS	0.00375–0.0375	0.0125–0.125	0.125–3.75	70–118	[196]
	CLF and MCB	Mineral water	Triazine, neonicotinoid triazole and imidazolinone pesticide	HPLC-MS/MS	–	0.005–0.5	0.5–15	102–120	[197]
	CTC	Water and soil	Fungicide carbendazim	UV–Vis	2.1	4	5–600	95.2–105.2	[198]
	CLF and MeOH	Tap and waste water	Pesticide	GC-MS	0.09–3.36	0.31–11.19	0.50–500	84–109	[199]
	Aliquat–336 in 1-octanol	Water and soil	Sulfonyl urea herbicide	HPLC-UV	0.5	–	1–100	89.8–110.1	[200]
	DBE	Aqueous sample and Fruit juice	OPPs	GC-FID	0.65–1.3	2.2–4.5	3–40000	67–95	[201]
	Tetrachloroethene	Sea and surface water	OPPs	GC-MS/MS	0.003	0.007–0.012	–	30–130	[202]
	Tetrachloroethene	Aqueous samples, fruit juices, and vegetables	OPPs	GC-NPD	0.012–0.056	0.044–0.162	0.05–60	84–92	[203]
	CLF and undecanol	Water and fruit samples	Fungicides	HPLC-MS/MS	–	0.001	–	86–116	[204]
	DCE	Tap water and wastewater	Pesticides	GC-MS	0.30–2.0	0.53–5.4	0.50–1000	86–107	[205]
	Decane	Honey, tomato and environmental water	Triazine herbicide	S-MEKC	0.07–0.69	–	0.3–100	86.4–114	[206]
	CLF and DCM	Natural water	Pesticides	HPLC-DAD	0.015–0.36	0.049–1.2	2.9–2500	84.8–106.1	[207]
	[C ₆ MIM][PF ₆]	Surface sample		HPLC-MS/MS	0.1–0.8	0.5–2.5	0.5–50	70–120	[208]
	Trichloroethylene	Environmental water	Herbicides	GC-MS	0.045–0.03	0.10–0.15	0.10–200	87.2–111.2	[209]
	CLF and DCE	Wastewater and sludge	OCs		0.16–1.5	0.52–4.9	0.50–500	82–108	[210]
	1-Dodecanol	Environmental water	Triazole fungicides	HPLC-DAD	5.1–9	16.9–29.9	50–5000	77.6–104.4	[211]
	ChCl(ASA) ₂ MeOH	Surface water	Fungicides, insecticides and acaricides	HPLC-MS/MS	0.002–2.3	0.006–7.7	0.9737–0.9975	18–96	[212]
	CLF	Waters, soil, food and beverage	Carbamate insecticides	IMS	1.04–1.31	–	2–100	96–101	[213]
	DES	Environmental water	Pyrethroid insecticides	HPLC-UV	0.30–0.60	1.00–2.00	0.9992–0.9995	80.93–109.88	[214]
	LiNTf ₂	Environmental water	Fungicides	HPLC-DAD	0.032–0.89	–	1–100	89.4–96.4	[215]
	[MimCH ₂ COOCH ₃][NTf ₂]	Environmental water	OPPs	HPLC-UV	0.7–2.7	–	1–100	96.3–114.2	[216]
	Toluene	Environmental water	Triazole fungicides	GC-MS	0.14–0.27	0.47–0.90	1–100	89.3–108.7	[217]
	1-Dodecanol	Environmental water	Pyrethroid	HPLC-VWD	0.37–0.75	–	1–500	91.3–98.1	[218]
	MWCNT, silica-based C18, and polymeric solid sorbent	Environmental water	OCs	GC-MS	0.005–0.22	–	500	–	[219]

(continued on next page)

Table 1 (continued)

Extraction method	Adsorbent/extraction solvent	Matrix	Analyte	Analytical method	LOD ($\mu\text{g L}^{-1}$)	LOQ ($\mu\text{g L}^{-1}$)	LR ($\mu\text{g L}^{-1}$)	R (%)	Ref.
MSPE-DLLME	MIP	Tap water, ground water, grape juice	Triazine herbicides	GC-FID	0.2–7	0.5–20	0.5–150	92–98	[220]
	C18	Water, milk, honey and fruit juice	Pesticide	GC-MS	0.0005–0.001	–	0.001–10	78.1–105	[221]
	MOF	Environmental water and tea	Pyrethroids	GC-ECD	0.008–0.015	0.028–0.050	0.05–10.0	78.3–103.6	[222]
QuEChERS	MGO	Environmental water	Atrazine herbicide	GC-MS	0.0006	0.002	0.005–5	96–102	[223]
		Water	Pesticide	GC-MS	0.3–4	0.95–13.69	0.995–0.999	85.3–107	[224]
		Soil and water	OCFs	GC-MS	0.26–1.89	0.86–9.99	0.9931–0.9999	70.0–118.0	[225]
		Food and water	Pesticides	HPLC-MS/MS	–	0.0001–0.0478	0.994–0.999	93–96	[226]

materials for surfactant modification as they present a high surface area and negative charges that facilitate the adsorption of cationic surfactants. Cetyltrimethylammonium ammonium bromide (CTAB)-modified zeolite has both hydrophilic and hydrophobic phases. Hence, it can be practical for the strongest functional groups of target compounds. Admicellar sorbent composed of CTAB-coated zeolite NaY has been demonstrated as a novel sorbent for effective extraction/pre-concentration of six carbamate pesticides, including carbofuran, promecarb, isoprocarb, methiocarb, aldicarb, and carbaryl. The quantitative retention of target pesticides on the admicellar sorbent is based on hydrophobic interaction with a hydrocarbon core of CTAB aggregates and π -cation interaction between the polar head of surfactant and aromatic rings of pesticides [27].

Mann and colleagues [34] developed an online-SPE-liquid chromatography (LC)-tandem mass spectrometry (MS) multi-residue method to analyze a wide range of different classes of pesticides and their degradation products in drinking, ground and surface water by using very low sample volumes and just one type of SPE sorbent. This method has been successfully used to monitor the aquifer treatment process, usually contaminated with thiamethoxam. Using online-SPE systems, less sample volume is needed, therefore minimizing sampling time, the amount of sample and manpower expenses for the on-site sampling [34].

Kadokami et al. [24] reported a comprehensive screening method for 311 organic compounds like pesticides with a wide range of physicochemical properties ($\log P_{ow}$ 2.2–8.53) in water samples by combining the SPE with LC-MS. In this study, five commercial SPE cartridges including C₁₈, PS2, Oasis hydrophilic lipophilic-balanced (HLB), AC2 and PLS-3 contain different absorbents were examined to find absorbents that extract a large number of target compounds. Recoveries of the pesticides with $\log P_{ow} < 2$ were low when using C₁₈, whereas PS2, HLB, and PLS-3 were able to extract pesticides with $\log P_{ow}$ below 0 to over 4. Also, the AC2 cartridge had high recoveries of 20 pesticides that were not extracted with high recoveries by the other four cartridges. From these results, it was inferred that tandem extraction using a combination of either PS2, HLB or PLS-3, and AC2 was most suitable for the simultaneous extraction of the widest range of organic compounds [24].

