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Review

Research Progress on Molecularly Imprinted Materials for the Screening and Identification of Organic Pollutants

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Abstract: Organic pollutants, distinguished by their persistence and bioaccumulation in the environment, pose significant ecological and health threats that surpass those of traditional pollutants. Crucial to understanding their environmental behavior, health risks, and mitigation strategies, is the screening and identification of these pollutants. This process indispensably employs functional materials, among which molecularly imprinted polymers (MIPs) prove to be particularly advantageous because of their specific recognition capabilities and extensive application range. This review presents cutting-edge techniques and strategies for the fabrication of MIPs, including surface imprinting techniques and dummy molecular strategies. It encapsulates the last five years' advancements in MIP research within the domains of sample pretreatment, as well as optical and electrochemical sensing analysis. The objective of this discourse is to potentially foster the evolution of MIP technology and establish the groundwork for its transition from lab-scale to commercial production.

Keywords: organic pollutants; screening and identification; molecular imprinting; sample pretreatment; optical sensor; electrochemical sensor

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1. Introduction

Anthropogenic activities have resulted in the release of a vast array of organic pollutants into the natural environment. These pollutants, particularly unstable organic matter with high degradability, contaminate surface waters, leading to an overgrowth of aerobic and anaerobic microorganisms. Overgrowth, such as that observed in black and smelly urban rivers and algal blooms in eutrophic waters, contributes to the production of greenhouse gases [1,2]. For instance, approximately 48% of the 3.59×10^{11} m^3/year of wastewater produced globally is discharged untreated into the environment, with South and Southeast Asia being the major contributors. This untreated discharge correlates with the high prevalence of black and smelly surface water in these regions [3]. Persistent organic pollutants (POPs), such as polychlorinated biphenyls and chlorinated paraffins, are resistant to degradation and are often toxic or harmful. They can bioaccumulate through the food web, posing threats to human health and ecological balance [4,5].

Polluted water contains numerous contaminants, including antibiotics, dyes, phenols, and antioxidants. A study by He [6] highlighted the widespread use of sulfonamides because of their low toxicity and high antibacterial performance. However, the residual effects of these compounds have raised significant concerns. Long-term use of sulfonamides can cause side effects such as hematopoietic turbulence, allergies, and hypothyroidism [7,8].

As a kind of drug that can inhibit and kill bacteria, antibiotics have been widely used in medical and health, livestock and poultry breeding, agricultural production, and other industries. However, antibiotics can accumulate in the human body through the food chain, causing organ damage and hearing loss even at low doses [9]. In addition, the abuse of tetracycline antibiotics can increase bacterial resistance, reducing the efficacy of bacterial infection treatments [10]. Rhodamine B (RhB), an organic dye commonly used in the leather, textile, and printing industries, can cause chronic toxic symptoms when inhaled by humans, thus leading to its classification as a Group III carcinogen by the International Agency for Research on Cancer (IARC) [11].

Phenolic pollutants, often originating from industrial and agricultural activities, are highly toxic. For example, Bisphenol A (BPA), used in the manufacture of various food cans, baby bottles, and plastic containers, can interfere with the human endocrine system and cause DNA damage. Most nitrophenols have mutagenic effects [12,13]. Other POPs, such as per- and polyfluoroalkyl substances, polychlorinated biphenyls, dioxins, and furans, are characterized by their high toxicity, persistence, bioaccumulation, and long-distance migration. These pollutants pose immeasurable threats to ecosystems and human health [14].

Environmental substrates affected by organic pollutants include water, soil, atmosphere, plants, and animals, which are the focus of environmental studies [15,16]. Several analytical methods, such as high-performance liquid chromatography (HPLC), gas chromatography (GC), and liquid chromatography–mass spectrometry (LC-MS), have been used to quantitatively analyze organic pollutants in different substrates [17,18]. Kaykhaii et al. [19] synthesized a thorium metal–organic skeleton as an adsorbent to carry out trace solid-phase extraction of BPA in bottled drinking water samples for detection by HPLC and revealed accurate detection of very low concentrations of BPA (0.002–0.456 ng $\mathrm{mL^{-1}}$). The detection limit was reported to be as low as 0.001 ng $\mathrm{mL^{-1}}$. Similarly, Desmarchelier et al. [20] used LC-MS to screen tetracycline and its isomers in meat, fish, and seafood products and various dairy products. The results of this method were verified by European laboratory residue guidelines, with the false negative and false positive rates of compounds in 93 samples being 0%, indicating high accuracy. Although chromatographic methods exhibit high accuracy, they involve bulky instruments and skilled operators and have strict requirements for sample pretreatment and concentration [21,22]. Therefore, the development of simpler, faster, cheaper, and more reliable techniques for the analysis and detection of emerging organic pollutants in the environment is crucial. In this regard, molecularly imprinted polymers (MIPs), which offer structure–activity predetermination and specific recognition, have garnered significant attention in recent years.

2. The principle of MIPs

2.1. Concept of Molecular Imprinting Technology

Molecular Imprinting Technology (MIT) leverages MIPs to discern specifically imprinted molecules, also known as template molecules. Owing to their exceptional selectivity, recognition, and practicability, MIPs have found extensive applications in areas such as chromatographic separation, solid-phase extraction, bionic sensing, and simulated enzyme catalysis [23].

The fundamental principle of MIT bears similarity to the antigen–antibody-specific binding theory. The process of creating MIPs encompasses the following three stages: preassembly, polymerization, and template elution (Figure 1). Preassembly of the functional monomer and the template molecule is performed through covalent or non-covalent bonds to form host–guest complexes, facilitated by the selection of an appropriate solvent. Subsequently, the polymerization system is supplemented with a suitable quantity of crosslinking agents and initiators, leading to the formation of a highly crosslinked polymer with robust mechanical properties under optimal temperature or light conditions [24]. The ultimate step entails the removal of template molecules from the polymer through the employment of suitable eluents, culminating in the creation of pores that align with the size and shape of the template molecules. These pores house functional groups that can interact with the template molecules, thereby bestowing the imprinted polymer with a specific recognition function and creating a bionic recognition system akin to the antigen–antibody system.

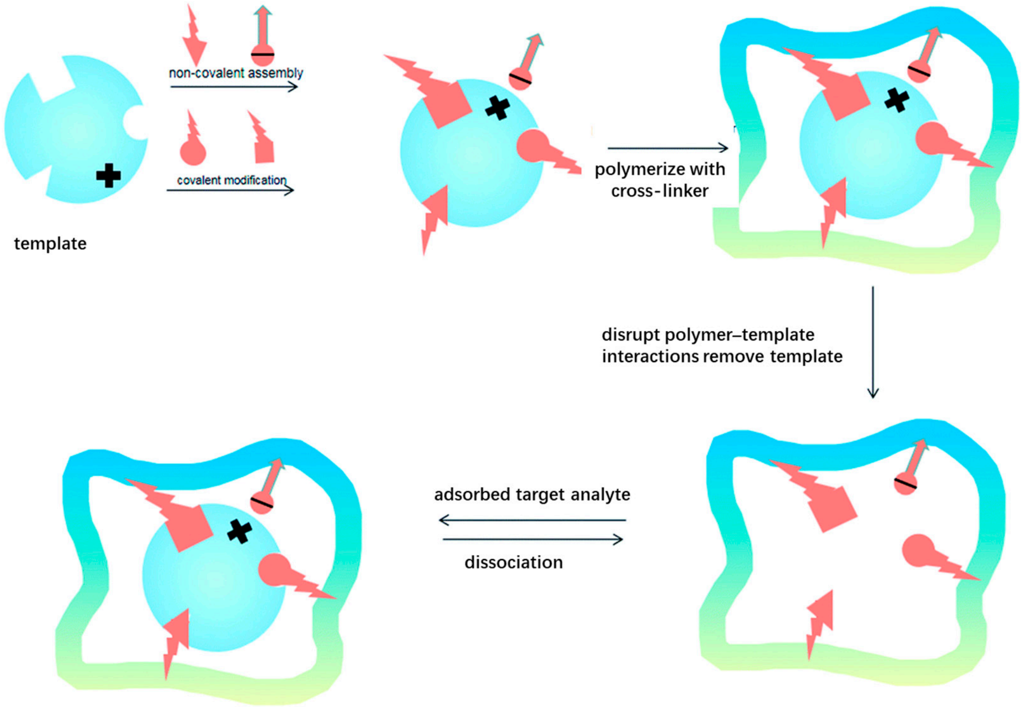

Figure 1. Schematic of the formation process of MIT. Reproduced with permission from [25].

2.2. Synthesis Mechanism of MIPs

MIPs are synthesized through a process that involves embedding template molecules during the copolymerization of functional monomers and crosslinkers. The initiation of polymerization is typically facilitated by thermal activation or ultraviolet activation of the initiator. Following elution, cavities are generated that mirror the shape and binding capacity of the template molecule. The process of polymerization consists of the following five components: template molecule, monomer, crosslinking agent, initiator, and solvent [26].

