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Review

Combining Fluorescent Quantum Dots with Molecularly Imprinted Polymers for the Screening of both Emerging and Classical Environmental Pollutants: A Review

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Emerging and classical pollutants, such as antibiotics, pharmaceuticals, pesticides, dyes and heavy metals derived from human activity, currently pose serious threats to the environment and human health. Despite the grave danger posed by these pollutants, there is still no adequate monitoring of their presence in the environment. The regular determination of these contaminants in the environment can play a crucial role in the protection of human health and the preservation of ecosystems. New analytical techniques allow the reproducible quantification of analytes at very low concentration levels. Molecularly imprinted polymers (MIPs), with selective recognition, have also been combined with quantum dots (QDs) and suggested as valuable materials in the construction of optical sensors. Several strategies have been proposed for the selective detection of these pollutants in recent years. Rather than employing expensive, time-consuming standard analytical methods, fluorescent quantum dots coupled with molecularly imprinted polymers can be used for developing simple, rapid and highly selective analytical methods for the detection of these pollutants. This review presents a brief discussion on the application of tailor-made polymeric materials in tandem with quantum dots for the rational construction of efficient sensors capable of determining the presence of these pollutants in aquatic environments.

Keywords: quantum dots, molecular imprinting, fluorescence spectroscopy, core-shell nanomaterials

1. Introduction

External pollutants are substances that negatively affect the quality of any freshwater, giving rise to contaminated wastewater. The primary sources of wastewater are household wastes, industrial wastes, and wastes derived from agricultural activities including crop production and livestock farming. The industrial sector, in particular, consumes a large amount of water and produces a substantial quantity of wastewater which is composed of both organic and inorganic effluents. With the ongoing widespread health problems caused by the rampant disposal of wastewater into the environment (particularly in water bodies) and the deteriorating quality of freshwater, researchers have been compelled to develop inexpensive and environment-friendly methods for monitoring emerging pollutants in wastewater. As wastewater contains complex matrices, it requires the use of highly efficient, selective, and sensitive techniques for the identification and quantification of

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hazardous target compounds present therein. Bearing that in mind, a wide range of powerful analytical techniques are capable of effectively determining compounds of interest in wastewater; among these analytical techniques include high-performance liquid chromatography in tandem with mass spectrometry (HPLC-MS) and gas chromatography coupled with mass spectrometry (GC-MS). Although these techniques are efficient when applied toward the determination of environmental pollutants in complex matrices such as wastewater, they exhibit some non-negligible limitations, which include the following: (*i*) the techniques involve relatively high set-up and operational costs; (*ii*) one requires highly trained analysts to carry out the detection techniques; (*iii*) the techniques involve high reagent consumption, and (*iv*) the techniques are often time-consuming.[1] The combined application of an ultrasensitive technique (fluorescence) and materials with high selectivity (quantum dots in tandem with molecularly imprinted polymers) can help overcome these constraints.

Being small in range, quantum dots (QDs) exhibit a number of useful properties and are widely employed for the development of sensing devices owing to their particularly suitable optical properties.[2,3] Fluorescence-based quantum dots (QDs) sensors are characterized by high quantum yield, broad absorption spectra, narrow fluorescence emission, and high photostability.[4,5] In addition, QDs display a narrow emission band and high stability compared to organic fluorescent dyes, and the signal of QDs-based devices is less prone to interferences.[5] As the surface of QDs allows simple and efficient chemical modifications, it is readily feasible to employ a fluorescent QDs sensing system for the detection of a wide variety of analytes.[6] Due to their outstanding properties, fluorescence-based QDs sensors have been successfully employed for the recognition of analytes in aqueous solutions with a low limit of detection, high stability, and operational simplicity. The main limitation of fluorescence-based QDs sensors lies in their poor selectivity because of the quenching phenomena.[2] To overcome this constraint, the surface of the QDs-based device can be modified with capturing materials, for example, molecularly imprinted polymers (MIPs) can be used to enhance the detection process by selectively capturing the analyte of interest. In this sense, the use of QDs in the development of sensing devices is an efficient way to produce highly selective and sensitive fluorescent sensors.