Ding et al. [11] presented a surface imprinting strategy for the preparation of magnetic super hydrophilic molecular imprinted resin (MIR) based on MWCNTs to detect triazines in the environmental water samples. The surface imprinting was carried out by one-pot condensation of formaldehyde, resorcinol, and melamine on the surface of MWCNTs@Fe₃O₄. The obtained MWCNTs@Fe₃O₄@MIR exhibited strong magnetic responsiveness, super hydrophilic, good recognition towards triazines and high adsorption capacity. The established method was successfully applied to detect triazines in environmental water samples and the satisfactory results were obtained. The established method exhibited good linearity range (LR), lower (LOD) (0.007–0.068 $\mu\text{g L}^{-1}$), and excellent precision (relative standard deviations (RSD) < 9%) [11].

Sun and colleagues [63] prepared a novel sequential-mode SPE cartridge using three kinds of sorbents employed for the extraction of pesticides in environmental water. The SPE approach consisted of the application of HLB polymer, strong cation and anionic exchange sorbents packed in layered sequence in a single cartridge. The method displayed low LOD for both water matrices (0.000001–0.04127 $\mu\text{g L}^{-1}$) and achieved good LR over the tested range of concentrations [63].

2.2. Dispersive solid-phase extraction

Dispersive solid-phase extraction (dSPE) has been used as a preconcentration method for the analysis of several compounds.

dSPE is based on the addition of a sorbent into the analytical solution followed by dispersion favoring the contact between the sorbent and the analytes. When the dispersion process is completed, the sorbent, with the analytes retained on its surface, is separated by a mechanical system, such as centrifugation or filtration. This method has advantages including short sample treatment time, simplicity, adaptability, and easy handling in comparison with the traditional techniques [231].

G, as a new two-dimensional carbon nanomaterial, has great interest among scientists in diverse fields. Due to its large specific surface area, chemical stability, flexibility, and π -electron-rich structure, make a good choice as an adsorbent for sample pretreatment [232].

Despite that, there are some problems for directly used G as an adsorbent such as aggregation of G in aqueous solution and blocked the frits in SPE cartridge or column. One way to overcome these problems is the decoration of the surface of G sheets by nanoparticles such as ZnO that leads to maintaining the advantageous characteristics of G sheets. Furthermore, the water-soluble analytes are adsorbed successfully by the hydrophilic surface of ZnO in water. Also, the high density of ZnO helps the superficial separation of RGO-ZnO from the water after adsorption by centrifugation [233].

Li et al. [76] synthesized RGO-ZnO, using a facile hydrothermal reduction strategy. The nanocomposites could be produced directly from GO in a facile one-step reaction, where the reduction of GO and the deposition of ZnO on G occur simultaneously. This method is simple, efficient and environment-friendly. Then, RGO-ZnO was used as dSPE sorbent for the enrichment of eight OPPs in apple, cucumber, and water samples prior to their determination by GC-MS. Under the optimal experimental conditions, good LR existed in the range of 0.0005–0.2 $\mu\text{g L}^{-1}$ for all the analytes with the correlation coefficients (R^2) ranging from 0.9894 to 0.9983. The LOD of the method for the compounds were between 0.00001 and 0.00005 $\mu\text{g L}^{-1}$. The recoveries of the method were in the range from 75.0 to 104.2% [76].

MOFs are a class of crystal ultra-porous materials with high surface area and porosity because of the ordered framework produced by the metal ions coordinated with organic linkers. The unique structures and properties of MOFs, such as uniform pores and structural diversity, make them appealing for use in sample pretreatment [234].

Amiri et al. [78] synthesized the zinc based-MOF that was used as a dSPE sorbent for the extraction and pre-concentration of OPPs in water and fruit juice samples. Based on the high affinity of zinc towards phosphoric groups, effective extraction of OPPs could occur. The optimized method exhibited good LR for all of the target analytes in the 0.1–100 $\mu\text{g L}^{-1}$ concentration range. The LOD for the established method was found to be 0.03–0.21 $\mu\text{g L}^{-1}$. This method can be used for the analysis of OPPs in water and fruit juice samples with recoveries of the analytes in the range of 91.9–99.5% [78].

Zhao et al. [83] synthesized a novel polyphenylene-based conjugated microporous polymer (PP-CMP) and employed as an efficient adsorbent for dSPE of trace amounts of polar phenoxycarboxylic acids herbicides in water systems. The proposed method based on PP-CMPs achieved high sensitivity with LOD of 0.00055–0.00384 $\mu\text{g L}^{-1}$, satisfactory correlation coefficients (≥ 0.9912), and good LR (0.05–10 $\mu\text{g L}^{-1}$). Moreover, this method was used for simultaneous measuring of the amounts of five herbicides in environmental water samples with satisfactory spiked recoveries (86.9–101.3%). All these facts demonstrated that this new dSPE technique based on PP-CMPs exhibited a promising potential for highly sensitive analysis of trace phenoxycarboxylic acids herbicides in complex samples [83].

Deep eutectic solvent (DES) has been proposed by Abbott et al. [235]. DES contained two or three compounds, which could interact by hydrogen bonding. One of the components is a hydrogen-bond acceptor (HBA) such as quaternary ammonium salts, and another

component is a metal salt or hydrogen-bond donor (HBD). DES has been classified as a new type of ionic liquids (ILs), because of the similar physicochemical properties to the IL. However, compared with traditional ILs, DES is easier to synthesize, lower cost, more biocompatible and biodegradable, and no need further. Shemirani et al. [74] investigated the application of DESs as a new class of carriers for CNT based nanofluids. By combining the MWCNT nanocomposite and DES, a highly stable (stability against sedimentation) viscous magnetic bucky gel or soft material was prepared without any additional stabilizer. They used this magnetic DES-based soft material in developing a new DES magnetic bucky gel-based dSPE. By coupling this method with gas chromatography-micro-electron capture detector (GC- μ ECD) and using a large-volume-injection technique they achieved low LOD (0.00004–0.00027 $\mu\text{g L}^{-1}$) and good recovery (83–115%). This method can be used as a pre-concentration technique in trace analysis of OCPs [74].

2.3. Magnetic solid-phase extraction

Magnetic solid-phase extraction (MSPE) is relying on the adsorption of the target analyte(s) on a magnetic adsorbent. Generally, in this procedure, the extraction is simple and fast with no need for expensive equipment and a large volume of the eluent. The adsorbed analyte(s) can be easily separated by an external magnetic field [236]. The extraction efficiency is thus enhanced as this procedure avoids column packing and possible blockage in the case of traditional column SPE. In addition, owing to its nano nature, magnetic nanoparticles (MNPs) possess large specific surface area, and the equilibrium time between the sorbents and sample solution is thus greatly shortened, which results in relatively higher extraction capacity.

Wan et al. [95], synthesized a new G-based silica-coated magnetic adsorbent ($\text{Fe}_3\text{O}_4@\text{SiO}_2\text{-G}$) and applied for the pre-concentration of four selected OCPs from aqueous media. G due to its honeycomb-like structure merits particular regard. Especially, the high surface area, remarkable adsorption capacity, diversity of benzene rings and especially rich π - π electron arrangement makes G a suitable extractant for the extraction of benzene-based pesticides. Magnetic Fe_3O_4 nanoparticles have provided important avenues to prepare new, stable and efficient extraction reagents as well as green sample preparation. SiO_2 is cheap, environmentally friendly, chemically stable and highly dispersive in a liquid due to its rich O–H groups. Therefore, generally, Fe_3O_4 nanoparticles are coated with different silane derivatives via the sol-gel method to increase the surface area porosity and effective binding sites. The higher extraction efficiency of the $\text{Fe}_3\text{O}_4@\text{SiO}_2\text{-G}$ adsorbent for non-polar as well as relatively polar pesticides is due to the existence of a benzene-ring part in the adsorbent structure which can form a strong π - π interaction with the benzene ring of the selected chlorpyrifos, hexaconazole and azaconazole pesticides. An H-bonding interaction was also observed and lindane affinity can be explained by hydrophobic and electrostatic interaction. Fig. 2 shows the schematic mechanism for the adsorption of the chlorinated pesticides by $\text{Fe}_3\text{O}_4@\text{SiO}_2\text{-G}$ [95].