The interaction between the template molecule and the functional monomer can be categorized into three types as follows: covalent bonding, non-covalent bonding, and semi-covalent bonding. In covalent bonding, reversible covalent bonds are formed between the target molecule and the functional monomer. However, the covalent force between MIPs and the analyte presents challenges in achieving thermodynamic equilibrium and issues such as slow binding and difficult dissociation. Non-covalent bonding, on the other hand, involves electrostatic attraction, charge transfer, hydrophobic action, and van der Waals forces. This type of bonding offers more flexibility in the selection of template and monomer types. Because of its ease of bonding and template removal, non-covalent bonding is more commonly used. Mehdinia et al. [27] synthesized MIP using methacrylic acid (MAA) as a functional monomer and ethylene glycol dimethacrylate (EGDMA) as a crosslinking agent. The template molecule p-Nitrophenol (4-NP) was bound through hydrogen bonding. The MIP, when combined with magnetic nanoparticles, was used for selective extraction and detection of 4-NP, demonstrating high adsorption capacity, good selectivity, and rapid analysis.

Semi-covalent blot hybridization can reduce non-selective binding. In this method, the template molecule is covalently bound to the functional monomer before polymerization, and the analyte is recombined through non-covalent interactions to improve selectivity and shorten the equilibrium time. Wang et al. [28] synthesized MIP using EGDMA as a crosslinking agent and MAA monomer. The template (dichlorodiphenyltrichloroethane) was removed by hydrolysis under acidic conditions, leaving an active site cavity in the MIP because of the non-covalent interaction of hydrogen bonds. This polymer was able to quickly absorb dicofol in aquatic products with high specificity.

2.3. Preparation of MIPs

The choice of polymerization technique plays a vital role in the synthesis of MIPs with specific size and shape. A variety of polymerization methods are routinely employed, encompassing bulk polymerization, suspension polymerization, precipitation polymerization, and electro-polymerization. These methodologies each possess their own unique advantages and drawbacks, which are succinctly summarized in Table 1. Table 2 summarizes different categories of MIPs for the identification of emerging pollutants. The selection of an appropriate polymerization method, therefore, requires careful consideration and is contingent on the desired properties of the resultant MIPs.

Table 1. The summary of commonly used polymerization methods.

Method	Principle	Advantages	Disadvantages	Reference
Bulk polymerization	The template, functional monomer, crosslinking agent, and initiator are uniformly mixed in a solvent and sealed in a vacuum environment for crosslinking polymerization.	• Convenient operation; • Single system.	• Irregular shape; • Complex post-processing process; • Long adsorption time; • Difficult template elution.	[29,30]
Suspension polymerization	The organic phase composed of the template, functional monomer, crosslinking agent, and pore-making agent is dissolved in a solvent. After adding a dispersant, the solvent is sealed and stirred at high speed.	• Uniform particle size; • Stable polymer; • Easy to operate.	• Complex operation; • Difficult preparation process; • Susceptible to material properties.	[31]
Precipitation polymerization	The template, monomer, crosslinker and initiator are dissolved in the reaction medium for polymerization, and an insoluble molecularly imprinted microsphere (MIM) is formed in the reaction medium.	• Simple operation; • Large specific surface area.	• Need to use a lot of solvents; • High viscosity requirements for solvents.	[32,33]
Sol-gel method	In the presence of the template molecule, the inorganic precursor is dissolved in a low-molecular-weight solvent medium and then formed into a gel by hydrolysis and polycondensation.	• Mild operating conditions; • Convenient preparation; • Good thermal stability; • Strong chemical stability.	• The material is brittle; • The requirement for pH is high.	[34,35]
Electrochemical polymerization	The polymerization of monomers on the electrode is due to oxidation or reduction or decomposition into free radicals or ions by electrochemical electrolysis in an appropriate electrolyte cell.	• Simple device; • Adjustable polymer structure; • Uniform surface impress.	• Electrodes require modification materials to increase sensitivity.	[31,36]

Table 2. Different categories of MIPs for the identification of emerging pollutants.

Pollution	Polymerization Method	Linear Range	LOD	Reference
Fluoroquinolones	bulk polymerization	0.023–0.033 μg L^{-1}	-	[37]
Norfloxacin	precipitation polymerization	1–200 μg L^{-1}	0.67 μg L^{-1}	[38]
Sertraline antidepressant drug	suspension polymerization	5.0×10^{-9}–7.5×10^{-7} M	1.99×10^{-9} M	[39]
Amoxicillin	sol-gel method	-	73 pM	[40]
Bisphenol A	sol-gel method	-	0.015 ng μL^{-1}	[41]
Chlorpyrifos	electro-polymerization	1–218.92 μg L^{-1}	0.36 μg L^{-1}	[42]
2,4,6-Trinitrophenol	electro-polymerization	-	21.5 nM	[43]
Perfluorooctanesulfonate	electro-polymerization	0.1–4.9 nM	9.5 nM	[44]

Depending on the location of the recognition sites, molecularly imprinted polymers are mainly prepared by the embedding method and surface molecular imprinting method. The recognition sites of molecularly imprinted polymers prepared by the embedding method are mostly distributed in the interior of the polymer, and the sites on the surface of the polymer are less distributed. In practice, molecularly imprinted polymers prepared by the embedding method have problems such as the difficulty of removing template molecules, the high mass transfer resistance inside the imprinted polymer, and the small effective size. At present, the embedding method mainly refers to precipitation polymerization, suspension polymerization, and bulk polymerization.

2.3.1. Embedding Method

Bulk polymerization is recognized as the predominant method for MIP aggregation. This technique involves the pre-arrangement of functional monomers (e.g., MAA) around the template molecule within an organic solvent. Subsequently, copolymerization is performed with an excess crosslinking agent (e.g., EGDMA) under photochemical or thermochemical conditions or in the presence of a free radical initiator. A study by Mirzajani et al. [37] synthesized MIP fiber on a stainless-steel wire using ciprofloxacin as a template. This method was used for solid-phase microextraction of Fluoroquinolones (FQs) in biological fluids and drug samples. Detection limits for the four FQs ranged from 0.023 to 0.033 μg L^{-1} (S/N = 5), with concentration ranges from 0.1 to 40 μg L^{-1}. The sample pretreatment system exhibited high thermal stability (300 °C), robust reproducibility, and long service life. It was successfully applied to the quantitative analysis of FQs in serum, plasma, and tablet formulations of actual samples, with a recovery rate between 97% and 102%.

However, MIPs prepared by bulk polymerization present certain limitations, such as a complex and time-consuming preparation process, limited recognition sites, and subpar binding ability. To circumvent these challenges, precipitation polymerization has been extensively studied. The principle of this method is that once the polymer chain grown in the solution reaches a certain mass, it precipitates out of the solution, forming a highly cross-linked polymer microgel. MIP is then obtained after centrifugation [45]. Lu et al. [38] developed a novel type of Enrofloxacin and norfloxacin double-template molecular-imprinted polymer (dt-MIPs) using precipitation polymerization (Figure 2). When combined with high-performance liquid chromatography–diode array detector (HPLC-DAD), two FQ drugs were detected in actual water samples. The recoveries ranged from 80.9% to 101.0%, with relative standard deviations of 0.9–6.9%. Their study not only demonstrates immense potential in the determination of FQs but also contributes to the research on double/multi-template imprinting methods. A.T. Mohd Din et al. [46] showed that SiO_2 core–shell MIP particles prepared by precipitation polymerization (Si@MIPs-CAP) demonstrated a higher affinity for CAP than their unimprinted counterparts (NIPs-CAP and Si@NIPs-CAP). Hence, Si@MIPs-CAP, as an effective water-compatible adsorbent, can be used to remove CAP in the aqueous phase.

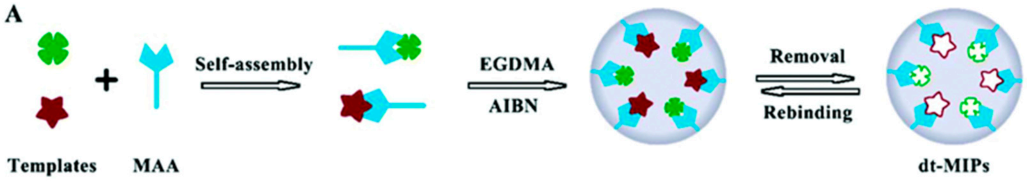

Figure 2. Schematic of dt-MIP preparation by precipitation polymerization. Reproduced with permission from [38].

Suspension polymerization is a type of heterogeneous polymerization in which droplets of the pre-polymerized mixture are suspended in a continuous phase, such as water, mineral oil, or perfluorocarbons. This method can produce spherical beads that range in size from a few microns to millimeters. Owing to its superior heat dissipation performance, suspension polymerization is particularly suited for industrial applications where heat transfer limitations are not a concern. Using ethylene glycol maleic acid acrylate as a crosslinking agent, acrylic acid as a functional monomer, and Camptothecin (CPT) as a template molecule, Wang et al. [47] prepared CPT MIP microspheres using membrane emulsification–suspension polymerization. The resulting MIPs exhibited a regular spherical surface with a porous surface, with particle size primarily ranging between 3 and 5 μm. A HPLC column prepared with MIPs as the stationary phase demonstrated high separation efficiency. Moreover, the MIP column was effectively applied to the separation and purification of CPT from the extract of Camptotheca acuminata fruit.