The use of synthetic receptors, such as MIPs, in environmental applications has attracted considerable attention among researchers due to the selectivity of these materials and their high affinity for emerging pollutants. The imprinting process yields MIPs with interesting properties, including large surface area and porosity, as well as excellent mechanical and chemical stability; apart from that, these MIPs have a significant number of binding cavities on their surface, which allow them to effectively capture the target molecule.[7,8] MIPs are commonly synthesized in the presence of an appropriate solvent (porogenic solvent), functional monomer, template (target molecule), structural monomer (cross-linker), and a radical initiator. After the polymerization process, the template is removed, thus creating selective cavities in the polymeric matrix.

This review focuses on current breakthroughs in the manufacturing and implementation of MIP-QDs sensor for detecting both emerging and classical environmental contaminants in aquatic environments. This study's ensuing parts provide a description of the various methods used to prepare MIP-QDs sensor, with a focus on the modifications that contribute most significantly to enhanced sensor performance. In addition, strive to give the reader up to date on the information concerning QDs.

2. Surface Chemistry and Properties of QDs

The study of the chemical composition of QD surfaces is a relatively new area in semiconductor materials. In addition to the manipulation of particle size, adjustment of the optical characteristics of a QDs suspension is also possible through careful control of the chemistry of the QDs surface. Even if only one molecule is attached to the surface, this can result in the introduction of novel functionalities, the properties which are given in Table 1.[9,10] The quantum confinement effect is a characteristic of semiconductors that become apparent as the diameter of a nanoparticle approaches that of the Bohr-exciton radius, and is responsible for the size dependence of the optical and electrical properties of semiconductor nanocrystals or quantum dots (QDs).[11] The semiconductors CdS, CdSe, and CdTe are examples of materials that exhibit quantum confinement effects at small particle sizes (5-6 nm),[12] (11 nm),[13] and (7.3 nm).[14] Due to their diminutive size, QDs contain far more atoms on their surface than in their interior.[15] Particle characteristics become increasingly governed by surface atoms as this ratio of surface to core atoms rises. Since the semiconductor lattice terminates at the QD surface, the atoms there are distinct from those in the core.

Recent developments in QDs surface chemistry to boost their fluorescence signal for detection of potentially risky desired molecules are summarized below.

2.1. Signal response of QDs materials

The use of QD nanomaterials for the detection of analytes of interest in an aqueous solution largely depends

Table 1. Properties of quantum dots (QDs) nanoscale material

No.	Name of properties	Description of properties
1	dimension	material with no dimensions
2	density	they are denser and sharper
3	particles size	2-10 nm particles size
4	band gap energy/size of QDs	quantum dot size and band gap are inversely proportional
5	radiation of QDs	size and band gap determine color irradiation/increase in size the emission color shifts to the red
6	luminescence properties	QDs have luminescent characteristics
7	emission of wavelength	QDs emits wavelengths between those of the ultraviolet and infrared spectra

Obtained from articles of Wang *et al.*[9] and Zhao *et al.*[10]