Ma et al. [89] prepared a simple, sensitive and robust method using the magnetic MOFs of $\text{Fe}_3\text{O}_4@\text{SiO}_2\text{-MIL-101}$ as MSPE sorbents that were successfully developed for the simultaneous determination of four pyrazole/pyrrole pesticides in environmental water samples followed by high-performance liquid chromatography-diode array detector (HPLC-DAD). Good extraction efficiencies and low LOD (0.3–1.5 $\mu\text{g L}^{-1}$) and limit of quantification (LOQ) (1.0–5.0 $\mu\text{g L}^{-1}$) were obtained. The analysis requires no complicated devices since the $\text{Fe}_3\text{O}_4@\text{SiO}_2\text{-MIL-101}$ could be separated quickly from solutions by an external magnet. The enrichment and

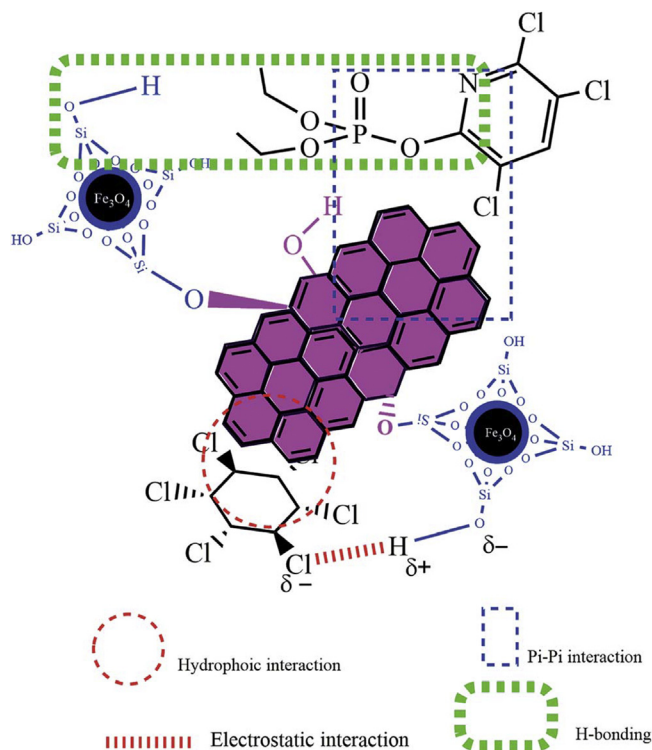


Fig. 2. The mechanism for the adsorption of the chlorinated pesticides by $\text{Fe}_3\text{O}_4/\text{SiO}_2\text{-G}$ (Reprinted from Ref. [95] with permission from RSC).

extraction of four pyrazole/pyrrole pesticides were convenient, rapid, cost-effective and eco-friendly. The developed MSPE-HPLC-DAD method provided great potential to analyze pyrazole/pyrrole pesticides in real water samples [89].

Porous carbon materials (PCM) have attracted considerable interest in analytical science because of their distinct chemical properties and extensive applications as adsorbents for various analytes [237]. The porosity and surface compositions of carbon materials are strongly dependent on the fabrication process and they can be modified according to the requirement. PCM can also be fabricated from MOFs through facile pyrolysis [238]. The well-organized skeleton of MOFs can be changed to porous carbon with uniform heteroatom decoration. Especially, the long-range ordering and high porosity of MOFs can also be partially preserved, exhibiting carbon as an excellent adsorbent with size selectivity. The potential usage of MOF-derived carbon as an adsorbent for solid-phase sorption-based extraction techniques has been reported [89,98]. Chen et al. [98] explored a magnetic PCM derived from a bimetallic MOF as an adsorbent for MSPE of OCPs in environmental water. The bimetallic MOF has the structural properties of both zeolite imidazole framework (ZIF)-8 and ZIF-67 and provides a promising template for nanoporous carbon (NPC) with good stability and high porosity. The developed method was successfully applied for the analysis of trace OCPs in drinking and environmental water samples. This method displayed good LR ($r > 0.99$) within the concentration range of $0.002\text{--}0.5\text{ }\mu\text{g L}^{-1}$. Low LOD ($0.00039\text{--}0.0007\text{ }\mu\text{g L}^{-1}$) and LOQ ($0.00145\text{--}0.002\text{ }\mu\text{g L}^{-1}$), as well as good precision, were also obtained [98].

Amiri et al. [106] prepared the magnetic poly (p-phenylenediamine-co-thiophene) and used as an adsorbent for extraction and determination of OPPs in environmental waters and fruit juices samples. This method showed a linear response between 0.3 and $500\text{ }\mu\text{g L}^{-1}$, LOD between 0.1 and $0.3\text{ }\mu\text{g L}^{-1}$ and relative recoveries in the range of 88.1–99.2%.

Magnetoliposomes, magnetic nanoparticles coated with the lipid bilayer, have been prepared for medical applications such as drug carriers. Magnetoliposomes are produced by the zwitterionic head groups of phosphatidyl choline attached to the surface of Fe_3O_4 nanoparticles. The adsorption process continues until the surface saturates. The hydrophobic interactions between long double hydrocarbon chains of phosphatidyl choline result in the formation of lipid bilayers [239]. Lipid bilayers provide adsorption sites for organic compounds through hydrophobic interactions, and zwitterion head groups endow the outer surface of magnetoliposomes with hydrophilicity to increase the dispersing property of adsorbents in aqueous solution. Magnetoliposomes have enticed attention in the scientific community due to their good hydrophilicity and excellent adsorption ability for organic pollutants. The limited studies have been reported on the application of magnetoliposomes as solid-phase adsorbents for environmental water analysis. Ding et al. [102] expanded a simple and efficient method based on magnetic separation for the extraction of OCPs from river water samples using magnetoliposomes as adsorbents.

Hydroxyapatite bioceramic as the useful Calcium Phosphate (Ca-P) compound has two ionic C and P binding sites on the surfaces of uniting cell, which is rich in cationic and phosphate ions, and is well known as a good adsorbent for many anionic and cationic compounds [240]. Amiri et al. [107] synthesized magnetic potassium substituted hydroxyapatite (KHA/ Fe_3O_4) for the extraction of OPPs from environmental waters and fruit juice samples. The OPPs were analyzed by GC-FID. Under optimum conditions, good LR was achieved for all analytes in the $0.1\text{--}200\text{ }\mu\text{g L}^{-1}$ range. The LOD was between 0.03 and $0.22\text{ }\mu\text{g L}^{-1}$. The recoveries of OPPs from spiked real water samples and fruit juices are between 89.0 and 99.7% [107].