Although traditional covalent and non-covalent MIP preparation methods offer certain advantages, they also have notable limitations, including easy template leaching, poor thermal stability, and suboptimal repeatability. The sol-gel method is an effective means of compensating for these limitations, as it produces MIPs with high chemical and mechanical stability. This method uses a catalyst (acid, base, or ion) to dissolve the inorganic precursor in a low-molecular-weight solvent medium for hydrolysis and polycondensation. The use of strong and stable silicon-based materials in the sol-gel method enables the production of longer-lasting imprinted selective cavities, thereby increasing service life. Wang et al. [48] used 3-aminopropyl triethoxysilane as a functional monomer to synthesize an efficient MIP on carbon quantum dots (MIP@CQDs) using the sol-gel method, as shown in Figure 3. The study found that MIP@CQDs could be utilized for the quantitative detection of trace 4-NP in tap water, wastewater, and seawater, with recovery rates ranging from 95.1% to 107.8%. The detection range was 0–144 μmol L^{-1}, and the detection limit was as low as 0.41 μmol L^{-1}.

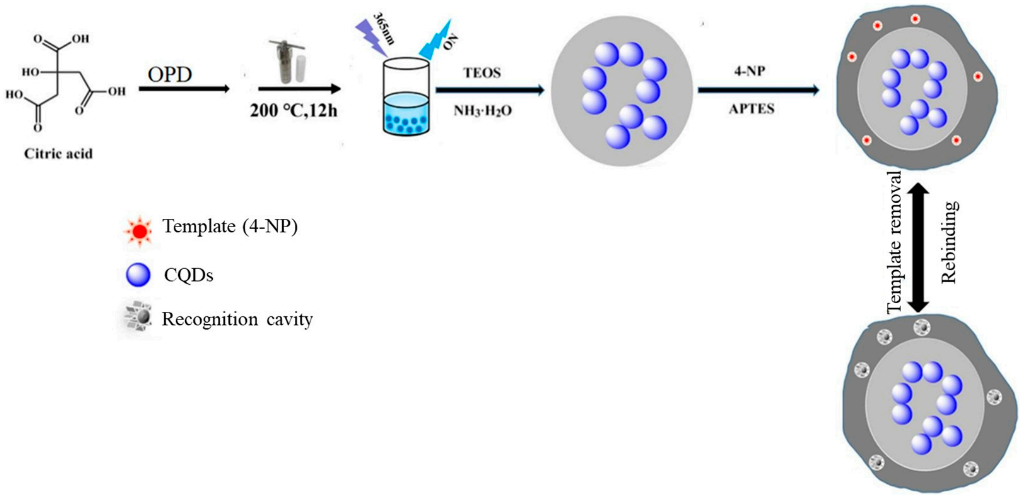

Figure 3. MIP@CQDs as the synthesis process for 4-NP sensing [48].

2.3.2. Surface Molecular Imprinting Method

With the development of molecularly imprinted polymer preparation methods, it has been realized that there are drawbacks such as the large particle size of the prepared imprinted polymer, uneven distribution, and low imprinting efficiency of the bulk polymerization method. Although suspension polymerization, precipitation polymerization, and other methods of preparing polymer microspheres have a greater improvement than the method of bulk polymerization and can obtain a uniform distribution of particles and a large specific surface area of the imprinted polymer, there are still shortcomings such as the template molecules are difficult to elute and there are few effective recognition sites. Therefore, surface molecular imprinting preparation technology has emerged and become a research hotspot.

Surface imprinting refers to the technique of preparing imprinted polymers by carrying out polymerization reactions on the surface of specific carriers and controlling the distribution of imprint recognition sites on the polymer surface or the carrier surface. The preparation methods of surface molecularly imprinted polymers mainly include the carrier sacrifice method, the electropolymerization method, and the carrier method. Among them, the electropolymerization method is a method of directly generating an imprinted polymer film by using electrochemical methods to make the functional monomers undergo a condensation reaction on the surface of the electrode in the presence of a template molecule and a functional monomer. The imprinted polymerized membrane prepared by this method is thin, and when the imprinted molecules are removed, the binding sites are uniformly distributed on the surface of the polymerized membrane, which can effectively improve the mass transfer rate of the responding molecules. In addition, the method can reduce the non-specific adsorption and the phenomenon of template molecule encapsulation. The thickness, pore size, and morphology of molecularly imprinted membranes prepared by electropolymerization can be achieved by controlling the experimental parameters (e.g., polymerization substrate, solution pH, supporting electrolyte, and functional monomers). Therefore, electrochemical polymerization is a simple, convenient, and controllable method for the preparation of imprinted membranes.

Willner et al. [49] successfully prepared a surface plasmon resonance sensor using the method of electropolymerization, which can successfully detect organic trinitrotoluene. For the detection of thiophenol, the detection limit of nano-gold electropolymerization is one order of magnitude lower than that of direct polymerization. Liu et al. [50] polymerized dopamine on the surface of a gold electrode to prepare a capacitive sensor of a molecularly imprinted polymer, which has excellent selectivity and recognition performance for nicotine, and its detection range is from 1.0 to 25.1 μmol L^{-1}. Although many scholars have successfully prepared molecule-imprinted polymers in the field of electropolymerization, the properties of the polymers are not ideal, and their crosslinking degree is low and relatively unstable. In addition, there are relatively few functional monomers that can be used for electropolymerization, including a few substances such as phenol, thiophene, aniline, dopamine, and pyrrole.

3. Application of MIPs in the Screening and Identification of Emerging Pollutants

In recent years, the application of MIT in the analysis of organic pollutants such as phenolic compounds, antibiotics, triazines, organic dyes, and synthetic hormones has gained significant traction. Figure 4 introduces prevalent techniques employed for the detection of organic pollutants based on MIT, including four methods for the pretreatment and quantification of organic pollutants [51]. Among them, MIP–Solid-Phase Extraction (MIP-SPE), Magnetic MIP (MMIP), and Dummy MIP (DMIP) are primarily used for sample pre-processing and concentration. On the other hand, the MIP sensor represents a detection method that amalgamates MIP with a variety of sensors. This technique serves as a testament to the versatility and adaptability of MIT in the field of organic pollutant sensing (Table 3).

Table 3. Application for detecting emerging organic pollutants based on MIPs.

Analytes	Used Technologies	Detection Techniques	Linear Range	LOD	Sample	Reference
Pentazolol, triazolone, triazolol	SPE	UPLC-MS/MS	0.0005–0.1 mg/L	-	Tobacco leaf	[52]
Polychlorinated biphenyls	SPE	GC-MS/MS	-	0.003–2.705 μg/kg	-	[53]
Polycyclic aromatic hydrocarbons	SPE	GC-MS/MS	-	1–100 pg/mL	Tap water	[54]
Diazinon	MMIPs	GC	0.02–20,000 ng/mL	0.005 ng/mL	Urine	[55]
Organophosphate pesticide	MMIPs	HPLC	0.5–2000 mg/L	0.062–0.195 mg/L	Vegetables and fruits	[56]
Triazine pesticides	MMIPs	UPLC-MS/MS	0.05–50 ng/g	0.005–0.02 ng/g	Corn	[57]

Table 3. *Cont.*

Analytes	Used Technologies	Detection Techniques	Linear Range	LOD	Sample	Reference
Nonyl phenol	MMIPs	HPLC	0.1–50 mg/L	0.1–0.3 μg/L	Lake water, milk	[58]
Bisphenol A	DMIP	-	0.1–5 μg/mL	0.03 μg/mL	Tap water	[59]
Disinfection by-product	DMIP	HPLC	-	2.3 ng/mL	Tap water	[60]
Bisphenol A	MIP electrochemical sensor	CV	0.05–50 nmol/L	0.015 nmol/L	Tap water, lake water	[61]
POPs	MIP electrochemical sensor	CV	0.21–300 nmol/L	0.07 nmol/L	lake water	[62]
PFAS	MIP electrochemical sensor	CV	0.025–2.5 μg/L	0.0075 μg/L	Potable water	[63]
Erythromycin	MIP electrochemical sensor	DPV	-	0.1 nM	Tap water	[64]
Cefixime	MIP electrochemical sensor	EIS	20.0–950.0 nM	7.1 nM	Urine, serum	[65]
Bisphenol A	MIP optional sensor	Fluorescence	-	12 μg/L	Beverage	[66]
Simazine	MIP optional sensor	PEC	0.1–500 μg/L	0.06 μg/L	Lake water	[34]
PFAS	MIP optional sensor	PEC	-	0.01 μg/L	Potable water	[67]