on the sensitivity of their fluorescence to the surface of the nanoparticles. Thus, sensing can occur as a result of chemical or physical interaction with the target analyte, resulting in either the enhancement of photoluminescence or quenching.[16] When energy flows between QDs and the analytes, this energy is absorbed by the donor (QDs) and transferred to the nearest acceptor through dipole-dipole interactions-this is referred to as the Forster resonance energy transfer (FRET) phenomenon. The sensing mechanism involving the use of QDs nanoscale material primarily depends on the energy flow between the QDs (donor) excited fluorophore and the analyte molecules (acceptor). The energy exchange occurs as a result of resonance interactions, in which an excited electron in a molecule provokes oscillatory waves, stimulating the electron transfer from the acceptor to an excited state. In the case of a fluorescent-based acceptor, the de-excitation of the acceptor will arise specifically via photon emission; however, it should be noted that if the acceptor fails to undergo fluorescence, it will return to its lowest energy state and undergo interaction with the solvent. The efficiency of the energy transfer between the donor and acceptor primarily depends on the following three factors: (*i*) distance (R) between the FRET pair, i.e., donor and acceptor; (*ii*) spectral overlap between the donor (fluorescence spectrum) and acceptor (absorption spectrum); and (*iii*) the fluorescence lifetime of the donor ought to be adequate for FRET.[17,18] The FRET phenomenon is considered to be in operation when there is a decline in the donor fluorescence and the excited-state lifetime and a rise in the acceptor fluorescence and lifetime (Figure 1). Thus, the use of QDs for the development of sensing devices is found to be highly promising due to the broad absorption spectra of QDs, in addition to their long fluorescence lifetime, and high quantum yields; all these factors are observed in FRET.[19,20] Nanotechnology has provided a conducive platform for the fabrication of fluorescence nanomaterials which are highly efficient for sensing applications.

2.2. QDs types and composition

The concept involving the development of QDs material is primarily derived from the desire to reduce the size or dimension of semiconductors.[21,22] Owing to the quantum effect, the optical and electronic properties of semiconductor materials at the nanoscale are completely

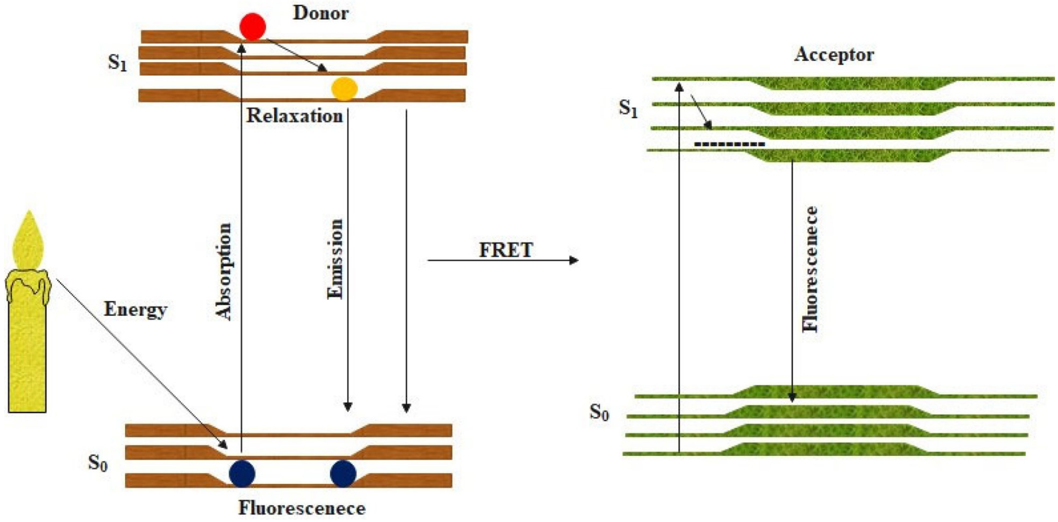


Figure 1. Simple Jablonski diagram illustrating one of the numerous transitions that can occur when a molecule has been photo-chemically energized.