Yang et al. [124] developed an MSPE technique coupled with HPLC for the determination of four sulfonylurea herbicides (sulfosulfuron, bensulfuron-methyl, pyrazosulfuron-ethyl, and halosulfuro-methyl) in aqueous samples. The key point of this method is the application of a novel magnetic nanomaterial (Fe_3O_4 @PDA-DES). Polydopamine (PDA) is a polymer formed by dopamine self-polymerization under alkaline conditions. Fe_3O_4 coated with PDA has exclusive adhesion and superb biocompatibility. In addition, PDA has a variety of functional groups, such as carboxyl and amino, which could be modified easily. In this work, dioctadecyl dimethyl ammonium chloride (HBA) and 4-chlorophenol (HBD) were mixed and heated to obtain a novel DES. This new DES could dissolve in water easily and accelerate the dispersion of sorbents in the sample. Under the optimum conditions, the LR of this method ranged from 5.0 to $200\text{ }\mu\text{g L}^{-1}$ for all analytes, with correlation coefficients (r) ≥ 0.9901 . The enrichment factors were reported between 495 and 630, and the RSDs were less than 3.6%. The LOD varied from 0.0098 to $0.0110\text{ }\mu\text{g L}^{-1}$. In the final experiment, the developed method has been successfully applied to the determination of sulfonylurea herbicides in the environment and drinking water samples, and the obtained recoveries were between 72.5% and 108.6% [124].

2.4. Solid-phase microextraction

Solid-phase microextraction (SPME) is a simple sample preparation method with benefits including low solvent usage, high speed, sensitive and easy coupling to GC and HPLC. The SPME first developed by Pawliszyn's group in the 1990s. This technique has been considered a promising pretreatment method and has been widely used in the fields of the environment [241,242], food [243,244], biology [245,246], clinical medicine [247,248], toxicology [249] and forensic medicine [250].

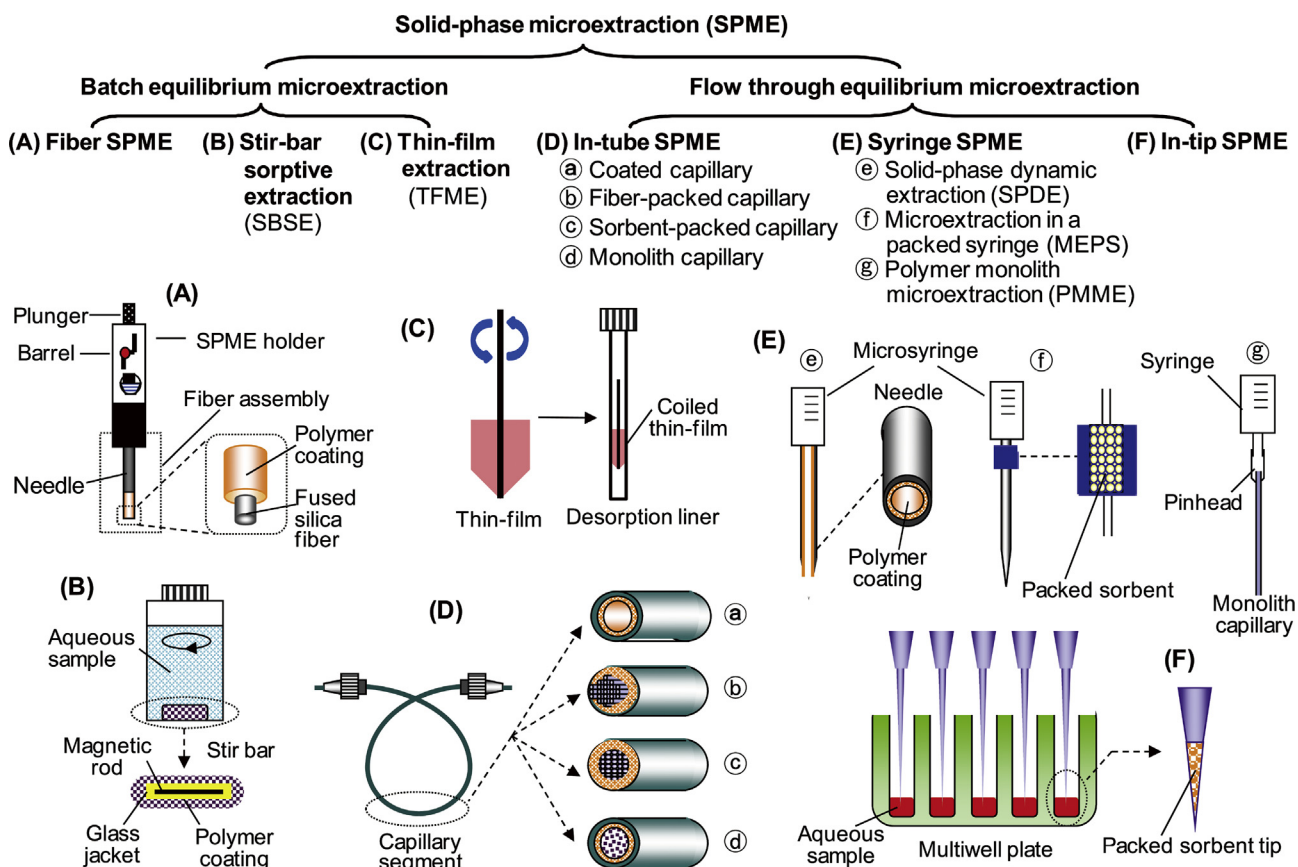


Fig. 3. Configurations of various devices for SPME and related microextraction techniques [Reprinted from Ref. [251] with permission from Elsevier].

Fig. 3 shows the main modes of SPME [251]. The SPME mechanism is founded on the basis of the distribution of analytes between the bulk and the fiber coating. Therefore, the stationary phase coated on the fiber plays a significant task in prospering the sensitivity, mass transfer velocity and repeatability of the method [252]. In the past decades, many endeavors have been made to expand the novel SPME coating materials with substantial chemical and thermal constancy, enhanced sensitivity and selectivity for the specified analytes.

Octadecylsilane (ODS), also called C18, is a kind of surface-modified amorphous silica. It is well known that the C18-bonded silica phase has good separation features, and is widely used in SPE to pre-concentrate or cleanup nonpolar and medium-polar components [36–40]. Besides SPE, the use of C18 as a hydrophobic stationary phase also has attracted considerable attention in SPME. The C18 functionalized SPME fibers were used to determine OCPs in water samples. Zhu et al. [132] prepared a C18 composite fiber by a column-assisted method with the stainless steel wire as a fiber substrate. With the assist of the GC column, the home-made C18 composite fiber can be equipped in a quite constant thickness of 35 mm and a batch of fibers can be prepared at one time. In addition, the fiber exhibited excellent properties, like good thermal stability and solvent resistance, wide LR range satisfied repeatability and reproducibility [132].

An environment-friendly method has been developed by Ouyang et al. [133] for the production of a 3D continuous G framework with hierarchical pores of macropores and in-plan nanopores. This kind of 3D G framework possesses the original continuity of G sheets and directly shortens the mass cross-plan distance due to the in-plan nanopores. Hierarchical pores graphene frameworks (HPGF)

exhibited excellent extraction performance for the five OCPs when compared with the GO and commercial fiber. In addition, the fiber also exhibited low LOD ($0.00008\text{--}0.00080\text{ }\mu\text{g L}^{-1}$) wide LR range ($0.01\text{--}30\text{ }\mu\text{g L}^{-1}$), satisfying repeatability, reproducibility, sensitivity and good recoveries for the real water sample, which shows that the developed method can be used as a simple, efficient extraction and pre-concentration technique for tracing OCPs in water samples. Fig. 4 shows the surface characteristics of the GO coating and the HPGF coating [133].

