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Review

Molecularly Imprinted Polymers for Dispersive (Micro)Solid Phase Extraction: A Review

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Abstract: The review describes the development of batch solid phase extraction procedures based on dispersive (micro)solid phase extraction with molecularly imprinted polymers (MIPs) and magnetic MIPs (MMIPs). Advantages and disadvantages of the various MIPs for dispersive solid phase extraction and dispersive (micro)solid phase extraction are discussed. In addition, an effort has also been made to condense the information regarding MMIPs since there are a great variety of supports (magnetite and magnetite composites with carbon nanotubes, graphene oxide, or organic metal framework) and magnetite surface functionalization mechanisms for enhancing MIP synthesis, including reversible addition-fragmentation chain-transfer (RAFT) polymerization. Finally, drawbacks and future prospects for improving molecularly imprinted (micro)solid phase extraction (MIMSPE) are also appraised.

Keywords: molecularly imprinted polymers; magnetic molecularly imprinted polymers; dispersive (micro)solid phase extraction

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1. Introduction

During the last two decades the large development of analytical instrumentation, mainly the introduction of mass spectrometry (MS) and tandem mass spectrometry (MS/MS), has facilitated the determination of analytes in biological, food, and environmental samples at trace concentrations. However, although the high sensitivity provided by the instrumentation and the direct injection/analysis of crude samples/extracts are not always possible, new sample preparation strategies are needed for potential interferences removal and analyte pre-concentration, for increasing the robustness and repeatability of measurements, for converting the analyte to a more suitable form for separation/detection, and also for avoiding conventional multiple-step pre-treatment methods [1]. Several extraction/pre-concentration techniques have been therefore developed and among those techniques, solid phase extraction (SPE) and solid phase microextraction (SPME) are nowadays well established and commercially available methodologies. However, the main drawback associated with them is the moderate selectivity of sorbents, which can require further extract clean-up stages [2].

Molecularly imprinted polymers (MIPs) are versatile materials that mimic natural antigen–antibody mechanisms and allow molecules/analytes recognition [2,3]. These materials are prepared by polymerizing monomers and cross-linkers around the template molecules, leading to a highly cross-linked three-dimensional network polymer. After polymerization, the template molecules are removed, and the shape and size of the binding sites are established complementary to the target analyte. Synthesized MIPs are stable and show resistance to wide range of pH values, temperatures, and solvents and interact with target molecule in a selective way. Due to their practical features, MIPs have been used as selective sorbents for (micro)solid extraction (μ-SPE) procedures leading to

molecularly imprinted (micro)solid extraction (MIMSPE), which allows advanced miniaturized sample pre-treatments for green procedures in Analytical Chemistry. This review intends to provide a snapshot of current state-of-art use of MIMSPE in sample preparation, describing several batch MIMSPE approaches such as membrane-protected molecularly imprinted polymers and dispersive (micro)solid phase extraction with magnetic MIPs and non-magnetic MIPs. In addition to several reviews regarding MIPs [2–4] and magnetic MIPs [5] as selective adsorbents for SPE, some other published reviews have focused on the applications of MIP-based adsorbents for drug analysis [6], for assessing pollutants, and in food [7] and environmental samples [7,8]. Benefits of dispersive solid phase extraction (dSPE) and dispersive microsolid phase extraction (D-μ-SPE) have led to several applications based on the use of quite different adsorbents for pre-concentration and/or clean-up purposes, and some procedures have consisted of using MIPs as adsorbents [9–11]. This review intends to provide a snapshot of current state-of-art use of MIPs (magnetic and non-magnetic composites) for dSPE and D-μ-SPE. Information inherent to the preparation of the MIP composites, mainly the magnetic core functionalization, is shown and new trends for surface functionalization, such as the use of boronic acids, are highlighted.

2. Dispersive (Micro)Solid Phase Extraction with MIPs

As shown in Figure 1, dSPE and D-μ-SPE [9–12] procedures consist of dispersing the adsorbent (a few milligrams or a very few milligrams) into the sample/extract by shaking (oscillators and vortex) and by applying ultrasounds, and, for magnetic adsorbents, by magnetic stirring [4]. Dispersion enhances target adsorption on the adsorbent (nano)microparticles, and the use of ultrasound and mechanical shaking (mainly vortex) favors adsorbent dis-aggregation and maximizes the surface area of the adsorbent particles. Vortex stirring is a soft and low-cost shaking technique and dispersion assistance is more repeatable when compared with ultrasounds because of the ultrasound fluency dependence on the position inside the water-bath tank [11]. Vortex assistance also prevents analyte degradation and adsorbent aggregation, although the technique offers lower extraction kinetics when compared to ultrasounds dispersion [13–15] (in fact, some reports have stated that ultrasounds change the absorption kinetics [16–18]).

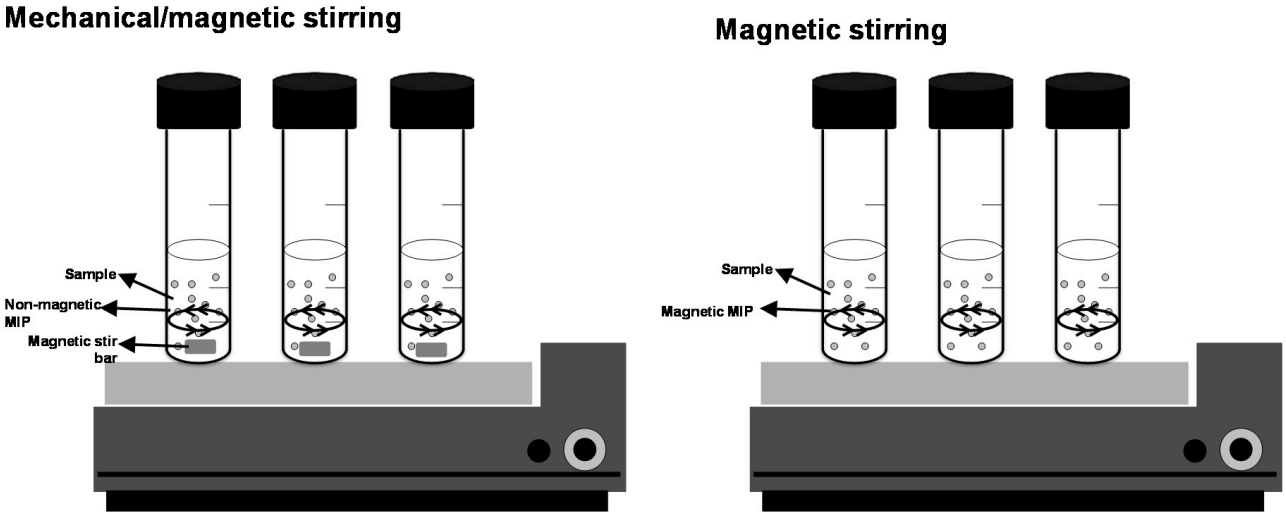

Figure 1. *Cont.*

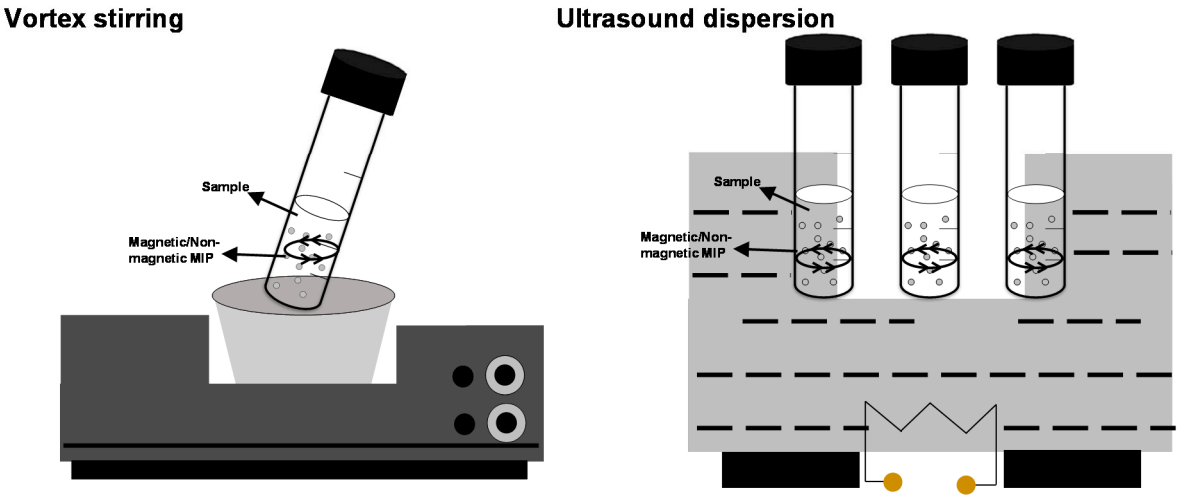

Figure 1. Schematic representation of dispersive solid phase extraction/dispersive (micro)solid phase dispersion (dSPE)/(D-μ-SPE) procedures with magnetic and non-magnetic molecularly imprinted polymer (MIPs).

2.1. Dispersive (Micro)Solid Phase Extraction with Magnetic Molecularly Imprinted Polymers (MMIPs)

MMIP beads were first introduced by Ansell and Mosbach in 1998 as a core–shell structure (magnetic iron oxide, magnetite, Fe_3O_4) for performing drug radioligand binding assays [19]. Then, MMIPs (magnetic nickel hexacyanoferrate, NiHCF, nanoparticles coated with a molecularly imprinted polymer for the herbicide chlorotoluron) were proposed for preparing selective modified electrodes [20]. MMIPs as selective adsorbents for SPE procedures offer advantages such as avoidance of drawbacks associated with conventional batch SPE/μ-SPE procedures, which need filtration/centrifugation steps for separating the adsorbent from the bulk sample after the loading stage and from the extract after analyte elution. In addition, losses of adsorbent particles are minimized since adsorbent separation is easily and quickly achieved by applying a magnet [21]. As previously mentioned, MMIP nanoparticles can be stirred (dispersed) in the sample/extract (loading step) and in the eluting solution (elution step), taking advantage of their magnetic properties, but stirring can be also performed by vortexing and by ultrasound dispersion.