different from those of their bulk form.[22] QDs have a wide range of properties, which include the following: narrow emission, tunable wavelength, excellent photoluminescence (PL), spectrum purity, and photochemical stability; due to these properties, QDs are considered a good potential substitute for traditional organic dyes.[23] Due to these excellent properties, QDs materials are found to be suitable for use in the detection of effluents present in environmental matrices. Although further improvements are needed in QDs technology, it is still considered an essentially important mechanism for the development of highly efficient sensors. There are different types of QDs and their classification is based on the elements in each group; the different types of QDs reported in the literature include the following: QD-II-VI group,[24,25] QD-III-V group,[26,27] QD-I-III-VI group,[28] and perovskites.[29,30] When it comes to semiconductor QDs, graphene QDs (GQDs) and carbon QDs (CQDs) have a leading edge over other semiconductor QDs due to their good photostability, effective photobleaching, low toxicity, and excellent biocompatibility.[31,32] In a previous study reported in the literature, Xu *et al.*[33] produced a mixture of fluorescent nanoparticles using single-wall carbon nanotubes. Islam *et al.*[34] also developed graphene quantum dots (GQDs) where the QDs nonparticles exhibited the following properties: ease of use, high yield, low cost, less damaging properties, great fluorescence quality, and particle size uniformity. A number of studies published in the literature[35,36] have also reported the use of a wide range of techniques for the synthesis of GQDs and CQDs. It should be noted that the size of the QDs material plays an important role in shaping its properties. Furthermore, some desirable features like large surface area, good selectivity, and high efficiency of the sensing device toward the target molecule (analyte) detection can be obtained by doping QDs with other materials, this makes QDs highly suitable for application in the construction of sensing devices.[37] Several studies have demonstrated the use of GQDs and CQDs in different areas, including biomedicine,[36] catalytic processes,[38] energy,[39,40] and photo-electronic processes.[41] However, more work needs to be done with a view to improving GQDs and CQDs technology and their application for the recognition/determination of effluents in environmental matrices.

Several nanoscale carbon-based fluorescence materials have been considered as suitable candidates for application in the detection of analytes via the fluorescence technique. A number of studies published in the literature[42,43] have provided the use of carbon-based fluorescence materials, such as graphene quantum dots (GQDs) and carbon quantum dots (CQDs), for the detection of analytes in aqueous solutions. These carbon-based QDs materials are regarded as an emergent alternative to other semiconductor-based (heavy metals) fluorescence materials because of their outstanding fluorescence properties, low toxicity, and excellent biocompatibility.[44] In their attempt to improve the detection mechanism under the fluorimetric approach, Li *et al.*[45] used GQDs together with the acetylcholinesterase (AChE) enzyme for the successful detection of paraoxon. More recently, Zhang *et al.*[46] successfully employed carbon quantum dots for the detection of folic acid. Figure 2 presents an illustrative mechanism involving the detection of folic acid using carbon quantum dots. Hou *et al.*[47] modified the surface of carbon dot nanomaterial with L-tyrosine methyl ester (Tyr-CDs) via hydrothermal reaction; the authors employed elemental composition analysis, including Fourier-transform infrared (FTIR) and X-ray photoelectron spectroscopy (XPS), to confirm the presence of L-tyrosine methyl ester on the carbon dots surface, and to investigate the surface state, respectively. The sensor developed in that study exhibited good sensitivity toward methyl parathion detection with a limit of detection of 4.8×10^{-11} M.

The center of QDs is referred to as the nucleus; this region is responsible for the narrow band gap energy (minimum energy required to excite electrons from lower energy level to high energy level) observed in the nanocrystal material.[48] Due to the effects of fraction atoms in the surface region, it is impossible to share chemical bonds, and this leads to instability in the fluorescence of the QDs nucleus. To circumvent this instability, one needs to incorporate a different semiconductor material with a relatively larger band gap energy (e.g., ZnS) around the center (nucleus) of the QDs to interact with the atoms in this area. This process is referred to as passivation, and it helps to improve the stability of the nanoparticles, leading to the production of a new structure called QDs core-shell.[49] Moreover, the shell structure reduces the aggregation that occurs in nanomaterials while improving the resistance to oxidation, photodegradation, pH, and other chemicals in the surrounding medium.[49] A wide range of functional agents have been successfully employed in the search for QDs with good biocompatibility for the desired application.