In another research conducted by Ouyang et al. [143] the $\text{NH}_2\text{-MIL-53(Al)}$ nanoparticles applied as the SPME fiber coating for efficient sample pretreatment owing to their unique structure and excellent adsorption properties. Compared with commercial SPME coatings, the $\text{NH}_2\text{-MIL-53(Al)}$ -coated fiber exhibited excellent characteristics, such as good thermal stability and solvent resistance, long lifetime, large enhancement factors, and good reproducibility for the SPME of OCPs in environmental water samples. Among the various MOFs, the material of Institute Lavoisier (MIL-53(M) (M is Fe, Al, or Cr) is of special interest. MIL-53 (Al) is built of infinite chains of octahedrons formed by coordination of Al (III) by terephthalate and OH groups with the pore walls consisting mainly of aromatic rings. The structure of MIL-53(Al) has remarkable flexibility, adjusting its cell volume in a reversible manner to optimize interactions between the guest molecules and the framework [143].

In-tube SPME (IT-SPME) is a sample preparation technique that can be employed in combination with either HPLC or GC for online analysis. This technique is very fast, automated with low consumption of organic solvents and samples [253]. Different extraction phases are generally used for IT-SPME, either filled into a metal

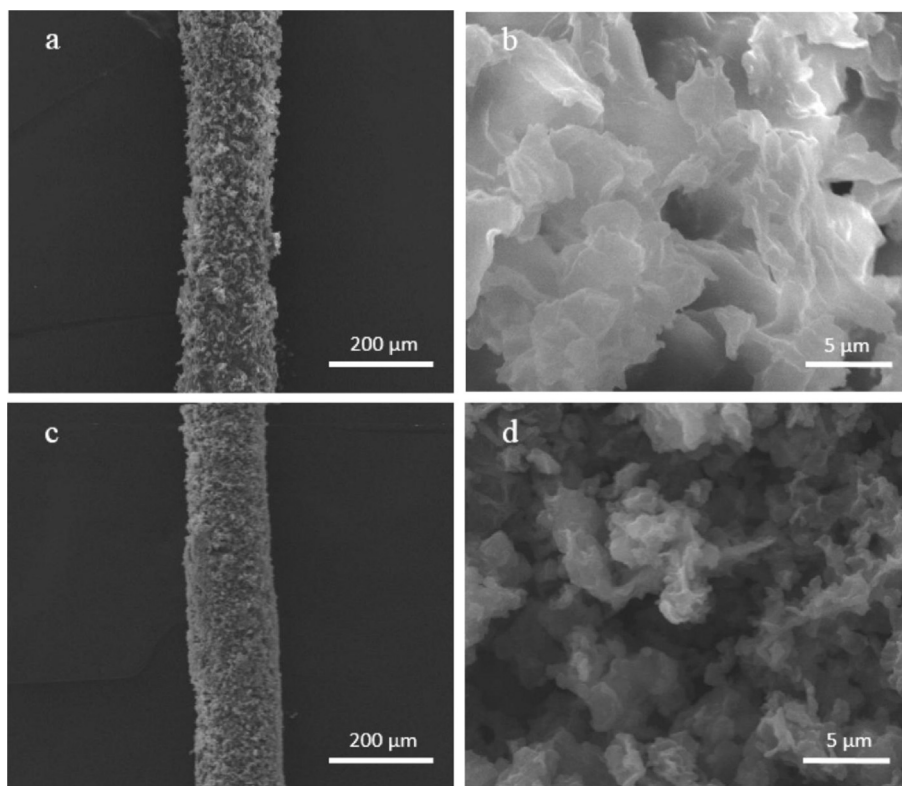


Fig. 4. SEM images (a,b) of GO and (c,d) HPGF fibers (Reprinted from Ref. [133] with permission from Elsevier).

tube or bonded onto the inner wall of a capillary by covalent or non-covalent interactions. Several studies have been performed on new materials as extraction phase for IT-SPME including monoliths [254], MIP [255], G and GO [256] and CNTs [257]. These materials can be improved the extraction efficiency, selectivity, and sensitivity of the analytical methods. Among these materials, G and its derivatives have received special attention in the last years. The π - π electrons system in G and its derivatives provide a good affinity for compounds with aromatic rings in pesticide structures. However, some problems can arise during the use of these materials as a sorbent in packed extraction techniques such as IT-SPME. For example, the high surface area of G or GO nano-sheets increases the extraction pressure and expels the sorbent from the frits. Lencas et al. [154] developed an optimized method to analyze three of the most common triazines in water samples by using GO covalently bonded to aminopropyl silica as sorbent. After this step, the extracts were transferred online to an HPLC-MS/MS for qualitative and quantitative determination. The method showed LOD of 0.0011 – $0.0029 \mu\text{g L}^{-1}$, presenting high sensitivity and good enrichment factor [154].

Huang et al. [156] developed an optimized method to analyze triazines in environmental water samples by using magnetism-enhanced monolith-based IT-SPME. Under the optimized conditions, this method showed adequate quantitative extraction efficiencies of the target analytes between 64.8% and 99.7%. The LOD and LOQ were in the ranges of 0.074 – $0.23 \mu\text{g L}^{-1}$ and 0.24 – $0.68 \mu\text{g L}^{-1}$, respectively. The proposed method applied for analysis of environmental water samples such as farmland, lake and river water with spiked recoveries in the range of 70.7–119% [156].

University of Oslo (UiO)-series MOFs are a class of zirconium-based building bricks possessing a large surface area, good thermal stability, and chemical resistance. UiO-66 is the most

well-known Zr-MOF, which is self-assembled from zirconium clusters $\text{Zr}_6\text{O}_4(\text{OH})_4$ and twelve terephthalate ligands, giving a three-dimensional arrangement of micropores with a high specific surface area and good water stability [258]. Fan et al. [150] proposed a new strategy for the application of UiO-66 for in-tube capillary monolith extraction. The optimized UiO-66-incorporated monolith had several advantages including improved permeability and faster mass transfer compared with the neat polymer monolith, preventing the loss of UiO-66 particles, and improved adsorption capacity for fungicides through π - π interactions, electrostatic forces, and hydrogen bonds. The UiO-66 monolith extraction coupled with LC-MS analysis was successfully applied for the simultaneous detection of five fungicides in pond water and soil samples. The developed method had low LOD (0.00134 – $0.0148 \mu\text{g L}^{-1}$), excellent repeatability, and high recoveries (90.4–97.5%) [150].

2.5. Stir bar sorptive extraction

The stir bar sorptive extraction (SBSE) is an equilibrium technique that was developed based on SPME in 1999 by Baltussen and co-workers [259]. It is characterized by higher adsorption capacity and recovery than SPME owing to the coating amount of stir bar 50–250 times higher than that of SPME fiber. Compared with SPME, a larger volume and surface area of coating used in SBSE show a higher extraction efficiency of target analytes and better reproducibility. Jen et al. [163] prepared a dumbbell-shaped magnetic stir-bar with sublimate sorbent for the SBSE of pesticides in an aqueous sample prior to GC- μ ECD. Cyclododecane (CDD) was coated onto a magnetic stir-bar surface as a sublimate adsorbent, and steel spheres were placed on both ends to make a dumbbell-shaped magnetic stir-bar for SBSE extraction. CDD is a nonpolar lipophilic organic solid at room temperature with a sublimation