There are several strategies for preparing MMIPs, which lead to a great varietyof magnetic adsorbents. Moreover, despite free radical polymerization mechanism(s), which are mainly used to prepare MMIPs (and also MIPs), the heterogeneity caused by the fast chain propagation and irreversible termination reactions has led to the use of controlled radical polymerization strategies such as reversible addition fragmentation chain-transfer (RAFT) polymerization for preparing MIPs [22] and also MIP coatings over magnetic and non-magnetic supports [23–26]. RAFT polymerization provides more accessible sites for target adsorption and faster mass transfer because of the more homogenous polymeric network [27].

2.1.1. Classification of MMIPs

Based on MMIP structure, four types of MMIPs can be established: core–shell MMIPs, magnetic nanotube-supported MIPs, magnetic nanosheet-supported MIPs, and magnetic hollow porous MIPs [28].

A core–shell structure is the most widely used, and it consists of a core magnetic phase (typically magnetite) and a polymeric phase shell [29]. Magnetic nanoparticles (Fe_3O_4) in the core–shell-based structures offer a high surface area for MIP anchorage, and the surface can be also modified (activated/functionalized) with hydroxyl groups and a SiO_2 layer to protect the core from oxidation or dissolution [30].

Magnetic nanotube-supported MIPs imply the presence of carbon nanotubes (CNTs) or multi-walled carbon nanotubes (MWCNTs) in the reaction medium during the co-

precipitation and solvothermal synthesis of Fe_3O_4 to prepare magnetic carbon nanotubes (MCNTs) in which the magnetic nanoparticles are linked onto the CNTs' surface [31]. The prepared MCNTs are then treated with a silica-based reagent to cover them with a SiO_2 layer (MCNTs–SiO_2) before MIP synthesis. Because of the large surface area of the CNTs, the prepared MCNTs–SiO_2 structures offer a higher specific surface area for MIP anchorage than that found in core–shell MMIPs, leading to larger binding/recognition sites for the target. On other occasions, MWCNTs, previously functionalized with carboxylic acid groups (COOH), are mixed with Fe_3O_4 nanoparticles in the pre-polymerization solution for direct MIP synthesis [32].

Similar to CNTs, the use of graphene oxide (GO) during the solvothermal synthesis of Fe_3O_4 leads to a GO–Fe_3O_4 composite in which the magnetite nanoparticles are linked to the GO nanosheet [33,34]. MIP synthesis can be then performed after grafting the Fe_3O_4@GO surface with acrylic acid, and the resulting magnetic nanosheet-supported MIP provides high specific surface area and high affinity for the target molecule, as well as an extremely fast absorption rate [33]. On other occasions, the magnetic Fe_3O_4@GO composite can be functionalized with silica and vinyled reagents before MIP synthesis [34]. Other approaches are based on activated CNTs (presence of carboxyl groups absorbed onto the surface of GO through π–π attractions after acidic treatment) which, after hydrothermal treatment for synthesizing Fe_3O_4 nanoparticles, lead to 3D magnetic GO-CNT composites [35].

Finally, well-designed magnetic hollow porous MIPs (magnetic HPMIPs) have been introduced as a sacrificial support in the molecular imprinting process. As reported, MIP synthesis is performed on the internal surface of mesoporous silica spheres (referred as MCM-48) followed by silica and template removal (typically hydrofluoric acid/ethanol mixtures) [36–39]. Previous to magnetite synthesis over the HPMIPs (co-precipitation method), a treatment with diluted perchloric acid was required to obtain 1,2-diol groups over the HPMIPs structures [37].

In addition to mesoporous silica, mesoporous carbon has also been found to be an excellent support for preparing hollow porous MIPs, with the advantage that carbon support is not removed (sacrificed) to obtain the required porosity of the material. D-glucose [40] and raw Pericarpium Granati (a medicinal plant) [41] have been used as sources of carbon for the synthesis of the magnetic mesoporous carbon (MMC) particles by hydrothermal methods (high temperatures as well as long synthesis times) in the presence of ferric and ferrous ions. Benefits of the hollow composites are the presence of high dense accessible recognition sites for molecular imprinting, and a high absorption capacity, which leads to higher enrichment factors when compared with traditional MIPs.

Hollow porous MIPs have been also designed by Fe_3O_4 nanoparticle surface modification by a sol–gel route with silica-based reagents such as tetraethyl orthosilicate (TEOS), which promotes hydroxyl groups on the surface of the magnetic nanoparticles, followed by MIP synthesis, and template and silica layer removal [42–44].

Other magnetic HPMIPs have been synthesized by using hollow Fe_3O_4 microspheres instead of conventional magnetite [45] (hollow Fe_3O_4 microspheres are obtained by one-pot hydrothermal methods [46]). In addition, other authors have prepared magnetic nanorings with abundant epoxy groups on the surface for imprinting purposes involving ring-opening reactions. The prepared material, named core–shell nanoring amino-functionalized magnetic molecularly imprinted polymer (CS-NR-Mag-MIP), was found to offer high absorption capacities for bisphenol A [47] and sulfonamides [48].

2.1.2. Magnetite Surface Functionalization for Core–Shell MMIPs

There are some procedures for MMIP preparation in which Fe_3O_4 nanoparticles are present in the polymerization mixture during MIP synthesis [49–51]. Although transmission electron microscopy (TEM) images show that the spherical Fe_3O_4 nanoparticles are well enwrapped by the MIP shell [51], on other occasions the used of un-functionalized Fe_3O_4 nanoparticles during MIP synthesis leads to uniform polymeric layer composites [50].

Dispersed spherical (nano)particles are preferred as adsorbents in SPE, and the spherical shape of a magnetite-based composite is guaranteed by performing MIP synthesis over surface functionalized magnetite. In addition, once Fe_3O_4 nanoparticles are synthesized (magnetite is also commercially available), functionalization of the nanoparticles' surface makes it favorable for MIP adhesion, and also promotes a high specific surface area and improves polarity [52]. Although magnetite surface functionalization can be performed after Fe_3O_4 synthesis or directly over commercial nanoparticles, one-step Fe_3O_4 synthesis and surface functionalization procedures have been also described. After surface modification, polymerization can be performed by several polymerization methods using the adequate template molecule, monomer, cross-linker, initiator, and porogen. The resulting composite adsorbent will offer good selectivity/recognition for the target molecule as well as good magnetic properties [52].

Magnetite surface functionalization can be performed mainly by using silica-based, diol-based, and vinyled compounds (Table 1). However, there are other functionalization mechanisms as well as several combinations of surface modifier reagents for Fe_3O_4 nanoparticle surface functionalization.

Table 1. Functionalization reagents for magnetite core–shell magnetic molecularly imprinted polymers (MMIPs).

Fe_3O_4@OH Functionalization	
Diol-based reagents	Ref.
Polyethylene glycol (PEG)	[53–58]
Poly(vinyl alcohol)	[59]
Acrylic acid	[60]
Methacrylic acid (MAA)	[61]
Boronic acids:	
2,4-Difluoro-3-formyl-phenylboronic acid (DFFPBA) [a,b]	[62,63]
4-Formylphenylboronic acid (FPBA) plus sodium cyanoborohydride ($NaBH_3CN$)	[64,65]
4-Vinylphenboronic acid (VPBA) [c]	[66]
3-Aminophenylboronic acid (APBA) [d]	[67]
Silica-based reagents	
Tetraethyl orthosilicate (TEOS)	[52,58,68–90]
Fe_3O_4@CH=C_2H_4 functionalization	
Oleic acid (OA)	[91–102]
Silica-based reagents:	
3-(Trimethoxysilyl) propyl methacrylate (TMSMA)	[103]
3-Methacryloxypropyltrimethoxysilane (MPS or KH-570)	[27,71,72,75–77,80–84,90,104–114]
Vinyl trimethoxy silane (VTMOS)	[72]
Vinyl triethoxy silane (VTEO or VTES)	[115–118]
Fe_3O_4@NH_2 functionalization	
Silica-based reagents:	
(3-Aminopropyl)triethoxysilane (APTES)	[78,105,119–124]
Methacryloyl chloride	[125]
Fe_3O_4@COOH functionalization	
Silica-based reagents	
Poly(ethylene glycol)bis(carboxymethyl) ether [e]	[123]
Fe_3O_4@X, *X= Cl or Br* functionalization	
Silica-based reagents	
4-Chloromethyl phenyl trichlorosilane (4-CPS) [f]	[74,77,126–131]
3-Bromopropyl trimethoxy silane (BPTS)	[132]

([a]) Fe_3O_4 functionalized with 1,6-hexanediamine to give Fe_3O_4@NH_2; ([b]) Fe_3O_4 functionalized with TEOS and APTES to give Fe_3O_4@SiO_2; ([c]) Fe_3O_4@pTiO_2 functionalized with γ-mercaptopropyltrimethoxysilane (γ-MPTS) to give Fe_3O_4@pTiO_2@SH; ([d]) Fe_3O_4@MCM-48 (mesoporous silica spheres) composite; ([e]) Fe_3O_4 functionalized with TEOS and APTES to give Fe_3O_4@NH_2; ([f]) Fe_3O_4 functionalized with TEOS to give Fe_3O_4@OH.

Surface Functionalization with Hydroxyl (Diol) and Vinyl-Based Reagents

Diol-based reagents such as polyethylene glycol (PEG) [53–57] interact with the nanoparticle surface through one of the hydroxyl groups, allowing the remaining hydroxyl groups to be available to react with the components of the pre-polymerization mixture (Figure 2). A similar mechanism is obtained for oleic acid [91–102], which interacts with the nanoparticle's surface through the hydroxyl groups but promotes the presence of vinyl groups in the modification layer. Similarly, poly (vinyl alcohol) [59] is also a source of hydroxyl and vinyl groups for reacting with the pre-polymerization components. A magnetic core surface rich in vinyl groups can be also obtained by treating the prepared Fe_3O_4 nanoparticles with acrylic acid [60] or by one-step co-precipitation of Fe_3O_4 in the presence of methacrylic acid [61].