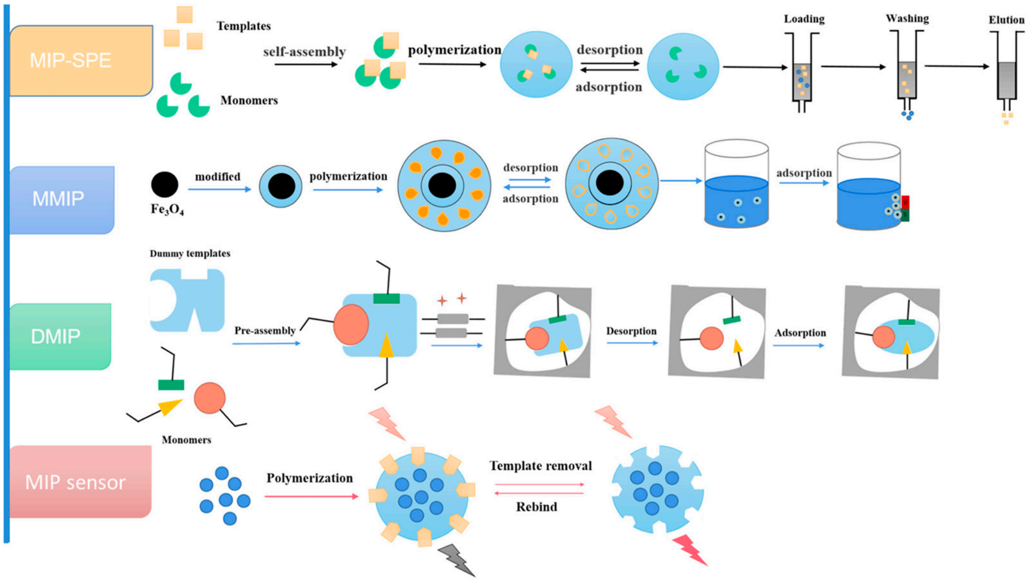

Figure 4. Schematic of four commonly used technologies for detecting organic pollutants based on MIT. Reproduced with permission from [51].

3.1. Sample Pretreatment

3.1.1. SPE

SPE is recognized as an efficient method for sample pretreatment. It includes the extraction of analyte, enrichment, and elimination of matrix interference, thereby enhancing the sensitivity and selectivity of the analysis. The purification analysis technique and preconcentration are crucial for analyzing samples of low concentration [68]. A combination of effective sample pretreatment technology and an appropriate instrumental analysis method can facilitate the identification of virtually all compounds within a sample.

SPE is often chosen as a sample preparation method because of its time efficiency and reduced solvent requirement compared with traditional liquid phase extraction. It also offers superior reproducibility and selectivity when analyzing multiple groups. For optimal results, the choice of SPE adsorbents becomes critical. Traditional SPE adsorbents such as C8 and C18 have disadvantages such as poor selectivity, a narrow application range, small adsorption capacity, and low extraction efficiency [64]. To say the least, molecularly imprinted solid-phase extraction (MISPE) can not only improve the selectivity and enrichment capability of target molecules but also greatly improve the accuracy of quantitative analysis and lower the detection limit [69]. Jing et al. [52] prepared MIPs using pentazolol (TBZ) and triazolone (TDF) as template molecules by a multi-template imprinting strategy and used them as SPE fillers for the pretreatment of tobacco leaves. A method was established for the simultaneous detection of TBZ, TDF, nitrazole, and triazolol residues in tobacco leaves. The method is simple, highly selective, and can achieve accurate analysis of triazole fungicide residues in tobacco leaves. Azizi et al. [54] prepared MMIPs for SPE by polymerization on a magnetic $Fe_3O_4@SiO_2$ NP surface and successfully enriched 16 kinds of polycyclic aromatic hydrocarbons (PAHs) in water samples. This method is suitable for screening PAHs of various concentrations in water samples in complex environments. Combined with instrumental analysis, it can provide a highly selective and sensitive monitoring method.

3.1.2. Magnetic MIPs

Magnetic MIPs (MMIPs) have emerged as a highly efficient and rapid material for sample pretreatment in separation processes, primarily because of their ability to be effortlessly separated from the sample using an external magnet [70]. This characteristic places them at an advantage over traditional MIPs, as MMIPs can interact with the target analyte more effectively and swiftly. Additionally, they exhibit high magnetic sensitivity, require shorter pretreatment time, and enable the recovery of magnetic nanomaterials via applied magnetic fields, thus qualifying as superior adsorbents for Dispersive Solid-Phase Extraction (DSPE).

The fabrication of MMIPs involves the incorporation of magnetic nanoparticles (MNPs), typically Fe_3O_4, into imprinted polymers. The popularity of Fe_3O_4 as the magnetic material of choice can be attributed to the abundance of -OH groups on its surface, which easily facilitate further modifications. The polymer shell is then coated on the magnetic Fe_3O_4 by chelating various functional groups in the polymer, such as hydroxyl and amino groups, with iron ions [71].

MMIPs overcome the challenge of separating traditional MIPs from matrix samples and increase the number of imprinting sites via surface polymerization. Given their simplicity, convenience, and speed, MMIPs have become a prevalent sample pretreatment material and method for preconcentration and extraction of organic pollutants [72].

The solvothermal method and co-precipitation method are used to synthesize Fe_3O_4, with the latter being more widely used because of its simplicity, low equipment requirements, and mild reaction conditions. For instance, as shown in Figure 5, $Fe_3O_4@SiO_2$ magnetic nanoparticles have been prepared using TEOS and 3-methylpropenoxy propyl trimethoxysilane and subsequently polymerized to form MMIPs. Electrodes employing MMIPs have demonstrated lower detection limits (LOD = 10 nM) in river water and industrial wastewater samples [73].

Fe_3O_4-SiO_2 core–shell MMIPs were synthesized using a surface imprinting technique for bisphenol-A with 4-VP at a magneto-actuated electrode, providing its rapid detection in a variety of environmental samples [74]. In an aqueous solution, water-compatible MMIPs were developed using 3-aminophenylboronic acid (APBA), a water-soluble FM, and hexadecyl trimethyl ammonium bromide (CTABr) surfactant for Diethylstilbestrol (DES). A composite of chitosan and MWCNTs (MWCNTs@CS) was applied to the surface of a magnetic GCE to enhance the electron transfer rate and electrode surface area. The coating of MIPs on a magneto-actuated GCE resulted in MIPs/MWCNTs@CS/CTABr/MGCE. The MWCNT@CS provided a larger surface area for loading a substantial quantity of MIPs, while the CTABr surfactant solubilized DES, thus enhancing sensitivity. The sensor demonstrated excellent selectivity with a faster response time in lake water samples [75]. The MMIP-based sensor exhibited decent LODs in the nM range and benefits of easy separation and reusability, which are important for cost-effectiveness. High surface functionalization can enhance binding capacity and signal response, but it can also create a thick layer that interferes with magnetism. Therefore, it is necessary to develop robust methods that allow for strong magnetism and better surface group coverage. The stability of the sensor is a critical function that can reduce the cost of MMIP-based sensors.

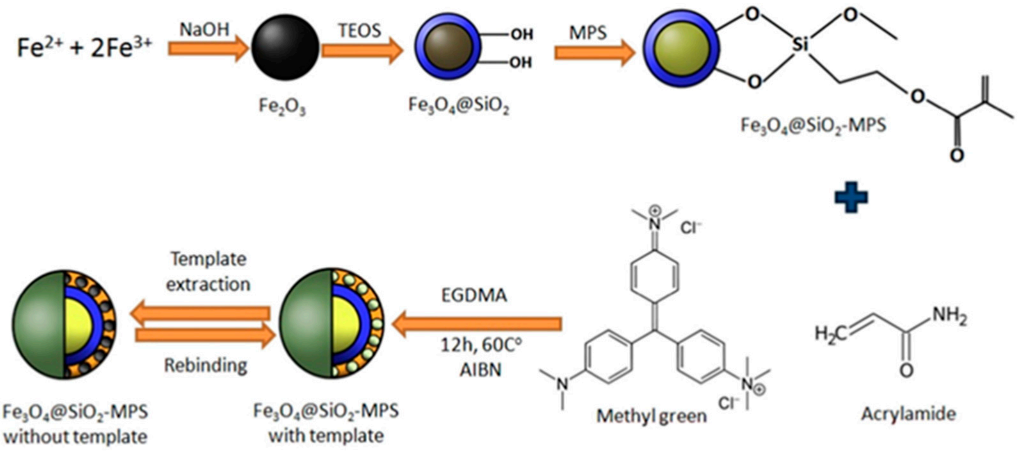

Figure 5. Schematic of methyl green-imprinted magnetic nanoparticles of $Fe_3O_4@SiO_2$ MMIPs. Reproduced with permission from [73].

3.1.3. Dummy MIPs

As can be seen, MIPs have garnered significant interest because of their exceptional selectivity. However, a notable drawback associated with MIPs is the leakage of template molecules. During the synthesis of MIPs, a considerable amount of template molecules becomes embedded within the imprinted polymers, and these cannot be entirely eluted [76]. Instead, they are gradually released into the sample, leading to sample contamination and a compromise in analytical accuracy. Dummy MIPs (DMIPs) are used to substitute the target molecule in the synthesis of MIPs. DMIPs not only retain the selective recognition of target molecule characteristics of MIPS, but they can also differentiate between dummy templates and target molecules through distinct chromatographic behaviors (chromatographic retention time), thereby avoiding interference from template leakage [77].