2.3. Functionalization using different functional groups

QDs are normally produced from organometallic precursors or heavy metals, and organic ligand layers of trioctylphosphine (TOP) or trioctylphosphine oxide (TOPO) are used to protect the particles from aggregation. Several ligands with attractive functional groups are used to

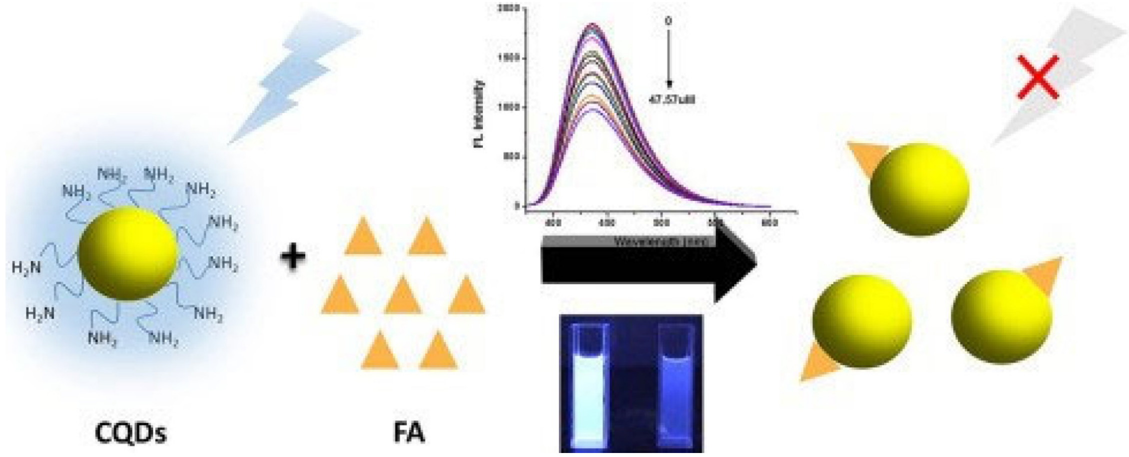


Figure 2. Graphically representation of the fluorescence sensor: interaction between carbon quantum dots (CQDs) and folic acid (FA) analyte (reproduced from reference 46 with copyright permission 2020 from Elsevier).

improve the surface of the QDs. For instance, Walia *et al.*[50] enhanced the recognition sites of the QDs nanomaterials for the detection of dicofol using glutathione; based on the technique proposed in their study, the chloride group present in the dicofol structure interacts with the $-NH_2$ and $-COOH$ groups of the glutathione ligand, and this leads to the improvement of the fluorescence efficiency of the QDs material. The use of this kind of ligands for surface modification may also cause interferences and decrease the capability of the sensing device.[51,52] This can be expressed in terms of selectivity which is derived from the reactivity of $-NH_2$ and $-COOH$ groups, low quantum yields,[53] and toxicity of unshielded cadmium core. Despite the aforementioned drawbacks, this kind of ligands (glutathione, among others) has been found to exhibit good compatibility. Furthermore, we will focus our discussion on surface modification of QDs using a wide range of materials, including enzymes, antibodies, nucleic acids, supramolecular polymers, and MIPs.[54,55]