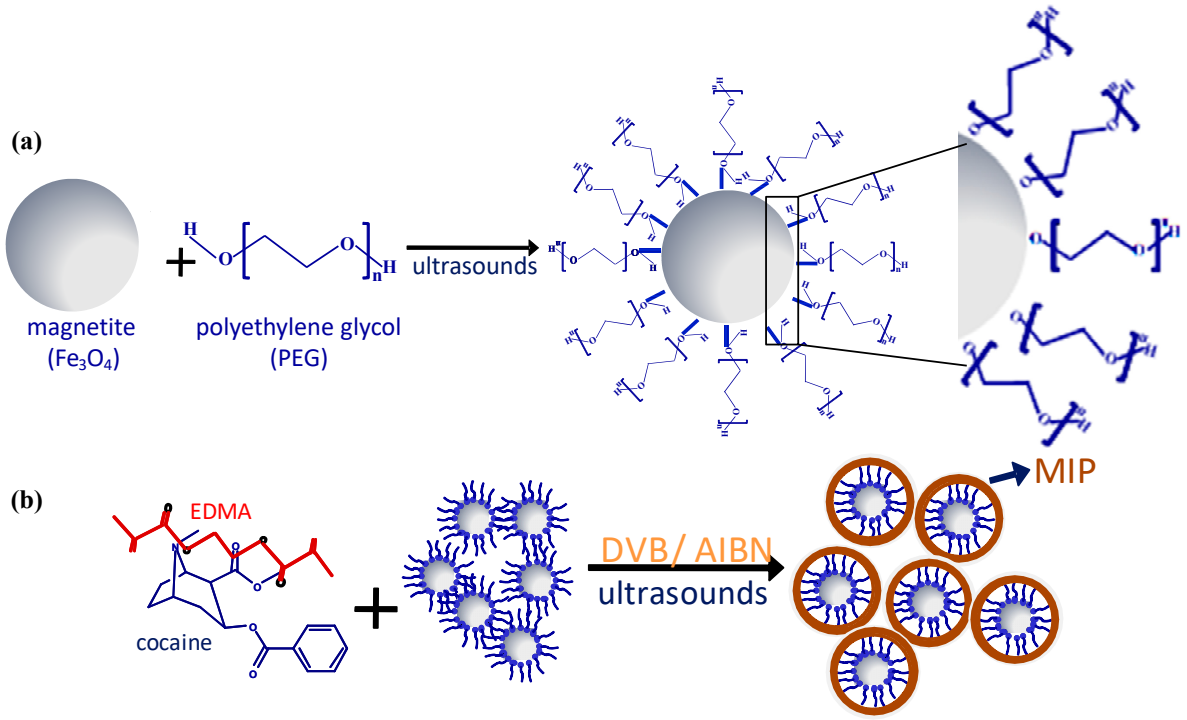

Figure 2. Schematic representation of binding mechanism between magnetite nanoparticles and polyethylene glycol (PEG) (**a**) and MMIP preparation (**b**), Adapted with permission from Ref. [55]. Copyright 2016 Elsevier.

The main advantage of using these reagents for magnetite surface modification is the simplicity of the procedure and the moderate operating conditions (room temperature or ice-bath). In addition, surface functionalization with these reagents avoids the electrostatic agglomeration of magnetite, which ensures the uniformity of magnetic nanoparticles in the pre-polymerization solution and a further MIP homogeneous embedding. However, drastic conditions, such as extreme pHs, when removing the template after MMIP synthesis can damage the link between the nanoparticle and the functionalization layer, which leads to a separation of the MIP layer (shell) from the magnetite nanoparticles (core).

Similarly, the presence of hydroxyl groups on the nanoparticle surface can also be achieved by using boronic acids that bind *cis*-diol-containing compounds and result as adequate for imprinting large biomolecules, such as proteins, and achieving oriented surface imprinting, depending on the affinity between the template molecule and the boronate residues [62–67] (Table 1). The boronic acid 2,4-difluoro-3-formyl-phenylboronic acid (DFFPBA) has been proposed for preparing MMIPs, which requires amino-functionalized magnetic nanoparticles before DFFPBA functionalization (easily achieved by Fe_3O_4 synthesis in presence of 1,6-hexanediamine). Fe_3O_4@NH_2 can be directly treated with DFFPBA (treatment at room temperature for 24 h) [62] or can be first silanized with TEOS

and (3-Aminopropyl) triethoxysilane (APTES), leading to (Fe_3O_4@SiO_2@DFFPB) [63]. In addition to DFFPBA, other boronic acids such as 4-formylphenylboronic acid (FPBA) in combination with sodium cyanoborohydride ($NaBH_3CN$) have been found to be effective to prepare the surface of Fe_3O_4@NH_2 [64] or Fe_3O_4 nanoparticles [65] for synthesizing MIPs for protein recognition. A similar strategy has been proposed using magnetite microspheres coated with porous TiO_2 (flower-like structure Fe_3O_4@pTiO_2 nanoparticles) prepared via a solvothermal method and further functionalized with γ-mercaptopropyltrimethoxysilane (γ-MPTS) to promote the presence of –SH before anchoring the boronic acid 4-vinylphenboronic acid (VPBA) [66]. More binding sites for templates (horseradish peroxidase), and thus, higher adsorption capacity, were found when using Fe_3O_4@pTiO_2 as a supporting material than when using Fe_3O_4@SiO_2 cores [65,66]. In addition, the strong electron-withdrawing effects of Ti(IV) endow the boronic acid with lowered pK_a value that makes the Fe_3O_4@pTiO_2@MIPs capture glycoproteins under moderate acidic conditions [66].

Finally, boronate-affinity magnetic hollow molecularly imprinted polymer sorbents for sialic acid (a compound exhibiting a *cis*-diol structure) have been also prepared by using mesoporous silica spheres (MCM-48) as a sacrificial support, glycidyl methacrylate (GMA) as a co-monomer to chemisorb Fe_3O_4 nanoparticles, and 3-aminophenylboronic acid (APBA) as boronic acid [67]. After MCM-48@APBA preparation, the template, the cross-linker, and the initiator are added for performing MIP synthesis, followed by MCM-48 dissolution in a hydrofluoric/ethanol mixture (B-hMIP composite). Fe_3O_4 nanoparticles are then synthesized (co-precipitation) in the presence of the prepared composite (B-hMIPs), leading to the magnetic hollow adsorbent [67].

Surface Functionalization with Silica-Based Reagents

Silica-based reagents (Table 1) are an alternative to vinyled and diol-based compounds in magnetite surface functionalization procedures for overcoming problems derived from core–shell breakdown as consequence of extreme pH and temperature operating conditions, since the resulting composites exhibit great stability [52]. TEOS is a typical silica-based compound used for Fe_3O_4 modification at moderate operating conditions, resulting in Fe_3O_4@SiO_2 composites. The TEOS layer over the magnetite nanoparticles is a source of hydroxyl groups for further interactions with the pre-polymerization reagents [68–89]. A typical diagram of a magnetic silica-based composite is illustrated in Figure 3.

An improved degree of functionalization can be achieved by using silica-based reagents containing other functional groups such as vinyl, amino, and halide groups (Table 1). This is the case of reagents such as 3-(trimethoxysilyl)propyl methacrylate (TMSMA) [103], 3-methacryloxypropyltrimethoxysilane (MPS, also known as KH-570) [71,72,75–77,80–82,84,104–114], vinyl trimethoxy silane (VTMOS) [72], and vinyl triethoxy silane (VTEO or VTES) [115–118], which promote the presence of vinyl groups on the Fe_3O_4 surface or on the Fe_3O_4@SiO_2 surface when Fe_3O_4 nanoparticles are previously functionalized with TEOS (extra functional groups on the Fe_3O_4@SiO_2 surface allowing its interaction with vinyled silica reagents). Functionalization with vinyl-based silica reagents can also be performed after a previous oleic acid functionalization (Fe_3O_4@oleic acid via a co-precipitation technique) followed by modifying the surface of the nanoparticles with a silica layer (TEOS) and double bonds introduction onto the Fe_3O_4@SiO_2 with KH-570 [90].

The presence of amino ($-NH_2$) groups is guaranteed by using APTES (Table 1), and MIP synthesis can be performed directly by mixing the functionalized nanoparticles with the polymerization reagents [78,119]. On other occasions, the monomer, such as methacryloyl chloride, can be fixed to the functionalized silica layer after reaction with the immobilized amino groups [125]. In addition, previously modified Fe_3O_4 with TEOS (Fe_3O_4@SiO_2) can be then covered with APTES for promoting the presence of $-NH_2$ groups [105,120–123].

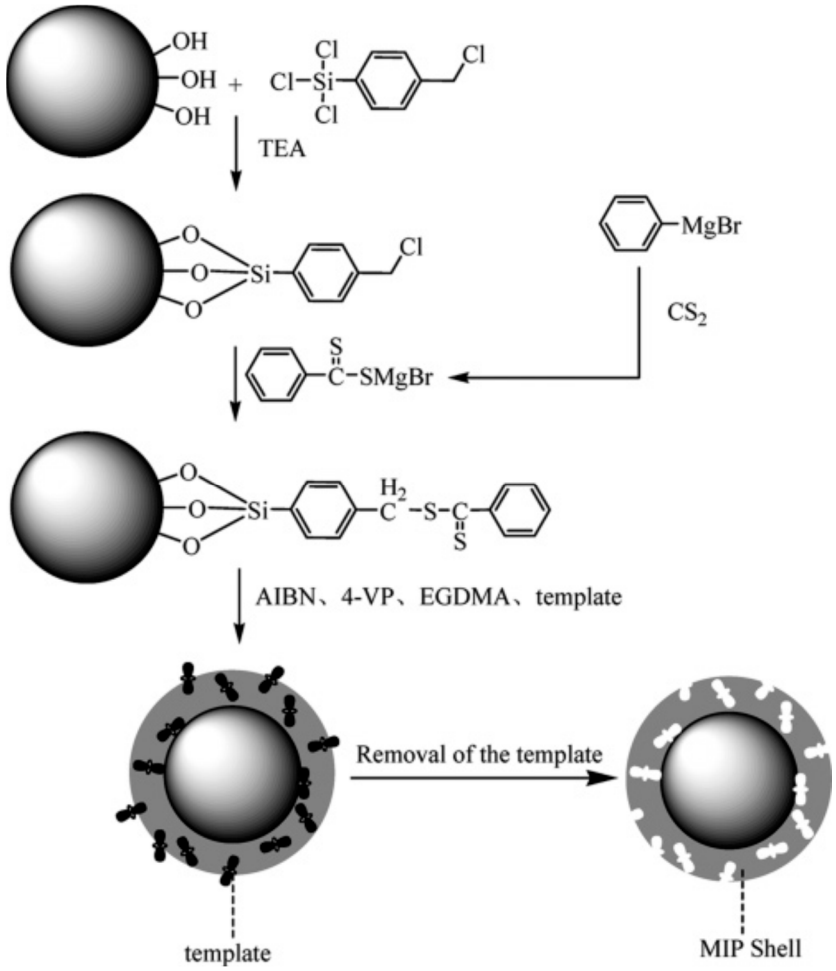

Figure 3. Schematic of the fixation of the reversible addition−fragmentation chain-transfer (RAFT) agent onto silica nanoparticles and the growth of the MIP shell from silica nanoparticles via surface RAFT polymerization, Adapted with permission from Ref. [79]. Copyright 2016 American Chemical Society.