A dummy template becomes necessary when the target analyte (i) is found in low quantities in the sample; (ii) is extremely unstable and sensitive to light, temperature, oxygen, and other factors; (iii) is highly toxic, with low safety levels, making experimental operations challenging; (iv) contains unstable functional groups in its structure during imprinting polymerization; (v) is prone to template leakage during the imprinting process because of incomplete elution and dissociation of compounds.

Xu et al. [78] employed a DMIP-coated barbell stir bar adsorption extraction method to analyze BPA in water. The DMIP-coated stir bar was prepared by in situ capillary polymerization using 2,2-bis (4-hydroxyphenyl) butane (BPB), 4,4′-dihydroxydiphenyl methane (BPF), 4-tert-butylphenol (PTBP), and tetrabrombisphenol A (TBBA) as dummy templates. The method is simple, user-friendly, and repeatable. The new DMIP-coated stir bar showed excellent selectivity for the target analyte BPA. The detection limit of BPA was 0.003 $\mu g\ L^{-1}$. The DMIP-coated stir bar is suitable for trace BPA analysis in real-environment water samples without template leakage and can be used at least 100 times.

Micro disinfection byproducts (DBPs) produced during the disinfection of drinking water have potential carcinogenicity, teratogenicity, and mutagenicity. Li et al. [60] prepared a molecularly imprinted solid-phase microextraction (MIP-SPME) fiber coating through in situ polymerization of dummy template molecules for the analysis of trace 2, 6-dichloindole-1, 4-benzoquinone (2,6-DCBQ) (Figure 6). The results indicated that the single SPME fiber prepared under optimized conditions possesses a porous structure, a large surface area, and good thermal stability. Given the strong structural recognition ability and molecular interaction with target molecules of the MIP-SPME, its imprinting factor for 2,6-DCBQ was 4.7, and it exhibited excellent extraction selectivity. A sensitive method for the determination of trace 2, 6-DCBQ in water samples by HPLC and UV detection was established, with the detection limit reduced to 2.3 ng mL^{-1}. Their recoveries were reported to be 84.4–122%, and the relative standard deviations were 1.0–13% (n = 3).

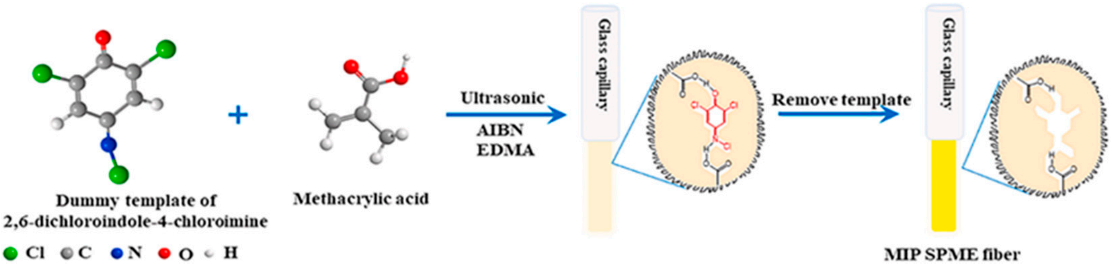

Figure 6. Preparation of 2,6-DCBQ MIP SPME fiber coating. Reproduced with permission from [60].

3.2. Molecularly Imprinted Electrochemical Sensor

The burgeoning interest in molecularly imprinted electrochemical (MIEC) sensors is attributable to their inherent advantages, including high sensitivity, high selectivity, low cost, ease of miniaturization, and automation [71–73]. These attributes make them particularly effective in the detection of organic pollutants. At present, the field of MIEC sensing is primarily focused on the design and development of new MIT for electrochemical sensors [79]. The aim is to enhance the electron transmission rate and improve the sensitivity and selectivity of these sensors.

MIEC sensors have found extensive application in the screening and identification of phenolic compounds [75–77]. Kaya et al. [80] successfully prepared a porous MIEC for the selective determination of bisphenol S (BPS). This was achieved by introducing N-methylacrylyl-L-tyrosine functional monomer. The sensor exhibited high sensitivity and excellent selectivity in the concentration range of 1–10 fM. The current peak of the sensor decreased significantly with an increase in BPS concentration, demonstrating a detection limit of 0.171 fmol L^{-1}.

Sarpong et al. [81] used surface imprinting technology and a bifunctional monomer imprinting strategy to imprint tetrabromobisphenol S (TBBPS) in the presence of MAA and 4-aminophenthiophenol bifunctional monomers. The resulting MIEC sensor successfully determined TBBPS in tap water, drinking water, and lake water, with an LOD of 0.029 nmol L^{-1}. As shown in Figure 7, Xu et al. [82] modified MIPs on the surface of functionalized carbon felt (CF) electrodes using simple I situ thermal polymerization and surface imprinting techniques. They developed a flexible electrochemical sensor based on the synthetic receptor of BPA, With a linear range of 0.5–8.0 nmol L^{-1} and a LOD of 0.36 nmol L^{-1}. The sensor demonstrated stable detection performance even after repeated bending and stretching, indicating its adaptability for installation on both flat and curved surfaces for field detection. The MIEC sensor was successfully used for the identification of BPA in milk, yielding satisfactory results. Their work provides a promising platform for the design of implantable, portable, and miniaturized sensors.

In the realm of sensor development, the predominant reliance on plasticized polyvinyl chloride (PVC) film has been observed. However, this material is not without its drawbacks, the most significant of which is the issue of plasticizer leaching. This problem prompted researchers to seek alternatives. A noteworthy development in this regard is the work of Liu et al. [83], who have pioneered the use of a copolymer of methyl methacrylate and 2-ethylhexyl acrylate as the sensing film (Figure 8). This has enabled the construction of a molecular-imprinted potential sensor that does not require a plasticizer. The sensor has demonstrated commendable sensitivity and selectivity, with an LOD of 32 nmol L^{-1}. This innovation has the potential to supersede the traditional PVC membrane sensor and offers a promising approach for the development of membrane-based MIEC.

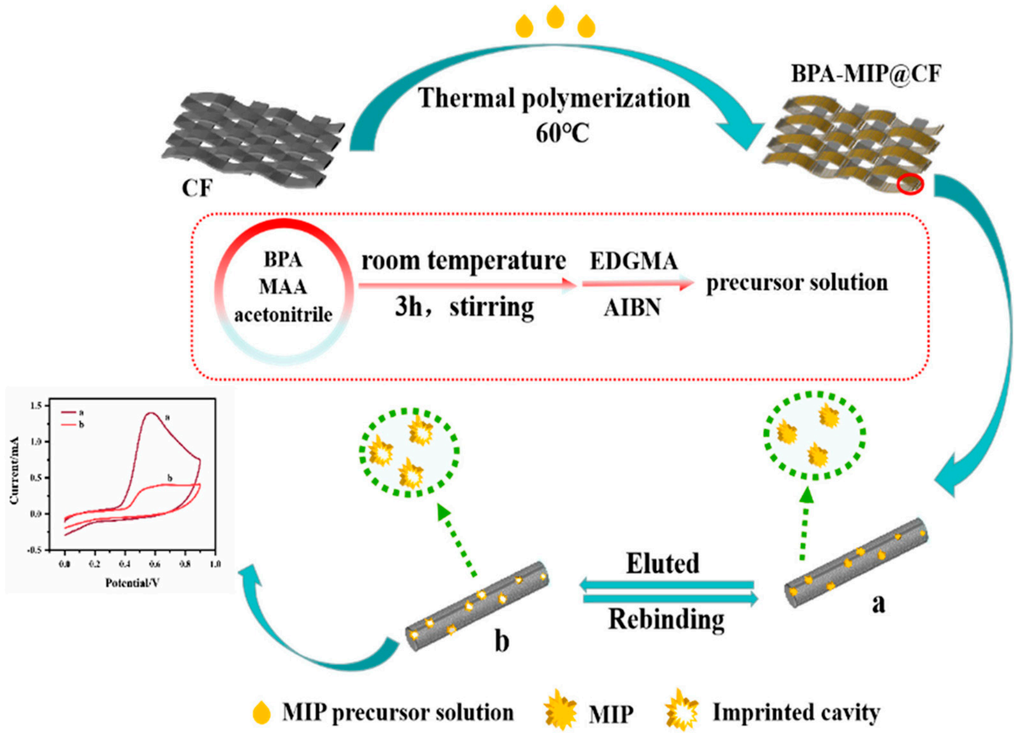

Figure 7. The preparation of an MIP@CF sensor for the detection of BPA. Reproduced with permission from [82].