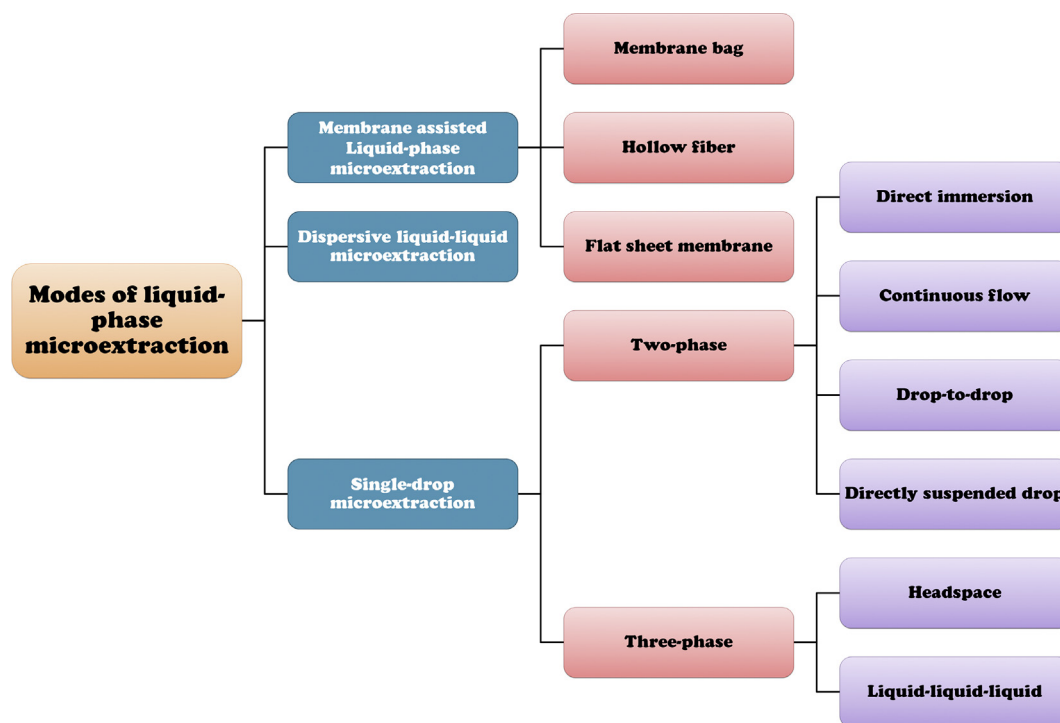


Fig. 5. Types of liquid-phase microextraction methods.

character. Due to its relatively slow evaporation in comparison with other volatile binding mediums and its purity, it does not leave any residue after sublimation [260]. The LR was $0.005\text{--}5\ \mu\text{g L}^{-1}$ with coefficients of determination ranging from 0.9950 to 0.9994. LOD of the method was $0.0004\text{--}0.0045\ \mu\text{g L}^{-1}$, and LOQ was $0.001\text{--}0.015\ \mu\text{g L}^{-1}$. The relative recoveries of the spiked samples were in the range of 83.2–98.7%. The proposed sublimation sorbent obtained superb enrichment factors (101–834) and provided a simple, sensitive, and eco-friendly sample preparation method.

2.6. Liquid-liquid extraction

Liquid-liquid extraction (LLE) is popular in sample preparation methods because of its simplicity, efficiency, and wide acceptance in many standard methods [261,262]. However, the LLE method is time-consuming, requires large amounts of toxic organic solvents, and offers low enrichment of analytes.

Salting-out assisted liquid-liquid extraction (SALLE) which coupling sample clean-up with enrichment (via salting-out extraction), is a technique based on LLE in which a proper concentration of salt is added to reach the separation of the aqueous phase from the partially miscible organic phase. This extraction method is an efficient preparation method for biological and high salinity samples [263].

Wen et al. [172] presented a SALLE combined with GC-MS method for the determination of four pyrethroid insecticides in high salinity and biological samples. It avoided the need for the elimination of salinity and protein in the sample matrix, as well as the clean-up of the SALLE extractant. Low LOD ($1.5\text{--}60.6\ \mu\text{g L}^{-1}$), wide LR ($5\text{--}5000\ \mu\text{g L}^{-1}$) and high recovery (74–110%) were attained for all the compounds. This method was showed to be simple, rapid and environmental-friendly [172].

Another extraction method, namely homogeneous liquid-liquid extraction (HLL), uses a phase separation phenomenon in a homogeneous solution. One version of HLL is salting-out homogeneous liquid-liquid extraction (SHLL) which has been used for

extraction and preconcentration of the selected analytes from aqueous samples [264].

Farajzadeh et al. [169] developed a new version of SHLL based on counter-current mode combined with DLLME for the extraction and preconcentration of some pesticides from aqueous samples and their determination by GC-FID. Under the optimum extraction conditions, the method exhibited broad LR for the target analytes. Enrichment factors and LOD for the selected pesticides were obtained in the ranges of 3480–3800 and $0.1\text{--}5\ \mu\text{g L}^{-1}$, respectively. The recoveries of analytes were in the range of 91–105% [169].

2.7. Liquid-phase microextraction

Liquid-phase microextraction (LPME) method has appeared as a strong technique for pre-concentration and matrix separation in the last decade, featuring simple operation, low cost, and high efficiency. In terms of the operation mode, LPME can be classified into three types, including single-drop microextraction (SDME), DLLME and hollow fiber (HF)-LPME [265]. Fig. 5 shows the classification of LPME based on their extraction modes.

Supramolecular solvents (SUPRASs) are water-immiscible nanostructured liquids composed of three-dimensional amphiphilic aggregates [266]. Due to their excellent physical and chemical properties, SUPRASs is a good alternative to organic solvents in the LPME method. These solvents provide a number of different interactions like ionic, hydrogen bonding, π -cation and hydrophobic with analytes in order to increase the extraction efficiency. A second major characteristic of SUPRASs is their high amphiphilic content. This property results in a high number of accessible binding sites for the extraction of analytes using low extractant volumes. Moreover, they are environmentally friendly solvents formed from inexpensive biosurfactants, in which the coacervation happens rapidly at room temperature. Shamsipur et al. [178] used a SUPRAS/oleic acid-coated magnetic particle system for the simultaneous LPME and determination of organophosphorus pesticide residues in water and fruit juice samples. Under the optimum experimental conditions, the

calibration curves reported being linear in the range of 0.5–400 $\mu\text{g L}^{-1}$ with correlation coefficients ranging from 0.9967 to 0.9984. The LOD ranged from 0.1 to 0.35 $\mu\text{g L}^{-1}$ [178].

SDME was presented in 1996 and was the first performance of such a miniaturized solvent-based extraction procedure for analytes from aqueous and gaseous samples. This method is simple, inexpensive, easy to operate, versatile, and has additional qualities like different extraction modes, high enrichment factor and can be fully automated [265]. Liu et al. [176] developed the SDME method using a nanofluid obtained by dispersing ZnO NPs in 1-hexyl-3-methylimidazoliumhexafluorophosphate for the simultaneous extraction of three fungicides (chlorothalonil, kresoxim-methyl, and famoxadone) in water samples prior to their analysis by HPLC. Under the optimized conditions, the LODs were in the range of 0.13–0.19 $\mu\text{g L}^{-1}$. This method was effectively applied to determine the three fungicides in real water samples including lake water and river water with recoveries in the range of 74.94–96.11% [176].