Introduction of carboxyl groups onto the Fe_3O_4@SiO_2 surface is performed by treating functionalized Fe_3O_4@-SiO_2–NH_2 nanoparticles (TEOS and APTES covering) with poly(ethylene glycol)bis(carboxymethyl) ether before MIP synthesis [123]. Comparison between Fe_3O_4@SiO_2–COOH@MIP and Fe_3O_4@-SiO_2–NH_2@MIP showed that the latter had high specific surface area and fast mass transfer rate toward the target (aminopyralid) [123].

Similarly, KH-570 (presence of vinyl groups) and APTES (presence of amino groups) have been also used for modifying Fe_3O_4@SiO_2 surfaces for further MIP polymerization by RAFT mechanisms [27,58,124] (Table 1). However, magnetite functionalization with –Cl groups is commonly preferred for RAFT synthesis, and silica-based reagents such as 4-chloromethyl phenyl trichlorosilane (4-CPS) have been widely used for reacting with Fe_3O_4@SiO_2 (magnetite functionalized with TEOS) and promoting –Cl before MIP synthesis by RAFT polymerization [74,77,126–131]. Other reagents, such as 3-bromopropyl trimethoxy silane (BPTS), have been also used when performing MIP synthesis by RAFT, although they are used directly on Fe_3O_4 to promote –Br groups [132] (Table 1).

These silanization procedures are time-consuming processes since they require a previous silanization stage (Fe_3O_4@SiO_2) followed by a treatment for incorporating the desired functional groups. In addition, the described procedures are reported to require reaction temperatures achieved by refluxing systems. Therefore, there have been described several one-step Fe_3O_4 synthesis (solvothermal method) and surface functionalization procedures by incorporating into the reaction medium diol-based reagents such as PEG and ethylene

glycol (EG) [133–140], and vinyled reagents (oleic acid [141] and hexanediamine [140]). Since the solvothermal method is required for Fe_3O_4 nanoparticle synthesis, special laboratory devices such as Teflon-lined stainless steel reactors are needed. In addition, the synthesis/functionalization is performed at high temperature (200 °C) and for long times (up to 24 h).

One-step procedures for Fe_3O_4 silanization (Fe_3O_4@SiO_2) and modification with functional groups such as $-NH_2$ have been also described for speeding-up the functionalization step. The use of APTES as a silica-based reagent (silanization) promotes the simultaneous functionalization with amino functional groups. In addition, if the modification is performed using APTES and mono acrylic acid at once, the resulting covering will also be rich in vinyl groups [142]. On other occasions, after Fe_3O_4 functionalization with oleic acid, a further reaction with KH-570 ensures stability (silica covering) and abundant vinyl groups for further MIP synthesis [90].

2.1.3. Magnetite Surface Functionalization for Magnetic Nanotube-Supported and Magnetic Nanosheet-Supported MIPs

As summarized in Table 2, surface functionalization of mixed magnetic composites involving the presence of CNTs [31] and MWCNTs [143–145] has been efficiently achieved by using diol-based reagents such as EG and PEG [31,143,146,147], although some authors have described the convenience of a previous MWCNT@Fe_3O_4 composite oxidation [144], reduction [145], or carboxylation [32,148] stage before functionalization/MIP synthesis. The activated surface of CNTs/MWCNTs improves the nanoparticles dispersion and the interaction of monomers with the CNTs/MWCNTs. However, some of these procedures require high temperatures and long times are also needed to complete the reactions [31,32,143,148]. After MWCNT@Fe_3O_4 composite synthesis, silanization procedures have also been reported by using KH-570 under moderate reaction conditions (stirring/sonication, N_2 atmosphere, 70 °C, 10 min), which leads to a stable magnetic composite and also increases the reactive activity as a consequence of the anchored vinyl groups [149]. Functionalization with KH-570 can be also performed after a previous silanization of the prepared MCNTs with TEOS (MCNTs@SiO_2) [150]. Methacryloxypropyl trimethoxysilane (MAPTMS) has been also proposed as a silanizing agent and as a vinyled monomer for further IIP synthesis (Pb (II) ions as template and dithizone as a ligand) [148].

Regarding magnetic nanosheet-supported MIPs (Table 2), the GO@Fe_3O_4 surface is usually functionalized by grafting with acrylic acid as shown in Figure 4 [33,151], which ensure the presence of vinyl groups for further polymerization. Acrylic acid was also used for surface modification of chitosan based GO@Fe_3O_4 composites [152]. Silanization with TEOS to prepare MGO@mSiO_2 (mesoporous silica) has been also reported for direct MIP synthesis [153] and for a further functionalization with vinyltrimethoxysilane (VTTS) [34] and APTES [154] in order to facilitate the subsequent polymerization via vinyl or amino groups, respectively. However, prepared GO@Fe_3O_4 nanoparticles [155,156], as well as 3D magnetic GO-CNT composites [35], were also directly used for MIP synthesis without functionalization.

Table 2. Dispersive (micro)solid phase extraction with magnetic nanotube-supported and magnetic nanosheet-supported MIPs.

Sample	Target	Composite	Functionalization Reagent/Monomer	Detection Technique	Sample Pre-Treatment:	Performance: LOD and Analytical Recovery	Ref.
Fruits	Pyrethroids	Fe_3O_4-CNT	TEOS and KH570/MAA	HPLC-UV	Sorbent: MMIP particles (10 mg) Sample volume: 10 mL (3 mL ACN extract) Rebinding media: Water/ACN Extraction time: 15 min (mechanical shaking) Desorption solvent: 97:3 ACN/acetic acid (2 × 1.0 mL), 30 s (ultrasound dispersion)	LOD: 0.0035–0.0072 mg kg^{-1} Recovery: 82.4–101.7%	[31]
Human urine and plasma	Sotalol	Fe_3O_4-MWCNT	–*/AM	HPLC-UV	Sorbent: MMIP particles (15 mg) Sample volume: 5.0 mL for plasma (30 mL after pH adjustment at 7.0 with phosphate buffer solution) Rebinding media: Water/Methanol Extraction time: 15 min (mechanical shaking) plus 2.0 min (vortex shaking) plus 5 min (ultrasound water-bath) Desorption solvent: 90:10 methanol/acetic acid (10 mL), 5 min (ultrasound water-bath)	LOD: 0.31µg L^{-1} Recovery: 94.6–102.5%	[32]
Heat processed foods	Acrylamide	GO-Fe_3O_4	AA (grafting)/AA	HPLC-UV	Sorbent: MMIPs particles (20 mg) Sample volume: 10 mL water/methanol extract from 2.0 g of sample Rebinding media: Water/Methanol Extraction time: 60 min (mechanical shaking) Desorption solvent: 9:1 methanol/acetic acid (2.0 mL), 120 min (mechanical shaking)	LOD: 15µg kg^{-1} Recovery: 86.7–94.3%	[33]
Water	BPA, 4-tert-OP, 4-NP	GO-Fe_3O_4@$mSiO_2$	CTAB, TEOS/VTTS	HPLC-PDA	Sorbent: MMIPs particles (20 mg) Sample volume: 50 mL (pH adjustment at 6.0) Rebinding media: Water Extraction time: 8.0 min (mechanical shaking) Desorption solvent: acetone (1.0 mL), 5.0 min (mechanical shaking)	LOD: 0.013, 0.010 and 0.010 µg L^{-1} Recovery: 81.5–104.1%	[34]
Milk powder	Melamine	GO-Fe_3O_4@$mSiO_2$	CTAB, TEOS/VTTS	UPLC-MS/MS	Sorbent: MMIPs particles (20 mg) Sample volume: 50 mL (pH adjustment at 6.0) Rebinding media: Water Extraction time: 8.0 min (mechanical shaking) Desorption solvent: acetone (1.0 mL), 5.0 min (mechanical shaking)	LOD: 0.00045 mg kg^{-1} Recovery: 90.3–95.7%	[35]
Water	4-nonylphenol	Fe_3O_4-MWCNT	PEG/4-VP	HPLC-UV	Sorbent: MMIPs particles (50 mg) Sample volume: 20 mL Rebinding media: Water Extraction time: 20 min (mechanical shaking) Desorption solvent: 90:10 methanol/acetic acid (2.0 mL), 40 min (mechanical shaking)	LOD: 0.15 µg L^{-1} Recovery: 88.6–98.1%	[143]

Table 2. *Cont.*

Sample	Target	Composite	Functionalization Reagent/Monomer	Detection Technique	Sample Pre-Treatment:	Performance: LOD and Analytical Recovery	Ref.
Food / human plasma	Curcumin	Fe_3O_4-MWCNT	PEG/AM	HPLC-UV	Sorbent: MMIPs particles (31 mg) Rebinding media: Water Extraction time: 20 min (ultrasound dispersion) Desorption solvent: 4:1 methanol/DMSO (0.25 mL), 40 min (mechanical shaking)	LOD: 0.028 μg L^{-1} Recovery: >98%	[144]
Urine	Morphine	Fe_3O_4-MWCNT	VTMOS/MAA	UV-Visible spectrophotometry	Sorbent: MMIPs particles (50 mg) Sample volume: pH adjusted at 4.0 Rebinding media: Water Extraction time: 20 min (ultrasound dispersion) Desorption solvent: methanol (1.0 mL), 3.0 min (mechanical shaking)	LOD: 0.18 mg L^{-1} Recovery: 96.4–105.6%	[145]
Urine	Levofloxacin	Fe_3O_4-CNT	–/MAA	HPLC-PDA	Sorbent: MMIPs particles (50 mg) Sample volume: 2.5 mL Rebinding media: Water Extraction time: 60 min (incubation) Desorption solvent: 6:4 methanol/acetic acid (5.0 mL)	LOD: 0.01 mg L^{-1} Recovery: 78.7–83.4%	[146]
Serum	Gatifloxacin	Fe_3O_4-CNT	–*/MAA	HPLC-PDA	Sorbent: MMIPs particles (50 mg) Sample volume: 2.5 mL Rebinding media: Water Extraction time: 60 min (incubation) Desorption solvent: 6:4 methanol/acetic acid (5.0 mL)	LOD: 6.0 μg L^{-1} Recovery: 79.1–85.3%	[147]
Water	Pb(II)	Fe_3O_4-MWCNT	MAPTMS, DTZ/AM	FAAS	Sorbent: MMIPs particles (15 mg) Sample volume: pH adjusted at 6.0 Rebinding media: Water Extraction time: 5.0 min (mechanical shaking) Desorption solvent: 0.5 M thiourea in 0.5 M HCl (5.0 mL), 15 min	LOD: 11 μg kg^{-1} Recovery: >98.4%	[148]
—	Dibenzothiophene	Fe_3O_4-MWCNT	KH570/MAA	UV-Visible spectrophotometry	Sorbent: MMIPs particles (10 mg) Rebinding media: Hexane Extraction time: 120 min (mechanical shaking) Desorption solvent: 9:1 methanol/acetic acid (mechanical shaking)	LOD: – Recovery: –	[149]
Porcine serum	Porcine serum albumin	Fe_3O_4-CNT	TEOS, MPS/zinc acrylate	HPLC-UV	Sorbent: MMIPs particles (40 mg) Sample volume: 4.0 mL (diluted in PBS, pH 7.0) Rebinding media: Water Desorption solvent: 10% (m/v) SDS and 10% (*v*/*v*) acetic acid	LOD: – Recovery: –	[150]
Extracts from of Evodiae fructus	Evodiamine and rutaecarpine	GO-Fe_3O_4	–*/MAA	HPLC-UV	Sorbent: MMIPs particles (20 mg) Sample volume: 20 mL (methanolic extract from 1.0 g of sample) Rebinding media: Methanol Extraction time: mechanical shaking Desorption solvent: 9:1 methanol/acetic acid	LOD: – Recovery: –	[151]