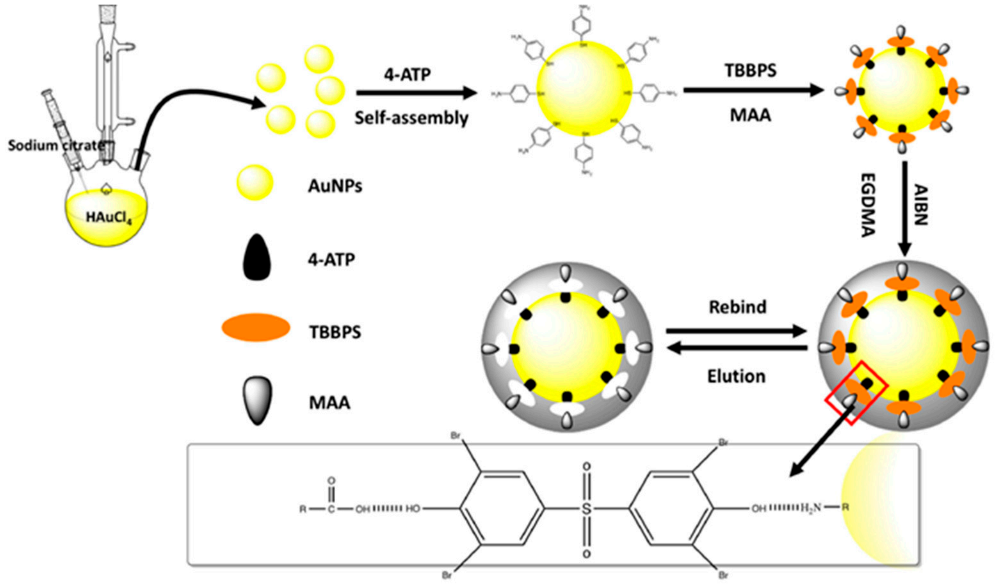

Figure 8. Representation of the steps involved in the preparation of MIP dimonomers at AuNPs. Reproduced with permission from [83].

In another seminal work, Huang et al. [61] prepared MIPs through the process of grafting carboxylated quantum dots onto aminated multi-wall carbon nanotubes. These modified nanotubes serve as the carrier, while BPA was utilized as the template and 3-aminopropyl triethoxysilane (APTES) as the monomer. The resulting MIEC sensor demonstrated successful application in detecting BPA in various water sources. The electrode exhibited high selectivity and robust anti-interference ability towards BPA. The current response demonstrated a linear range of 0.05–50 nmol L^{-1}, with a LOD of 0.015 nmol L^{-1}. This sensor holds the potential for advancing research in the detection of trace pollutants.

Furthermore, MIEC sensors are widely used in detecting POP residues, including pesticides and insecticides [62]. In a related study, Maria et al. [62] prepared an imprinted polymer film for 2,4-dichlorophenol (2,4-DCP) detection. This was achieved by electropolymerizing 3,4-ethylenedioxythiophene onto a carbon fiber paper electrode. A highly selective MIEC sensor was produced and achieved high selectivity in determining 2, 4-DCP in different water sources. The sensor response range was noted to be 0.21–300 nmol L^{-1}, with an LOD of 0.07 nmol L^{-1}. Ayankojo et al. [64] prepared an electrochemical sensor based on MIP for detecting erythromycin in water-based media. The sensor could be used to monitor this antibiotic as well as other macrolides in environmental water. The sensor was prepared by direct synthesis of erythromycin-selective MIP using the electrochemical polymerization of m-phenylenediamine on a screen-printed electrode. The resulting MIP sensor could successfully distinguish erythromycin in tap water and PBS from its very close analogs (azithromycin and clarithromycin) with an LOD of 0.1 nM. In another work, Dehghani et al. [65] used an MIP-based electrochemical sensor to determine cefixime in biological samples (urine and serum). They modified the surface of the GCE with gold nanowires and expanded graphene oxide, and then electropolymerized the polyaniline-based MIP layer. CV, DPV, and EIS technologies were used to analyze and characterize the sensor, and a linear response was obtained at a concentration range of 20.0–950.0 nM and a LOD of 7.1 nM.

3.3. Molecularly Imprinted Optical Sensor

Optical sensors are designed to measure the optical properties of various materials and transform alterations in light into electronic signals. A key component of this process is the detection of change in the optical signal and its subsequent conversion. In recent years, fluorescence detection has emerged as a widely utilized technology, largely because of its simplicity and sensitivity [84]. One of the most promising applications of this technology is the use of semiconductor quantum dots (QDs) as probes of fluorescence sensors. This is because of their unique optical properties, particularly their narrow emission and resistance to fluorescence quenching.

The integration of MIT, QDs, and fluorescence detection significantly enhances the potential applications of a sensor [76,85]. An important study by Li et al. [86] introduced a novel method for the preparation of ratio-fluorescent MIPs (Figure 9). The researchers grafted a green fluorescently labeled 2,4-dichloropphenoxyacetic acid MIP layer onto the pre-formed red CdTe QD-labeled SiO_2 microspheres, leading to the production of ratio-fluorescent MIPs. This innovative strategy lays a solid foundation for the effective development of a broad spectrum of complex biological sample-compatible proportional fluorescent MIPs. Such advancements hold significant promise in biological analysis and diagnosis.

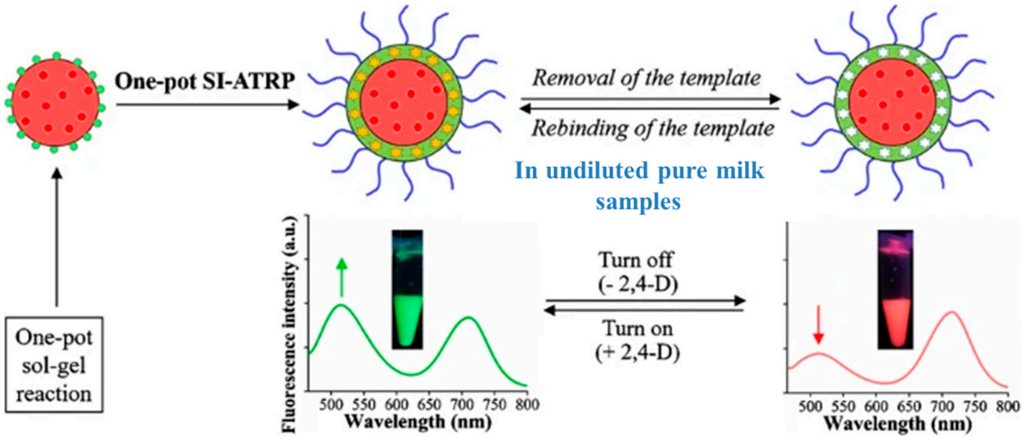

Figure 9. The preparation process of ratiou-fluorescent MIPs. Reproduced with permission from [86].

In their study, Fan et al. [85] employed Mn:Zn QDs as a fluorescence source to fabricate a core–shell molecularly imprinted phosphorescence sensor on the ZIF-8 surface. This was performed to attain high sensitivity and selectivity in the detection of chlorpyrifos (CPF). The phosphorescence intensity at room temperature showed a rapid response to CPF within 5 min, with an LOD of 0.89 mmol L^{-1}. This method amalgamates the benefits of phosphorescence emission and MIT, paving a new path for the identification and detection of water contaminants with high sensitivity and selectivity.

However, it is important to note that traditional QDs pose certain limitations, such as biological toxicity and environmental pollution. This has led to a growing interest in carbon points (CDs), which exhibit great characteristics like stable photoluminescence, green synthesis, and biocompatibility. Considering the challenges in preparing the MIP layer on CDs, such as time consumption and low controllability of the imprinting layer, mussel-inspired dopamine (DA) self-polymerization may be considered as an alternative approach. DA, as a functional monomer in molecular imprinting techniques, can be efficiently polymerized under mildly alkaline conditions to form polydopamine.

Bhogal et al. [87] developed a self-polymerizing molecularly imprinted sensor (MIS) based on DA on fluorescent CDs to suppress the fluorescence intensity of CDs through photoinduced electron transfer (Figure 10), achieving an LOD for 17β-estradiol (E2) of 0.34 ng mL^{-1} with a linear range of 1–50 ng mL^{-1}. The sensor was successfully applied to tap water, river water, and milk samples, yielding high recovery (96.4–102%). This provides a new avenue for the simple detection of E2 in complex substrates.

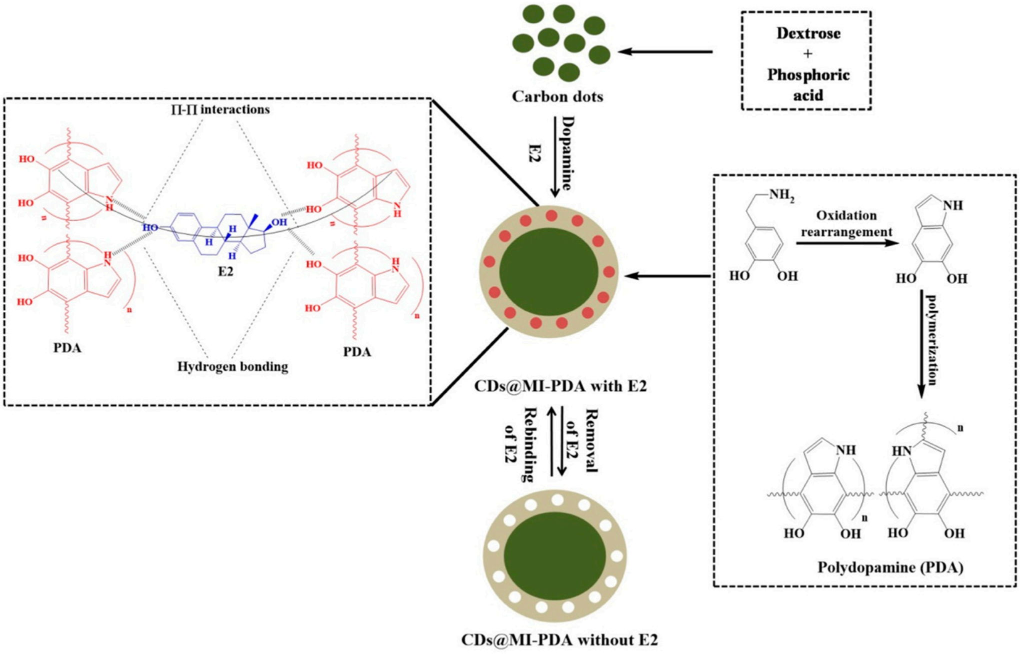

Figure 10. Schematic of the synthesis procedures for polydopamine-imprinted coatings over CDs (CDs@MI-PDA) for the selective fluorescence detection of 17β-oestradiol. Reproduced with permission from [87].