In 2007, Yamini et al. [267] developed a new mode of LPME termed as solidified floating organic drop microextraction (SFODME), which was then successfully applied for polycyclic aromatic hydrocarbons analysis in water samples. This technique is similar to SDME, but a specific holder is not required for supporting the organic micro drop and the sample solution can be agitated at high speed. SFODME has advantages such as simplicity, inexpensive, minimum organic solvent usages, and high enrichment factor. Hu et al. [179] proposed a method based on SFODME with vortex-assisted (VA) emulsification for the preconcentration of OPPs in water samples, followed by GC with flame photometric detection (FPD). The LODs were found to be in the range of 0.12–0.56 $\mu\text{g L}^{-1}$ for the five target OPPs, and the LR was from 0.6 to 100 $\mu\text{g/L}$. The proposed method applied to the analysis of five OPPs in East Lake water, well water and rainwater samples, and the recoveries for spiked samples were in the range of 83.7–112% [179].

2.8. Dispersive liquid-liquid microextraction

DLLME, first described in 2006, has been successfully applied for the extraction of pesticides from water samples. The fundamental rule of DLLME is the dispersion of an extraction solvent (immiscible with water) and a disperser solvent (miscible in water and extraction solvent) in an aqueous solution that supplies a greater contact area between the aqueous phase and the extraction solvent [268]. The major advantages of DLLME upon conventional techniques are simple operation, quick, low cost, easy handling, low usage of organic solvents, high recovery, high factor enrichment and adaptability with chromatographic techniques such as LC and GC [269]. Farajzadeh et al. [203] proposed an improved DLLME method for the extraction and pre-concentration of OPPs from various samples such as surface water, fruit juices, and vegetable matrices. Neutral and polar molecules such as polyols dissolve quickly in water because of the stabilizing effect of hydrogen bonds formed between hydroxyl groups of polyols and water molecules. When a polyol is added into an aqueous phase containing organic analytes, some original hydrogen bonds are substituted by new hydrogen bonds formed between polyol and water molecules. Thus, the analytes molecules release water and then are extracted into an extraction solvent. The experimental results indicated that this extraction technique coupled with GC-nitrogen phosphorus detector (NPD) exhibits much eligibility such as superb extraction performance, higher efficiency extraction, more sensitivity and good repeatability. In view of these advantages, the expanded process is a high-efficiency pre-concentration technique for the monitoring of the ultra-trace level of OPPs in aqueous [203].

In 2009, a modification of traditional DLLME, called dispersive liquid-liquid microextraction with solidification of floating organic

drop (DLLME-SFO) was proposed for the extraction of OCPs from water samples [270]. It involves the use of extraction solvents with lower density than water and a melting point near room temperature (in the range 10–30°C) that is readily solidified at low temperatures. In DLLME-SFO, the mixture of extraction solvent and disperser solvent is rapidly injected into the sample solution. After injection, centrifugation is applied to the flotation of the organic phase at the top of the aqueous phase. This is followed by cooling in a beaker containing crushed ice to ensure the solidification of the floated organic droplet. This approach resolves the method of drawing off an extraction phase lighter than water from the surface of the aqueous phase [271]. Primel et al. [191] developed a simple, rapid, and sensitive method for the determination of atrazine, simazine, cyproconazole, tebuconazole and epoxiconazole in mineral water employing the DLLME-SFO with determination by LC-MS. The LOQ of the method was 0.0125–1.25 $\mu\text{g L}^{-1}$. The recoveries ranged from 60 to 120% for 84% of pesticides [191].

In situ IL-DLLME combined ultra-small Fe_3O_4 magnetic nanoparticles was developed by Cao et al. [182] to detect pyrethroid pesticides in water samples. The method was successively applied to the determination of pyrethroid pesticides in different kinds of water samples with the LOD ranged from 0.16 to 0.21 $\mu\text{g L}^{-1}$. The offered method is actually nanometer-level microextraction (average size 80 nm) with the advantages of simplicity, and sensitivity [182].

Effervescence is a novel assisted method for simplifying the dispersion of solvents or adsorbents. The mechanism is that it produces carbon dioxide by the reaction of acid and carbonate/bicarbonate. Compared with conventional methods such as ultrasound and vortex, it is a simple, efficient and energy-free extraction technique [272]. Zhou et al. [193] proposed a sensitive method, named as effervescence assisted dispersive liquid-liquid microextraction based on the cohesive floating organic drop (EA-DLLME-CFO). It was examined for the analysis of triazine herbicides and triazole fungicides in three environment water samples (tap, river, and reservoir) and grape juice. Under the optimum conditions, the proposed method showed good LR within the range of 0.05–10 $\mu\text{g L}^{-1}$ with correlation coefficients of 0.9987–0.9999. The recoveries of analytes were in the range of 72.4–101.5% with RSD ranging from 2.6% to 11.7%. The LOD was varied from 0.0027 to 0.0097 $\mu\text{g L}^{-1}$. This method has been successfully used to simultaneously analyze triazine herbicides and triazole fungicides in surface waters and grape juice [193]. Gentili et al. [212] developed a procedure based on the use of a low transition temperature mixture (LTTM) for the DLLME of fungicides, insecticides, and acaricides from surface waters. The LTTM preparation involves the heat-mixing of choline chloride and acetylsalicylic acid in a molar ratio of 1:2 ($\text{ChCl}(\text{ASA})_2$). Under optimum conditions, the LODs and LOQs of the method were 0.002–2.3 and 0.006–7.7 $\mu\text{g L}^{-1}$.

2.9. Coupling techniques

Some authors recommended the coupling between SPE and DLLME as an approach to achieve larger volumes of water samples (more than 100 mL) and to be able to collect the analytes in very small amounts of extraction solvent. The association between these two methods was first offered by Fattahi et al. [273] who developed a method for the extraction and pre-concentration of chlorophenols from water samples. The same approach was used by Montes et al. to perform the determination of fungicides in wine samples, with LOD in the low $\mu\text{g L}^{-1}$ range [274]. The SPE-DLLME procedure offered the advantages of both methods such as simplicity, low solvent usage and exposure, high recovery and enrichment factor, excellent extraction efficiency and desirable purification ability, which dramatically improved the environmental sustainability in complex matrixes. In this method, the

Table 2

Comparison of the extraction methods.