Table 2. *Cont.*

Sample	Target	Composite	Functionalization Reagent/Monomer	Detection Technique	Sample Pre-Treatment:	Performance: LOD and Analytical Recovery	Ref.
Water, urine drug capsules	Fluoxetine	GO-Fe_3O_4-Chm	AA/MAA	UV-Visible spectrophotometry	Sorbent: MMIPs particles (20 mg) Sample volume: pH adjusted at 4.5 Rebinding media: Water Extraction time: 10 min (mechanical shaking) Desorption solvent: 9:1 methanol/acetic acid (0.1 mL), 5.0 min (ultrasound dispersion)	LOD: 0.03 μg L^{-1} Recovery: 95.7–104.0%	[152]
Water	PAEs	GO-Fe_3O_4- mSiO_2	CTAB, TEOS/PTMOS, APTES	GC-MS	Sorbent: MMIPs particles (20 mg) Sample volume: 100 mL (pH adjusted at 7.0) Rebinding media: Water Extraction time: 30 min (mechanical shaking) Desorption solvent: ethanol (3.0 mL), 5.0 min (mechanical shaking)	LOD: 0.01–0.05 μg L^{-1} Recovery: >92.9%	[153]
Rhododendrons species	Flavonoids	Fe_3O_4@SiO_2-GO	APTES, THPMP/4-VP	HPLC-MS	Sorbent: MMIPs particles (20 mg) Rebinding media: ACN Desorption solvent: 6:4 methanol/acetic acid, 11 min (mechanical shaking)	LOD: 0.06–0.08 μg L^{-1} Recovery: >64.0%	[154]
Water	4-Nitrophenol	GO-Fe_3O_4	–*/PTEOS, TMOS	HPLC-UV	Sorbent: MMIPs particles (20 mg) Sample volume: 100 mL Rebinding media: Water Extraction time: 5.0 min (incubation) Desorption solvent: 4:1 methanol/acetic acid (5.0 mL)	LOD: — Recovery: 94.7–101.2%	[155]
Water	Microcystin-LR	GO-Fe_3O_4	–*/Dopamine	HPLC-UV	Sorbent: MMIPs particles (10 mg) Rebinding media: Water Extraction time: 25 min (ultrasound dispersion) Desorption solvent: 8:2 methanol/acetic acid (0.1 mL)	LOD: 0.08 μg L^{-1} Recovery: 86–113%	[156]

4-NP, 4-nonylphenol; 4-tert-OP, 4-tert-octylphenol; AA, acrylic acid; 4-VP, 4-vinylpyridine; ACN, acetonitrile; AM, acrylamide; APTES, (3-aminopropyl)triethoxysilane; BPA, bisphenol A; Chm, chitosan; CNTs, carbon nanotubes; CTAB, cetyl trimethylammonium bromide; DTZ, dithizone; FAAS, flame atomic absorption spectrometry; GC, gas chromatography; GO, graphene oxide; HPLC. High performance liquid chromatography; KH570, γ-methacryloxypropyltrimethoxysilane; LOD, limit of detection; MAA, methacrylic acid; MAPTMS, methacryloxypropyl trimethoxysilane; MMIPs, magnetic molecularly imprinted polymer; MPS, 3-methacryloxypropyltrimethoxysilane; MS, mass spectrometry; MS/MS, tandem mass spectrometry; MWCNTs, multiwalled carbon nanotubes; PAEs, phthalates esters; PBS, phosphate buffered saline; PDA, photodiode array detector; PEG, polyethyleneglycol; PTEOS, phenyltriethoxysilane; PTMOS, phenyl trimethoxysilane; SDS, sodium dodecyl sulfate; TEOS, tetraethyl orthosilicate; THPMP, 3-trihydroxymethylsilyl-propylmethylhosphate; TMOS, tetramethyl orthosilicate; UPLC, ultra performance liquid chromatography; UV, ultraviolet; VTMOS, vinyltrimethoxysilane; VTTS, vinyltrimethoxysilance. (*) No functionalization reagent.

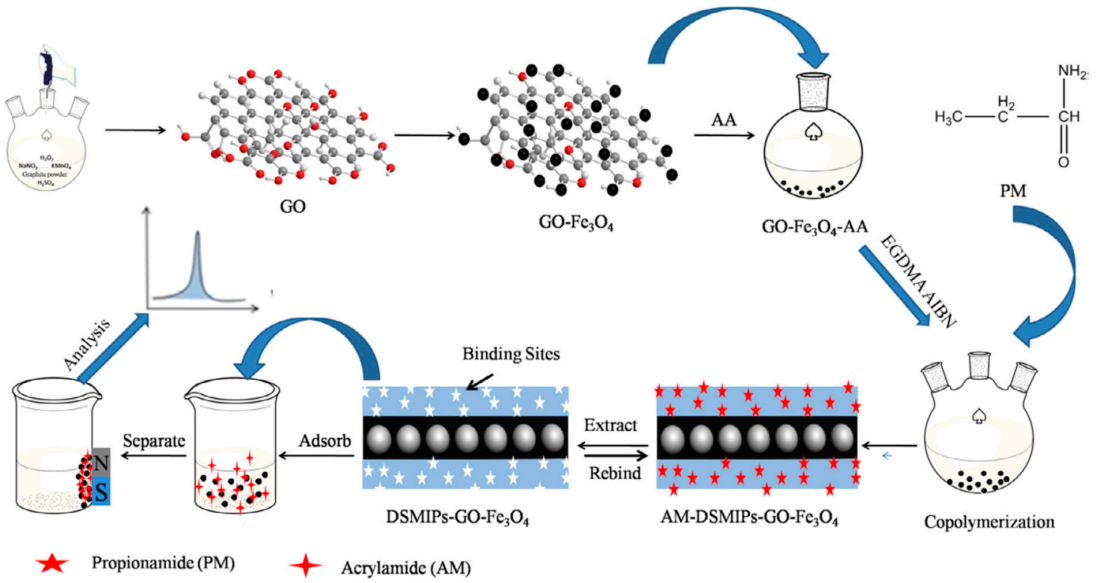

Figure 4. Schematic representation for preparation of acrylamide-dummy-surface molecularly imprinted polymers-graphene oxide-Fe_3O_4 (AM-DSMIPs-GO-Fe_3O_4), Adapted with permission from Ref. [33]. Copyright 2017 Elsevier.

2.1.4. Magnetite Functionalization for Magnetic Porous MIPs

As previously commented, functionalization in HPMIPs based on mesoporous silica (1,2-diol groups over the HPMIPs) can be achieved by treating the composite with diluted perchloric acid (Figure 5) [37]. Silica-based reagents such as KH-570 [40] and APTES [41] have been used for surface functionalization of MMC particles (vinyl −C=C− bridge introduction) [40] and –NH_2 [41] groups for subsequent MIP synthesis (Table 3). Similarly, silanization with KH-570 (vinyl group functionalization) has also been proposed for preparing MMIPs based on hollow magnetite [45].

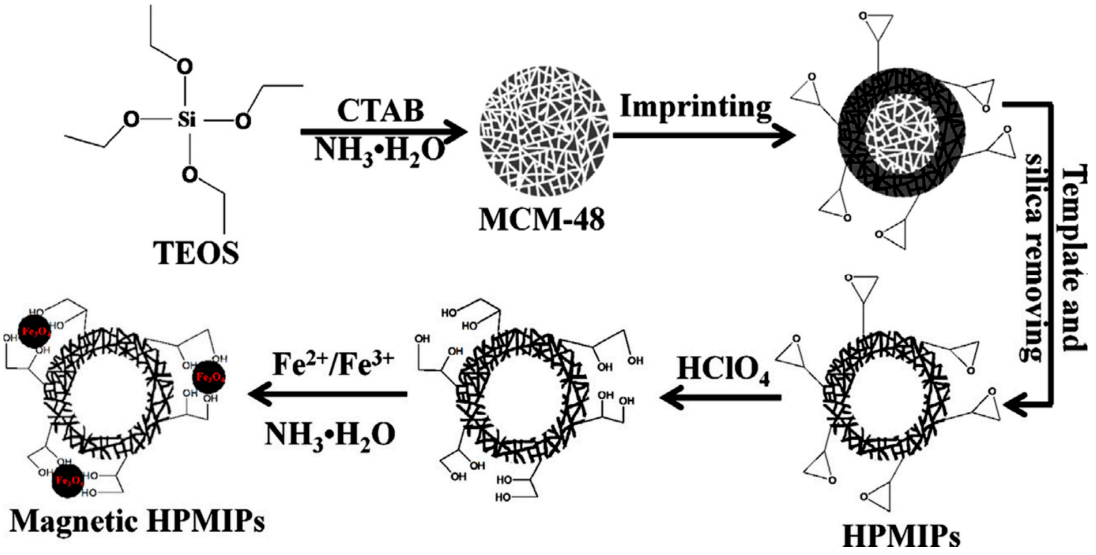

Figure 5. Schematic representation for preparation of magnetic hollow porous molecularly imprinted polymers (HPMIPs), Adapted with permission from Ref. [37]. Copyright 2015 Elsevier.