Wang et al. [66] proposed a one-pot synthesis strategy based on magnetic covalent organic skeleton support (MCOF) and CDs embedded in MIPs (Figure 11). The constructed MCOF@CDs@MIPs sensor, characterized by its high sensitivity and selectivity, was efficaciously used for the detection of BPA in canned food and boxed beverages, boasting an LOD of 12 μg L^{-1}. In their study, MCOFs were synthesized via a facile in situ growth of COF crystals on Fe_3O_4 nanoparticles. This was subsequently supported by a sol-gel molecular imprinting process carried out at ambient temperature, thereby providing a large surface area for the deposition of CDs embedded in the MIP layer. The fluorescence sensing platform, constructed with renewable magnetic COF@MIP composite material, integrates the magnetism of Fe_3O_4, the signal transduction functionality of CDs, the signal amplification effect of COFs and MIP, and the specific recognition behavior of MIP for the template molecule BPA. This innovation provides a simplistic, economical, and reliable method for the screening, identification, and detection of new contaminants in food or the environment.

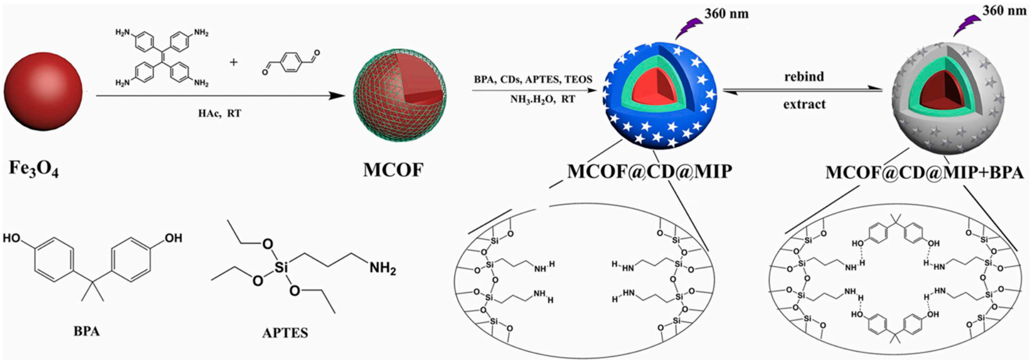

Figure 11. The fabrication of the MCOF@CD@MIP sensor for BPA. Reproduced with permission from [66].

The speed of detection is a key consideration when searching for methods to screen and identify new pollutants. The advent of portable paper-based MIS presents a viable solution to the challenges associated with rapid field detection. Dolai et al. [88] pioneered the development of a nanocomposite material derived from paper (Figure 12). The unique feature of this material lies in the molecular imprinting within a materialist structure, which facilitates the selective capture of dibutyl phthalate (DBP). Moreover, the incorporation of graphene oxide into the material design enabled fluorescent "on" detection of DBP. This was achieved through competitive binding interactions with fluorescein. The paper-based nanocomposite material, underpinned by MIPs, can realize the rapid detection of DBP in sewage. This approach holds significant potential for broad applications, particularly in the identification and screening of other environmental pollutants. These findings underscore the transformative potential of paper-based MIS in addressing contemporary challenges.

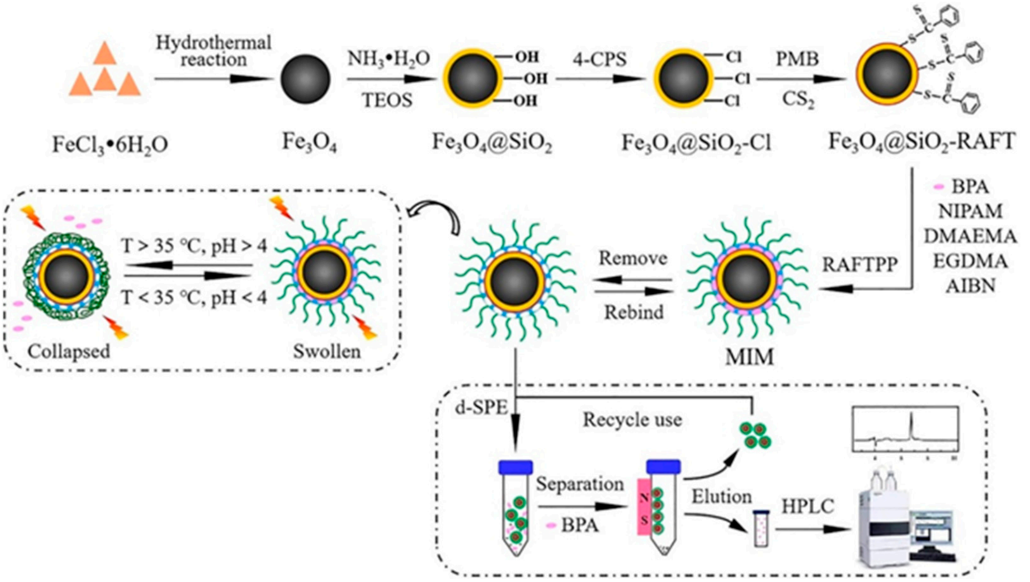

Figure 12. The preparation and extraction processes, and the possible stimuli–response mechanism using MIMs regulated by pH/temperature. Reproduced with permission from [88].

The electrochemical luminescence (ECL) detection method transmutes electrochemical signals into optical ones, which further advances the field of sensing technology. The integration of photoelectric characteristics into this method has been found to enhance sensing performance. A groundbreaking research study by Pan et al. [89] led to the development of a unique molecularly imprinted electrochemical luminescence (MIECL) sensor. This sensor was designed based on the luminescent properties of an MIP perovskite (MIP-CSPBBR 3) layer, and $Ru(bpy)_3{}^{2+}$ and was specifically tailored for the detection of simazine. As depicted in Figure 13, the MIP-CsPbBr 3 layer was firmly affixed to the surface of a GCE, serving as a signal capture probe $Ru(bpy)_3{}^{2+}$. The coreactant tripropylamine exhibited a strong ECL. Under optimal conditions, the MIECL sensor demonstrated a detection range of 0.1–500 μg L^{-1}, with an LOD of 0.6 μg L^{-1}. The MIECL sensor outperformed traditional detection methods in terms of sensitivity and detection speed. Moreover, it showed excellent selectivity, sensitivity, and reproducibility in the real-world analysis of aquatic products, further underscoring its potential for practical applications.

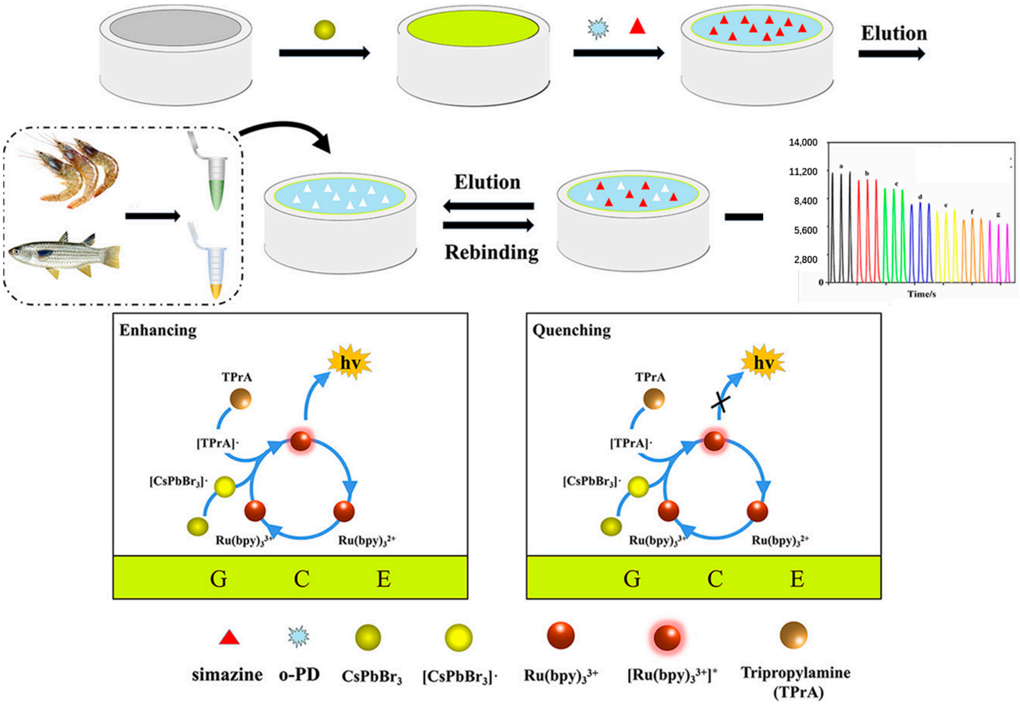

Figure 13. The preparation process of MIECL. Reproduced with permission from [89].