Extraction method	Advantages	Disadvantages	Suggestions
LLE	1. Simple and inexpensive equipment	1. Low selectivity 2. Labor intensive 3. High consumption of organic solvents 4. Production of toxic vapors 5. Multi-step extraction process 6. Low concentration factor 7. Time-consuming 8. Possible formation of emulsion	1. Tune the solvent polarity 2. Use of microextraction methods 3. Use of green solvents 4. Use of bed sorbents
SPE	1. Larger enrichment factor compared to LLE 2. High recoveries 3. Decreased volume of organic solvent 4. Increased extraction efficiency 5. No problems with emulsion formation 6. Good repeatability 7. Automation	1. Poor reproducibility of isolation and enrichment 2. Clogging of sorbent bed (both in SPE tubes and extraction disks) by suspended matter present in a sample 3. Memory effect 4. Expensive and commercially limited cartridges	1. Knowledge of absorbent and samples structure 2. Optimization of effective parameters 3. Use of bio/green/disposable adsorbent
dSPE	1. Fast 2. Inexpensive 3. Easy to use 4. High extraction efficiency 5. High selectivity	1. Need to filtration or centrifugation	1. Use of magnetic adsorbent
MSPE	1. Fast 2. Inexpensive 3. No centrifugation 4. The convenience of preparation and surface modification of the MNPs (high selectivity) 5. High extraction efficiency 6. Good reusability	1. Inadequate reproducibility	1. Optimization of effective parameters
SPME	1. Short analysis time 2. Automation 3. Low analyte losses 4. Easy to use 5. Possibility of implementing an analytical procedure on-line 6. Possibility of using liquid, gaseous and solid samples 7. High sensitivity 8. No sample pretreatment (e.g. centrifugation or filtering) 9. Solvent-free 10. Linear results for a wide range of concentrations of analytes 11. Miniaturization 12. Highly consistent, quantifiable results from very low concentrations of analytes	1. Relatively expensive (fiber cost) 2. The limited-time of fiber use 3. Matrix effects 4. Fiber damage 5. The commercial fibers are fragile 6. Instability of the fibers: breaking and stripping of coatings	1. Use of sol-gel based coatings 2. Use of resistant fibers (such as metal substrates)
SBSE	1. Stirs and extracts in one step 2. High recovery 3. Low detection limits 4. More sensitivity than SPME	1. Difficulties in removing the stir-bar from the samples and its rinsing 2. Desorption requires several steps	1. Cold trapping or preconcentration
HF-LPME	1. Simple 2. Low cost 3. Good preconcentration factor 4. Good recovery 5. High sample cleanup	1. Memory effect 2. Need to membrane preconditioning 3. Long extraction time 4. Possibility of fiber pores getting blocked	1. Use of electromembrane extraction
DLLME	1. High recovery 2. Simple 3. Low cost 4. Easy operation 5. Fast extraction kinetics 6. High enrichment factors 7. Good absolute recovery 8. Requires the use of a small amount of sample and organic solvents	1. Low selectivity 2. Requires the use of three solvents 3. Limited solvent choice 4. Requires centrifugation 5. Not appropriate for samples with a complex matrix composition 6. Post-extraction steps such as liquid-liquid back-extraction are often required for applying in complex matrices 7. Collecting difficulty of the acceptor phase in complex matrices 8. Average precision	1. Select the proper extraction and dispersion solvents 2. Use of ILs or DES solvents
QuEChERS	1. No chlorinated solvents included 2. Covering a wide range of pesticides 3. Simple instrumentation 4. Flexible and effective	1. Low enrichment factors 2. Requires centrifugation	1. Use of preconcentration methods

dispersion solvent used in DLLME is used as the elution solvent in SPE with the advantage of being water-soluble and consequently, it is not necessary to dry totally the SPE column before analyte elution. The water will be removed from the column together with the analytes and be removed in the subsequent phase separation during the DLLME step.

Salemi et al. [219] developed a micro-SPE-DLLME for the determination of OCPs in water samples, using GC-ECD. The extraction has been optimized with three different solid sorbents: MWCNT, polymeric Lichrolut® EN, and silica-based Lichrolut® C18. The LOD of the optimized technique was less than $0.26 \mu\text{g L}^{-1}$. The calibration functions were linear up to $500 \mu\text{g L}^{-1}$ with R^2 values of more than 0.995, for all sorbents. The figures of merit of the procedure showed that each of the studied sorbents was suitable [219].

Hou et al. [222] applied an efficient magnetic MOF composite as a sorbent for MSPE coupled with the DLLME-SFO method for the determination of trace pyrethroids in environmental water and tea samples. Target analytes were quantified by GC-ECD. The linear range was attained in the range of $0.05\text{--}10.0 \mu\text{g L}^{-1}$. Excellent sensitivity levels were achieved with the LOD of $0.008\text{--}0.015 \mu\text{g L}^{-1}$ and the LOQ of $0.028\text{--}0.050 \mu\text{g L}^{-1}$. Under optimal conditions, this method was effectively established for the determination of pyrethroids in real samples with acceptable recoveries ranging from 78.3 to 103.6%. Generally, this method presented the advantages of remarkable sensitivity, ease of operation, rapidity, low cost and environmental friendliness [222].

2.10. Quick, easy, cheap, effective, rugged, and safe

Quick, Easy, Cheap, Effective, Rugged, and Safe (QuEChERS) was introduced for the first time by Anastassiades et al. [275]. QuEChERS procedure consists of a number of simple analytical steps that are fast, easy and do not require specific implementations. QuEChERS provides high recoveries for a broad range of pesticides belonging to numerous chemical classes. QuEChERS is widely used for the extraction of pesticide residues from fruits and vegetables, cereals, herbs and other foodstuff using 1:1 sample-solvent ratio [276]. However, employing the QuEChERS method in the preparation of water samples has been reported by few researchers. Ghani et al. [224] developed a miniaturized, QuEChERS based, LLE method followed by GC-MS for pesticide residues determination in water. The recovery ranged from 85.3 to 107% with RSD ranging from 1.8 to 15.4%. The LR showed a reliable range (0.995–0.999) and the LOD ranged from 0.3 to $4 \mu\text{g L}^{-1}$.

3. Comparison of extraction methods

The various extraction methods have been used for the pre-concentration and extraction of pesticides in environmental samples. The main purpose of these methods is to reduce the analysis time and use of organic and toxic solvents. The advantages, disadvantages, and ways to overcome these disadvantages, for the different extraction methods, are summarized in Table 2.

4. Conclusions and future perspectives

In this review, we compiled and discussed a variety of pre-concentration and extraction methods of pesticides, with regard to sorbent type, since 2015. Pesticides are among the most dangerous environmental pollutants due to their mobility and long-term effects on living organisms. They typically remain in the aquatic environment at very low levels and in complex matrices for a long time. This creates problems for the analyst, such as spending extra time to prepare samples for chemical analysis, which can be a source of even more pollution. Because of the complexity of the

environmental samples, the clean-up steps of extracts are necessary before the final determination.

The ideal sample preparation method should compromise among cost, accuracy, selectivity, and sensitivity. Unfortunately, the traditional liquid solvent extractions frequently fail to meet these goals, being time-consuming, labor-intensive, complicated and expensive. They also produce considerable quantities of waste and provide insufficient LOD. Often, many physically and chemically different compounds need to be determined rather than one or a single class of analytes, and therefore it is necessary to develop sample preparation methods for the analysis of pesticide multi-residues in environmental samples. Other sample preparation methods, such as SPE, MSPE, SPME, and LPME might offer the extraction and clean-up in one step, which not only reduces the consumption of organic solvent and operation time but also dose simplify the experimental procedure and decreases the experimental errors. Driven by the advances in science and technology and the quest for accurate analytical results, the sample preparation methods are expected to continue developing rapidly.

Along with the request for new materials, novel nanomaterials have become the top materials, which offer an emerging field with great potentials for sample preparation. The main feature of nanomaterials as sorbents is their high surface area which provides higher sorption capacities than conventional microscale sorbents. Nanomaterials can strongly adsorb the analytes, so consequently, the desorption process should be slower and needs harsher conditions in comparison to microscale and bulk materials. However, the agglomeration leads to lessening the sorbent specific surface area.

According to the data in Table 1, it can be concluded that the most widely used methods are the SPE, MSPE, SPME, and DLLME. In addition, by selecting the adsorbent with proper structure, a lower detection limit can be obtained.

Nowadays, there are notable opportunities for the development of new extraction methods for the separation and enrichment of target compounds. Due to the importance of extraction steps in the analysis, designable structures and the ability to make hybrids of extraction media provide many opportunities to make a more effective extraction phase such as green adsorbents/solvents and the combination of different extraction techniques. On the other hand, more works should be done for the development of new extraction methods to attain complete automation and to enhance the preconcentration step. The future development of extraction methods has led to significant advances in in-vivo analysis, which allows the direct extraction of target analytes from the real water samples.

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