Table 3. Dispersive (micro)solid phase extraction with magnetic porous MIPs and other mixed composite MMIPs.

Sample	Target	Composite	Reagents/Monomer	Detection Technique	Sample Pre-Treatment:	Performance: LOD and Analytical Recovery	Ref.
Spices	Protocatechuic acid	Fe_3O_4-HPMIP	TEOS, CTAB/4-VP, GMA	HPLC-PDA	Sorbent: MHPMIP particles (10 mg) Sample volume: 3.0 mL Rebinding media: ACN Extraction time: 25 min (mechanical shaking) Desorption solvent: 9:1 methanol/acetic acid (0.8 mL)	LOD: 0.4 mg L^{-1} Recovery: 94.2–101.1%	[37]
Rat urines	Aristolochic acid I and II	MMC@MIP	APTES/MAA	HPLC-UV	Sorbent: MMC@MIP particles (80 mg) Sample volume: 3.0 mL Rebinding media: Water Extraction time: 30 min (mechanical shaking) Desorption solvent: 3:1 methanol/DES-1 (3.0 mL)	LOD: 0.03 and 0.17 mg L^{-1} Recovery: 86.7–94.3%	[41]
Potato chips	Acrylamide	Fe_3O_4@ DMIP	TEOS/APTMS	HPLC-UV	Sorbent: Fe_3O_4@ DMIP particles (30 mg) Sample volume: 10 mL (aqueous extract, pH 4.0, from 1.0 g of sample) Rebinding media: Water Extraction time: 35 min (ultrasound dispersion) Desorption solvent: 45:45:10 ACN/methanol/acetic acid (2.0 mL) (ultrasound dispersion)	LOD: 0.35 µg kg^{-1} Recovery: 94–98%	[42]
Urine	Baclofen	SMIBP	TEOS, Chm/–*	HPLC-UV	Sorbent: SMIBP particles (35 mg) Sample volume: 10 mL (pH adjusted at 11) Rebinding media: Water Extraction time: 24 min (ultrasound dispersion) Desorption solvent: 45:45:10 methanol/deionized water/ammonium hydroxide (2.0 mL) (ultrasound dispersion)	LOD: 0.26 µg L^{-1} Recovery: 94–98%	[43]
Water, fruit juices, human serum	Benzoic acids	MMIR	Melamine, formaldehyde	HPLC-UV	Sorbent: MMIR particles (20 mg) Sample volume: 3.0 mL Rebinding media: Water Extraction time: mechanical shaking Desorption solvent: 3:3:1 methanol-water-acetic acid (3.0 mL)	LOD: 0.02–1.0 mg L^{-1} Recovery: 81.8–108.7%	[44]
Chicken meat	Sulfonamides	CS-NR-Mag-MIP	TEPA/GMA	HPLC-MS/MS	Sorbent: CS-NR-Mag-MIP particles (15 mg) Sample volume: 3.0 mL (aqueous extract, pH adjusted at 5.0 from 1.0 g of sample) Rebinding media: Water Extraction time: 10 min (mechanical shaking) Desorption solvent: 2.0% ammonia solution in methanol. (3 × 0.5 mL)	LOD: 0.013–0.099 µg kg^{-1} Recovery: 81.8–108.7%	[48]

Table 3. *Cont.*

Sample	Target	Composite	Reagents/Monomer	Detection Technique	Sample Pre-Treatment:	Performance: LOD and Analytical Recovery	Ref.
Water, urine, plasma	Gallic acid	HKUST-1-MOF-Fe_3O_4-MIP	HKUST-1 MOF, VTMOS/–	UV-Visible spectrophotometry	Sorbent: CS-NR-Mag-MIP particles (1.6 mg) Sample volume: 10 mL (pH adjusted at 3.0) Rebinding media: Water Extraction time: 2.0 min (ultrasound dispersion) Desorption solvent: ethanol (0.18 mL), 2.0 min (ultrasound dispersion)	LOD: 1.377 μg L^{-1} Recovery: 92.3–100.6%	[157]
Pork meat, fish	Estrogens	Fe_3O_4@ZIF-8-MIP	Fe_3O_4@ZIF-8/APBA	HPLC-PDA	Sorbent: coated-SPME fiber Rebinding media: n-hexane Desorption solvent: 99:1 methanol/acetic acid	LOD: 0.4–1.7 μg kg^{-1} Recovery: 92.3–100.6%	[158]
Corn	Aflatoxins	ZIF-L-based Co-MNPC@MIP	Co-MNPC/MAA	HPLC-MS/MS	Sorbent: ZIF-L-based Co-MNPC@MIP particles (80 mg) Sample: methanol/water extract Rebinding media: Water Extraction time: 10 min (mechanical shaking) Desorption solvent: 6:4 ACN/water (1.2 mL), 5.0 min (ultrasound dispersion)	LOD: 0.05–0.07 μg L^{-1} Recovery: 75.1–99.4%	[159]
Corn	Hydroxychloroquine	Ni@MIL-100(Fe)@MIP	Ni@MIL-100(Fe)/APTES	HPLC-UV	Sorbent: Ni@MIL-100(Fe)@MIP particles (23 mg) Sample volume: 10 mL (pH adjusted at 9.0) Rebinding media: Water Extraction time: 1.0 min (mechanical shaking) Desorption solvent: methanol (50 μL), 3.0 min (ultrasound dispersion)	LOD: 0.2 μg L^{-1} Recovery: 96–103%	[160]
Vegetables	Carbendazim	Fe_3O_4-β-CD@MIP	β-CD, Au NPs/MAA	UHPLC-MS	Sorbent: Fe_3O_4-β-CD@MIP particles (packaged SPE column) Sample volume: (ACN extract from 25 g of sample), flow rate 1.0 mL min^{-1} Rebinding media: ACN Desorption solvent: methanol/acetic acid (flow rate 1.0 mL min^{-1})	LOD: 3.0 ng L^{-1} Recovery: 90.5–109%	[161]
Water	PAEs	MIP@$mSiO_2$-β-CD@Fe_3O_4	β-CD, APTES/MAA	GC-MS	Sorbent: MIP@$mSiO_2$-β-CD@Fe_3O_4 particles (30 mg) Rebinding media: Water Extraction time: 10 min Desorption solvent: 8:2 methanol/acetic acid (6.0 mL), 10 min	LOD: 1.0–5.0 μg L^{-1} Recovery: 80.2–103%	[162]
Urine, serum	Carbamazepine	Fe_3O_4@CS@MIP	CS DCMA, DCC/4-VP	HPLC-DAD	Sorbent: Fe_3O_4@CS@MIP (4.0 mg) Sample volume: 4.0 mL serum, 20 mL urine (pH adjusted at 9.0) Rebinding media: Water Extraction time: 30 min (mechanical shaking) Desorption solvent: 8:2 ethanol/acetic acid (3 × 4.3 mL for serum; 2 × 4.6 mL for urine), 28.7 min (serum), 25.3 min (urine), mechanical shaking	LOD: 1.0 μg L^{-1} (urine), 9.6 μg L^{-1} (serum) Recovery: 88.2–101.2%	[163]

Table 3. *Cont.*

Sample	Target	Composite	Reagents/Monomer	Detection Technique	Sample Pre-Treatment:	Performance: LOD and Analytical Recovery	Ref.
Water, rice, vegetables	As	Fe_3O_4@OA-IIP	2-ABT/4-VP	HG-AAS	Sorbent: Fe_3O_4@OA-IIP (88.18 mg) Sample volume: 4.0 mL serum, 20 mL urine (water and acid digests adjusted at pH 7.25) Rebinding media: Water Extraction time: 47.63 min, 30 °C (ultrasound dispersion) Desorption solvent: 0.75 M nitric acid (0.50 mL)	LOD: 0.003 μg L^{-1} Recovery: 88.2–101.2%	[164]

2-ABT, 2-acetyl benzofuran thiosemicarbazone; 4-VP, 4-vinylpyridine; ACN, acetonitrile; APBA, 3-amino phenylboronic acid; APTES, (3-aminopropyl)triethoxysilane; APTMS, 3-aminopropyltrimethoxysilane; CD, cyclodextrin; Chm, chitosan; CTAB, cetyl trimethylammonium bromide; CS, magnetic chitosan; DAD, diode array detector; DCC, N, N-dicyclohexylcarbodiimide; DCMA, 2-(dodecylthiocarbonothioylthio)-2-methylpropionic acid; DES-1, deep eutectic solvent 1; GC, gas chromatography; GMA, glycidilmethacrylate; HG-AAS, hydride generation—atomic absorption spectrometry; HPLC. High performance liquid chromatography; LOD, limit of detection; MAA, methacrylic acid; MHPMIP, magnetic hollow porous molecularly imprinted polymer; MMC@MIP, magnetic mesoporous carbon-molecularly imprinted polymer; MMIR, hydrophilic magnetic molecular imprinted resin; MNPC, magnetic nanoporous carbon; MOF, metal-organic framework; MPS, 3-methacryloxypropyltrimethoxysilane; MS, mass spectrometry; MS/MS, tandem mass spectrometry; NPs, nanoparticles; OA, oleic acid; PAEs, phthalates esters; PDA, photodiode array detector; SMIBP, superparamagnetic molecularly imprinted biopolymer; SPE, solid phase extraction; SPME, solid phase microextraction; TEOS, tetraethyl orthosilicate; TEPA, tetraethylenepentamine; UHPLC, ultra-high performance liquid chromatography; UV, ultraviolet; ZIF-8, zeolite imidazolateframework-8 coated magnetic iron oxide. (*) monomer was not used.

2.1.5. Other Mixed Composites for MMIPs

Various types of magnetic composites (Table 3) have been used as magnetic cores for MMIPs such as metal-organic frameworks (MOFs) and zeolite imidazolate frameworks (ZIFs). In some cases, the synthesis of Fe_3O_4 nanoparticles following the hydrothermal process is performed in the presence of the framework (HKUST-1, a Cu-based porous MOF) and EG (diol groups), which also act as capping agents for avoiding aggregation [157]. Surface functionalization is further performed with VTMOS [157]. The procedure is time-consuming, and a Teflon-lined stainless steel autoclave (synthesis at 200 °C) is required. On other occasions, the previously synthesized magnetite nanoparticles are allowed to react with the framework at moderate temperatures and short times. This is the case of Fe_3O_4@ZIF-8 composites, in which poly (styrenesulfonate sodium salt) is added to the reaction medium to allow the ZIF-8 shell growth (with the presence of 2-methylimidazolate as a precursor), and for which a further surface functionalization is not required [158]. Moreover, ZIF-L-based Co-based magnetic nanoporous carbon (Co-MNPC) is also directly mixed in the polymerization medium for preparing a magnetic selective Co-MNPC@MIP sorbent to aflatoxins [159].