The detection mechanism of photoelectrochemical (PEC) sensors is predicated on the conversion of photons into electricity during illumination, resulting in light-induced charge separation. The molecularly imprinted PEC (MIPEC) sensor is a promising detection method as it combines the benefits of optics, electrochemistry, and MIT [71,90]. A study conducted by Wang et al. [90] resulted in the development of a highly sensitive and selective PEC sensor. This was achieved by integrating TiO_2/CdS heterostructures with inorganic frame MIT (Figure 14). The molecularly imprinted TiO_2/CdS (MI-TiO_2/CdS) heterostructures were synthesized using a continuous ion layer adsorption process combined with inorganic skeleton molecular imprinting. The formation of the heterojunction, facilitated by the energy distribution compatibility of TiO_2 and CdS, promoted photogenic charge separation and enhanced PEC transformation. The fabricated PEC sensor exhibited a high photocurrent response and good selectivity towards BPA under simulated sunlight irradiation. This unique heterostructure and specific recognition ability of MI-TiO_2/CdS resulted in a linear relationship between the photocurrent and the concentration of BPA (1–100 pmol L^{-1}) and the minimum detection limit of 0.5 pmol L^{-1}.

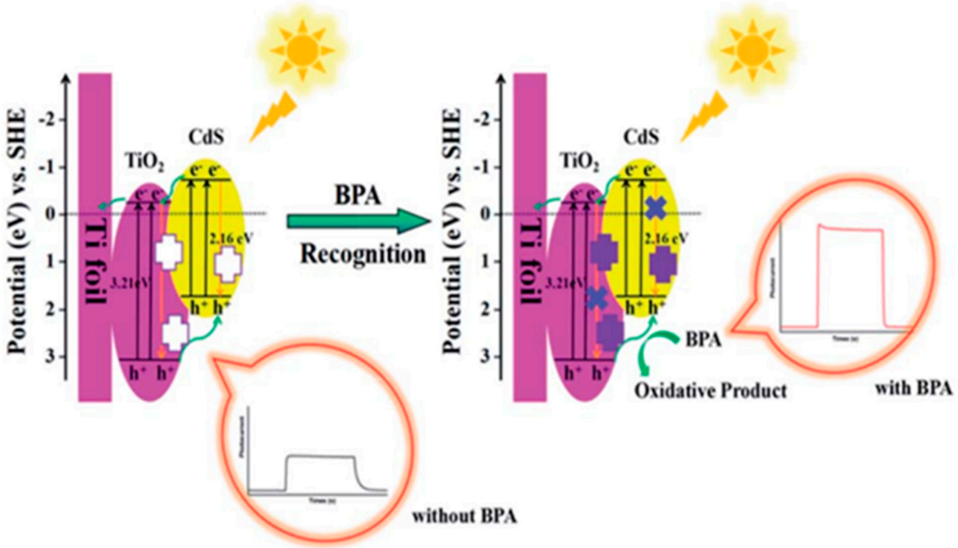

Figure 14. Fabrication of MI-TiO$_2$/CdS-based PEC sensor for BPA detection. Reproduced with permission from [90].

Xu et al. [91] prepared a PEC sensor by modifying an indium tin oxide (ITO) photosensitive electrode with MIPs based on In$_2$S$_3$/Cd^{2+}-sensitized flower-like AgBiS$_2$. The detailed preparation process of the PEC sensor is illustrated in Figure 15. The photosensitive electrode was sequentially modified on an ITO electrode, and the MIM was fabricated by electrodeposition of pyrrole solution onto the electrode surface. The synthesized AgBiS$_2$ exhibited a three-dimensional flower-like structure, which was assembled from nanosheets. This distinct structure provides a favorable surface topography for anchoring other photoactive nanomaterials. The photosensitizer demonstrated an enhanced PEC response due to the synergistic effect of the matching band gap and electron transport. The PEC sensor was effectively used for the detection of BPA in real water samples, achieving an LOD of 0.18 nmol L^{-1}. Their study provides a new strategy for sensing technology and opens up more possibilities for improving the detection sensitivity of electrochemical sensing analysis.

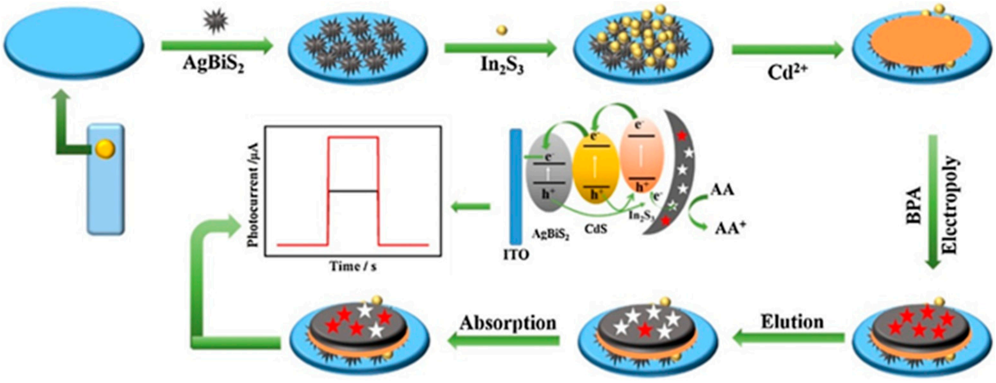

Figure 15. The fabrication process of the MIP-based PEC sensor for BPA detection. Reproduced with permission from [91].

Perfluoroalkyl and polyfluoroalkyl substances (PFASs) have been used in firefighting foams, non-stick cookware, and waterproof and stain-resistant clothing [92,93]. PFAS pose a risk to human health, particularly because of their ability to act as endocrine disruptors and their environmental safety as persistent and biocumulative. They are currently classified as persistent organic pollutants by the Stockholm Convention and the United States Environmental Protection Agency [94]. In PFAS sensors based on PEC, AgI nanoparticles and BiOI nanosheet arrays (AGI-BioINFS) fixed on fluorine-doped tin oxide (FTO) are used as photosensitive electrodes, and the array is subsequently grafted with PFOA MIP. The LOD for PFOA is estimated to be 0.01 μg L^{-1}, which is comparable to what can be achieved with LC-MS/MS. Ultrathin graphite carbon nitride nanosheets (utg-C_3N_4) modified with MIP were used to detect PFOA [67,95], in which 5 mM pyrrole and 1 mM PFOA were electropolymerized by cyclic voltammetry at pH 6. The results showed that the ECL signal intensity decreased linearly with the increase in PFOA concentration. The LOD was estimated at 0.01 ng mL^{-1}, significantly lower than the 25 ng L^{-1} reported using chromatographic methods [96].

4. Summary and Outlook

The development of molecularly imprinted polymers (MIPs) has made significant progress in both binding properties and selectivity, and they can be used as solid-phase extraction materials for the separation and enrichment of chemical pollutants during sample pretreatment. In addition, MIPs have excellent high-temperature resistance, acid and alkali resistance, and easy storage characteristics, so they are suitable as sensor-sensitive materials for the analysis and detection of actual samples. So far, MIPs have been developed from a single template to a composite template, and the preparation process is constantly optimized to improve its application in various fields such as environmental pollutant analysis, food quality and safety, and biological sample separation and enrichment. However, there are still some problems that need to be explored and solved as follows:

(1) Real samples often contain complex media, including solid particles and high ion concentrations, which can potentially interfere with the pollutant screening and identification process facilitated by MIPs. The presence of both hydrophobic and hydrophilic structures in MIPs allows for a variety of adsorption mechanisms, with the hydrophilicity of the particle core binding site enhancing its selective interaction with the target molecules. However, surface contamination from outer layer adsorption can compromise the selectivity of MIPs, necessitating future research to enhance the applicability and anti-interference capabilities of MIPs in practical samples.

(2) Strategies to improve MIP performance include enhancing binding ability, increasing hydrophilicity, and minimizing template leakage. The preparation of high-performance MIPs with controllable structures can mitigate the matrix effect and offer an innovative approach for detecting low-content pollutants in complex matrix samples.

(3) Finally, the use of non-degradable micro- and nanomaterials in MIPs preparation should be minimized. In the context of pollutant screening and identification, the synthesis of environmentally friendly micro–nano MIP materials should be prioritized, and comprehensive research on green imprinting techniques should be undertaken.

Efforts to solve these problems have never stopped. In the future, the combination of MIPs and different analytical instruments to realize the artificial detection system is also an ideal goal that people continue to pursue.

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