Ni@MIL-100(Fe) MOF has also been used as a support for MIP synthesis [160]. In this case the framework exhibits magnetic properties, and after mixing with the template (hydroxychloroquine) and with the functional monomer (APTES) and the cross-linker (TEOS), MIP synthesis can be directly carried out.

Fast procedures for synthesizing magnetic composites have been also described for thiolated β-cyclodextrin assembled to gold nanoparticles (β-CD/Au), whose presence during the Fe_3O_4 synthesis (co-precipitation method) leads to a β-CD/Au/Fe_3O_4 composite functionalized for further MIP synthesis [161]. Other proposals suggested the previous synthesis of Fe_3O_4@mSiO_2 functionalized with APTES (presence of $-NH_2$ groups) magnetic core before surface grafting of β-CD and MIP synthesis (phthalic acid ester as a template) for preparing magnetic plasticizer MIPs [162]. In addition to the high selectivity inherent to the MIP layer, the prepared composite material was found to show large adsorption capacity and fast kinetic equilibrium.

The aminopolysaccharide nature of the biopolymer chitosan (CS) has also taken advantage of modifying magnetite for achieving a surface rich in functional groups for further polymerization. Preparation of Fe_3O_4@CS nanoparticles is easily performed following the hydrothermal synthesis of Fe_3O_4 in the presence of CS [163]. Finally, one-step co-precipitation under alkaline conditions (Fe_3O_4 synthesis) in the presence of the diazonium salt BF_4 ($^+N_2$–C_6H_4–CH_2–DEDTC) also generates a magnetic core that offers adequate functional groups for mixing with the pre-polymerization mixture and starting the MIP synthesis [165]. On other occasions, surface-modified Fe_3O_4 with oleic acid was allowed to polymerize with arsenic (III)- 2-acetyl benzofuran thiosemicarbazone complex as template, and methacrylic acid as a monomer (ionic imprinted polymer for As(III)) before a Pickering emulsion in the presence of nanoparticles of chitosan [164]. Extraction of As(III) from acid digests from rice and vegetable was achieved after pH adjustment, assisting the loading/elution process by ultrasounds [164].

2.2. Dispersive Solid Phase Extraction and Microsolid Phase Extraction with Non-Magnetic MIPs

As previous commented, dSPE/D-μ-SPE [9–11] can be performed by dispersing MMIP nanoparticles, and also non-magnetic MIP beads, by vortex and ultrasound stirring [10]. Table 4 summarizes the main features regarding dSPE/D-μ-SPE with non-magnetic MIPs. The adsorbents can be obtained by precipitation [166–174], and bulk [175–177] polymerization has been used for dSPE/D-μ-SPE by shaking the sample/extract-MIP bead mixtures for times varying from 5.0 min [166] to 3.0 h [169]. Absorption times can be reduced to 1 min when assisting the procedure by ultrasounds, enough time for isolating phenolic compounds in aqueous samples using 10 mg of MIP [170]. However, sonication times of 3.0 h have been proposed for fluoroquinolone pre-concentrations from waters using a dual-template MIP (dt-MIP) for norfloxacin and enrofloxacin as templates [172]. Authors,

however, did not report insights regarding low adsorption rate for reaching the equilibrium between the targets and the dt-MIPs. The procedures were found to be effective and selective for isolating fungicides from cucumber [168], sulfonamides from milk [166], bioactive compounds (polydatin) from rat's plasma and urine [169], progesterone hormones from plasma, urine and waters [175], and also for purifying extracts (pre-concentrating targets) such as polydatin from Chinese medical medicines [169], antibiotics from pork [167], aflatoxins from cultured fish [171], pyraclostrobin from ginseng [174], folic acid from foodstuff [176], and herbicides from shellfish [177].

Table 4. Dispersive solid phase extraction and microsolid phase extraction with non-magnetic MIPs.

Sample	Target	Composite	Reagents/Monomer	Detection Technique	Sample Pre-Treatment:	Performance: LOD and Analytical Recovery	Ref.
Urine	Glibenclamide	HPMIP	TEOS, CTAB/MAA	HPLC-UV	Sorbent: MHPMIP particles (30 mg) Sample volume: 10 mL (pH adjusted at 4.0) Rebinding media: Water Extraction time: 15 min (ultrasound dispersion) Desorption solvent: 5:5:1 DMSO/ethanol/acetic acid (3.0 mL), resuspension	LOD: 3.5 μg L^{-1} Recovery: 87.7–104.3%	[38]
Water	Bisphenol A	HM-DMIP	TEOS/ICPTES	HPLC-UV	Sorbent: HM-DMIP particles (30 mg) Sample volume: 10 mL Rebinding media: Water Extraction time: 30 min (static absorption conditions) Desorption solvent: 90:10 methanol/acetic acid (3.0 mL), static absorption conditions	LOQ: 0.2 mg L^{-1} Recovery: 98.7–101.7%	[39]
Milk	Sulfamethazine	MIP	–*/MAA	CE-UV	Sorbent: MIP particles (10 mg) Sample volume: 10 mL (pH adjusted with 20 mM phosphate buffer) Rebinding media: Water Extraction time: 5.0 min (mechanical shaking) Desorption solvent: 9:1 methanol/acetic acid (0.30 mL), 10 min (mechanical shaking)	LOD: 1.1 μg L^{-1} Recovery: 89–110%	[166]
Pork meat	MALs	MIP	–*/MAA	HPLC-MS/MS	Sorbent: MIP particles (20 mg) Sample volume: 5.0 mL (1% (v/v) acetic acid in ACN extract from 1.0 g of sample) Rebinding media: ACN Extraction time: 30 min (mechanical shaking) Desorption solvent: 10% (v/v) acetic acid in methanol (5.0 mL), 10 min (ultrasound dispersion)	LOD: 0.2–0.5 μg kg^{-1} Recovery: 68.6–95.5%	[167]
Cucumber	Azoxystrobin	HMIM	–*/HPMA	HPLC-UV	Sorbent: HMIM particles (100 mg) Sample volume: 5.0 mL (methanol extract from 25 g of sample) Rebinding media: Methanol Extraction time: 30 min (water-bath oscillation plus 30 min without oscillation) Desorption solvent: 9:1 methanol/acetic acid (8.0 mL), water-bath oscillation	LOD: 0.324 μg kg^{-1} Recovery: 85.9–88.9%	[168]

Table 4. *Cont.*

Sample	Target	Composite	Reagents/Monomer	Detection Technique	Sample Pre-Treatment:	Performance: LOD and Analytical Recovery	Ref.
PCRR, and plasma and urine from rat	Polydatin	MIP	–*/4-VP	HPLC-UV	Sorbent: MIP particles (10 mg for PCRR, 5.0 mg for plasma and urine) Sample volume: 1.5 mL (extracts from PCRR), 0.20 mL (plasma), 0.050 mL (urine) Rebinding media: 8:2 water/methanol (extracts from PCRR), water (plasma and urine) Extraction time: 3.0 h, mechanical shaking Desorption solvent: 1.5 mL of 8:2 water/methanol and 3.0 h (mechanical shaking)	LOD: 0.125 mg L^{-1} Recovery: 89.2–91.6%	[169]
Water	Phenolic compounds	MIP	–*/MAA	CE-DAD	Sorbent: MIP particles (10 mg) Sample volume: 10 mL Rebinding media: Water Extraction time: 1.0 min (ultrasound dispersion) Desorption solvent: 9:1 ACN/acetic acid (30 µL), 4.0 min (ultrasound dispersion)	LOD: 0.18–0.44 µg L^{-1} Recovery: 70.7–106.7%	[170]
Fish	Aflatoxins	MIP	–*/MAA	HPLC-MS/MS	Sorbent: MIP particles (40 mg) Sample volume: 1.5 mL (60:40 ACN/phosphate buffer extract from 1.g of sample) Rebinding media: 60:40 ACN/phosphate buffer, pH 6.0 Extraction time: 3.0 min (mechanical shaking) Desorption solvent: 97.5:2.5 ACN/formic acid (0.50 mL), 4.0 min (mechanical shaking)	LOD: 0.29–0.61 µg kg^{-1} Recovery: 83–98%	[171]
Water	FQs	dt-MIP	–*/MAA	HPLC-DAD	Sorbent: dt-MIP particles (10 mg) Sample volume: 10 mL Rebinding media: Water Extraction time: 3.0 h (mechanical shaking) Desorption solvent: 90:10 methanol/acetic acid (0.15 mL), 5.0 min (ultrasound dispersion)	LOD: 0.2 (NOR) and 0.67 (ENR) µg L^{-1} Recovery: 80.9–101.0%	[172]
Rice	Inorganic As	IIP	1-vinylimidazole-/MAA	HPLC-ICP-MS	Sorbent: IIP particles (50 mg) Sample volume: 1.5 mL (1:1 methanol/water extract from 1.g of sample) Rebinding media: 1:1 methanol/water (pH 8.0) Extraction time: 1.0 min (mechanical shaking) Desorption solvent: water (0.15 mL), 1.0 min (mechanical shaking)	LOD: 0.20 (As(III)) and 0.41 (As(V)) µg kg^{-1} Recovery: 95–103%	[173]
Ginseng	Pyraclostrobin	MIP	–*/MAA	HPLC-UV	Sorbent: MIP particles (100 mg) Sample volume: 2.0 mL (ACN extract from 25 g of sample) Rebinding media: ACN Extraction time: 50 min (mechanical shaking) Desorption solvent: 9:1 methanol/acetic acid (8.0 mL), 50 min (mechanical shaking)	LOD: 0.01 mg kg^{-1} Recovery: 95–103%	[174]

Table 4. *Cont.*

Sample	Target	Composite	Reagents/Monomer	Detection Technique	Sample Pre-Treatment:	Performance: LOD and Analytical Recovery	Ref.
Water, urine, serum	Progesterone	MIP	–*/pyrrole	GC-FID	Sorbent: MIP particles (100 mg) Sample volume: 20 mL (pH adjusted at 6.5) Rebinding media: Water Extraction time: 35 min (ultrasound dispersion) Desorption solvent: methanol (0.5 mL), 40 min (ultrasound dispersion)	LOD: 0.625 μg L^{-1} Recovery: 88–101%	[175]
Food	Folic acid	MIP	–*/VBTMAC	HPLC-MS	Sorbent: MIP particles (50 mg) Sample volume: 10 mL (aqueous extract) Rebinding media: Water Extraction time: 20 min (mechanical shaking) Desorption solvent: 1:1 methanol/hydrochloric acid (10 mL), 30 min (mechanical shaking)	LOD: 0.003 mg L^{-1} Recovery: 79–83%	[176]
Seafood	Herbicides	MIP	–*/MAA	GC-MS/MS	Sorbent: MIP particles (50 mg) Sample volume: 10 mL (ACN/acetic acid aqueous extract from 2.0g of sample) Rebinding media: ACN/water Extraction time: 15 min (mechanical shaking) DSPE for clean-up	LOQ: 0.03–8.88 μg kg^{-1} Recovery: 81–109%	[177]
Chicken meat	TCs	MIP-MOF	UiO-66 MOF/MAA	HPLC-UV	Sorbent: MIP-MOF particles (5 mg) Sample volume: 10 mL (aqueous extract, pH adjusted at 4.0, from 1.0 g of sample) Rebinding media: Water Extraction time: 15 min (mechanical shaking) Desorption solvent: methanol (1.0 mL), 5.0 min (mechanical shaking)	LOD: 0.2–5.0 μg L^{-1} Recovery: 69.6–94.7%	[178]
Urine, milk	Nicotinamide	MIP-MOF	HKUST-1 MOF/MAA	UV-Vis spectrophotometry	Sorbent: MIP-MOF particles (2 mg) Sample volume: 10 mL (pH adjusted at 5.0) Rebinding media: Water Extraction time: 5.0 min (ultrasound dispersion) Desorption solvent: ACN (0.20 mL)	LOD: 1.96 μg L^{-1} Recovery: 95.8–101.3%	[179]
Water	Estrogens	MIHS	Colloidal silica, KH570/MAA	HPLC-UV	Sorbent: MIHS particles (10 mg) Sample volume: 1.0 mL Rebinding media: Water Extraction time: 15 min (dispersion) Desorption solvent: 8:2 methanol/acetic acid (1.0 mL)	LOD: 0.1–0.26 μM L^{-1} Recovery: 69.6–94.7%	[180]
Urine	Valsartan and losartan	HP-MIN	CNPs/TEOS	HPLC-UV	Sorbent: HP-MIN particles (40 mg) Sample volume: 15 mL (pH adjusted at 6.0) Rebinding media: Water Extraction time: 27 min (ultrasound dispersion) Desorption solvent: 90:10 methanol/acetic acid (2.0 mL), ultrasound dispersion	LOD: 1.5 (VAL) and 1.4 (LOS) μg L^{-1} Recovery: 93–99%	[181]

Table 4. *Cont.*

Sample	Target	Composite	Reagents/Monomer	Detection Technique	Sample Pre-Treatment:	Performance: LOD and Analytical Recovery	Ref.
Beverages	DOP	MWCNT-MIP	MWCNTs/MAA	GC-MS	Sorbent: MWCNT-MIP particles (60 mg) Sample volume: 20 mL (ACN extract) Rebinding media: ACN Extraction time: 30 min (oscillation) Desorption solvent: 9:1 methanol/acetic acid	LOD: 2.3 ng L^{-1} Recovery: 88.6–93.0%	[182]
Water	DEHP	GO-MIP	GO/MAA	HPLC-UV	Sorbent: GO-MIP particles (20 mg) Sample volume: 600 mL Rebinding media: Water Extraction time: 30 min (mechanical shaking) Desorption solvent: acetone (6.0 mL), 5.0 min (ultrasound dispersion)	LOD: 0.92 μg L^{-1} Recovery: 82–92%	[183]

AAm, acrylamide; 4-VP, 4-vinylpyridine; ACN, acetonitrile; CE, capillary electrophoresis; CNPs, carbon nanoparticles; CTAB, cetyl trimethylammonium bromide; DAD, diode array detector; DEHP, bis(2-ethylhexyl) phthalate; DOP, dioctyl phthalate; DSPE, dispersive solid phase extraction; dt-MIP, dual-template molecularly imprinted polymer; ENR, enrofloxacin; FID, flame ionization detector; FQs, fluoroquinolones; GO, graphene oxide; HM-DMIP, hollow mesoporous silica surface dummy molecularly imprinted polymer; HMIM, hydrophilic molecularly imprinted microsphere; HPLC, high performance liquid chromatography; HPMA, hydroxypropyl methacrylate; HPMIP, hollow porous molecularly imprinted polymer; HP-MIN, hollow porous molecularly imprinted nanosphere; ICP, inductively coupled plasma; ICPTES, (3-isocyanatopropyl)triethoxysilane; IIP, ionic imprinted polymer; KH570, γ-methacryloxypropyltrimethoxysilane; LOD, limit of detection; LOQ, limit of quantification; LOS, losartan; MAA, methacrylic acid; MALs, macrolide antibiotics; MIH, molecularly imprinted hollow sphere; MIP, molecularly imprinted polymer; MOF, metal-organic frameworks; MS, mass spectrometry; MS/MS, tandem mass spectrometry; MWCNTs, multiwalled carbon nanotubes; NOR, norfloxacin; PCRR, *Polygoni Cuspidati Rhizoma et Radix*; SiO_2@MPS, methacryloxypropyl modified silica nanoparticle; TCs, tetracyclines; TEOS, tetraethyl orthosilicate; UHPLC, ultra-high performance liquid chromatography; UV, ultraviolet; VAL, valsartan; VBTMAC, vinylbenzyl trimethylammonium chloride; VTTS, vinyltrimethoxysilance. (*) no reagent was used.

Ionic molecularly imprinted polymers (IIPs) have also been proposed for dSPE [173]. The bifunctional monomer 1-vinylimidazole was used for reacting with the template ((meta)arsenite, As(III)) and providing vinyl groups for polymerization [173]. The dSPE implied portions of 50 mg of IIP and vortexing at 1000 rpm for 1 min allowed the selective pre-concentration of inorganic arsenic species (As (III) plus As(V)) from rice extracts [173].

MIP synthesis around non-magnetic nanoparticles, such as silica nanoparticles, has been also performed to obtain stable adsorbents. Therefore, silica nanoparticles functionalized with KH-570 (SiO_2@KH-570) by a base-catalyzed reaction of TEOS and KH-570 have been used as a core for preparing a selective MIP composite for the enantioseparation of racemic tryptophan (L-tryptophan recognition) in aqueous solutions [184]. Experiments were performed by oscillating the MIP-aqueous sample mixtures for 32 h at room temperature and using only 2 mg of adsorbent [184].

In addition, the excellent properties of MOFs have led to preparation of MOF-MIP composites based on UiO-66 MOF [178] and HKUST-1 MOF [180] by direct MIP polymerization on the MOF's surface. The dSPE was performed with 5 mg of UiO-66-MIP and shaking for 15 min to recover tetracyclines from chicken extracts [178]; whereas 2 mg of HKUST-1-MIP (vortexing for 2 min) proved adequate for nicotinamide pre-concentration [179].

Hollow non-magnetic composites based on silica [180] and carbon [181] have been also prepared for dSPE/D-μ-SPE. In both cases, after MIP synthesis over the nanoparticle, the supporting material was removed (hydrofluoric acid for silica [180], and calcination at 500 °C for carbon [181]) leading to a porous material with high surface area. Synthesis of hollow silica-based MIP composites required functionalization with KH-570 (source of vinyl groups) for an effective MIP synthesis and anchorage (estrogen recognition/pre-concentration from water [180]). However, TEOS and aluminum chloride were used for MIP synthesis (valsartan as a template) when preparing the hollow carbon-based aluminum-doped silica composite, promoting hydrolysis to generate silanol groups (Si−OH) followed by condensation of the silanols to obtain a polysiloxane (O−Si−O) [181]. Estrogen pre-concentration was designed by using 10 mg of the composite and shaking for 1.0 h [180]; whereas, valsartan and losartan isolation required 40 mg of adsorbent and sonication for 27 min [181].

Other composites such as MWCNT-MIPs have also been demonstrated to be effective adsorbents for dSPE of dioctyl phthalate in beverage samples [182]. Vinyl groups were incorporated on MWCNTs by reaction with sodium ethoxylate before MWCNT oxidation (presence of carboxyl groups), and MIP (dioctyl phthalate as a template) was further synthesized. The dSPE procedure was performed by mixing 60 mg of MWCNTs-MIPs with treated beverage samples (juice, dairy drinks, and carbonated drinks) and incubating at room temperature for 30 min on an oscillator [182]. Finally, graphene oxide-based MIPs (GO-MIPs) have also been used for dSPE when pre-concentrating bis(2-ethylhexyl) phthalate from waters by shaking 20 mg of adsorbent with the sample (water) at 600 rpm for 30 min [183].

3. Drawbacks and Future Prospects

MIMSPE procedures have been revealed as excellent approaches for miniaturization of SPE-based techniques in analytical chemistry, offering selective extraction/pre-concentration when analyzing complex samples. Dispersive SPE/μ-SPE procedures based on MIPs (mainly MMIPs) have shown high potential of miniaturization, which implies the use of low amounts of adsorbents as well as low volumes of organic solvents for performing the elution stage.

However, MIPs and MMIPs face a number of challenges during the preparation (synthesis) stage and also during the application. MMIPs are synthesized in nonpolar solvents to avoid the disruption of the hydrogen bonding between monomer and templates. The generated hydrophobic surfaces lead to adsorption of interferences such as proteins. RAFT polymerization is a good alternative to overcome this problem since it allows the preparation of highly hydrophilic MIPs (or MIP external layers over nanoparticles), which

can lead to efficient adsorbents for samples of a wide polarity range. Improvements have also been addressed to automate the techniques (similar to on-column/cartridges SPE) since batch MIMSPE procedures require several steps (conditioning, loading, washing, elution) and the procedures are not appealing processes when coping with hundreds of samples. In addition, the coupling (and also automation) of the MIMSPE devices directly with analytical instruments has not been explored yet.

In any case, MIMSPE procedures open a fascinating window to analyzing compounds from complex matrices, and continuous efforts in this research area should open more and more novel applications.

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