2.4. QDs modified by macrocyclic compounds

Macrocyclic arenes have drawn considerable interest in the field of supramolecular chemistry.[56] Ogoshi *et al.*[57] initially reported the macrocyclic host molecule including pillar[*n*]arenes; it should be noted that calixarenes, cyclodextrins, and calixarenes may also belong to this class of macrocyclic host molecules.[58] The precise pillar geometry of pillar[*n*]arenes makes them more suitable for application toward the modification of QDs material and macrocyclic compound-based QDs.[59] The macrocyclic molecules that have suitable cavities can also be used along with QDs for the effective recognition of target molecules in wastewater. For instance, the hydrophobic cavities present in calix[*n*]arene supramolecule are potential receptors for the target compound,[19] the selectivity toward the model compound (target molecule) can be improved by tuning the material to obtain the appropriate size of calix[*n*]arene molecules. This kind of molecule is then used to functionalize the surface of QD nanomaterials with a view to improving the selectivity of the material. The host-guest strategy which involves the use of calix[4]arene has been described elsewhere,[60] under this technique, the core of silica embedded with QD material is coated with calix[4]arene to enhance the sensing performance. Similarly, Qu *et al.*[61] used *p*-sulfonatocalix[4]arene supramolecule coupled with CdTe QD nanomaterial to improve the affinity of a fluorescent sensor toward the target molecule. Based on the findings of the study, the sensor signal increased linearly in the concentration of up to 10^{-4} M (fenamithion) and 10^{-3} M (acetamiprid) under optimized conditions. The results obtained from this study also showed that the sensor proposed by the authors exhibited excellent selectivity toward fenamithion and acetamiprid, with limits of detection of 1.2×10^{-8} M and 3.4×10^{-8} M, respectively.[61] Although this procedure seems promising, it should be noted that the binding affinity of the sensor can be influenced by a range of variables; these include the binding mechanism, pH value, stereo-electronic effects, and allosteric impact.[62] Thus, a sensor primarily constructed using ZnS:Mn QDs chemically modified with acetamiprid aptamer will encounter extreme obstacles in terms of repeatability and this will certainly undermine its application.

2.5. Silica nanoparticles-modified QDs

The incorporation of silica in QD sensors contributes toward enhancing the limit of detection and selectivity of the sensing devices. For purposes of illustration, Li *et al.*[63] used silica-coated CdTe-QDs to monitor the pesticide glyphosate. The signal of the fluorescence sensor was linearly enhanced from 1.0 to 25.0 nmol L^{-1} and the limit of detection was 0.0725 nmol L^{-1}; in addition, the fluorescence sensor displayed excellent selectivity and was successfully applied for the detection of glyphosate. The technique adopted by Li *et al.*[64] was also replicated for the development of $CdSe@SiO_2$ materials with enhanced selectivity by incorporating tailor-made MIPs into the sensing platform.

The $CdSe@SiO_2$-MIP was effectively applied for the analysis of pyrethroids in the concentration range of 0.1-1000 µM where a limit of detection of 3.6 µg L^{-1} was obtained. In another study reported in the literature, Li *et al.*[60] used the sol-gel technique to produce tailor-made receptor on the surface of silica nanoparticles (calix[4]arene-SiO_2) where the selectivity of the material was further enhanced by the application of a QD material (CdTe). The technique employed resulted in a rise in the fluorescent sensing intensity where a limit of detection of 0.08 µM was obtained with the concentration of the target molecule methomyl increasing from 0.1 to 50 µM. A previous study reported in the literature[65] employed the ratiometric fluorescent technique to monitor the target analyte where changes in the ratio of photoluminescence (PL) intensities were recorded at two wavelengths. Wang *et al.*[66] synthesized ratiometric fluorescent sensor using silica doped QDs and gold nanoparticles (AuNPs) and applied the device for the determination of pesticides. Based on their findings, under optimized conditions, the fluorescent sensor exhibited excellent performance in the concentration range of 0.04-400 ng mL^{-1}, with a limit of detection of 0.018 ng mL^{-1}. The synthesized sensor displayed excellent affinity toward the target molecule, and the recovery percentages obtained from the application of the sensor in agriculture and environmental samples were found to be in line with the expected values. This study developed the synthesized similar fluorescent sensors for environmental applications in which the QDs silica nanoparticles ($CdTe@SiO_2$) were developed using the reverse micro-emulsion approach. The coating of silica shell by the micro-emulsion technique has become widely popular recently because of its simplicity, excellent particle size control, and good size distribution compared to the adapted Stöber approach.[66,67] Several studies reported in the literature[68] have shed light on the preparation techniques involving the modification of QDs with Si nanoparticles; the incorporation of these nanoparticles has been found to enhance the chemical stability (reduced leaching) of the material in addition to decreasing the toxicity potential of the heavy metals used in the synthesis of the QDs material. Silica nanoparticles are an interesting material known to possess good properties; this material is suitable for application in fluorescence detection because of its chemically inert and optically transparent nature.[69]

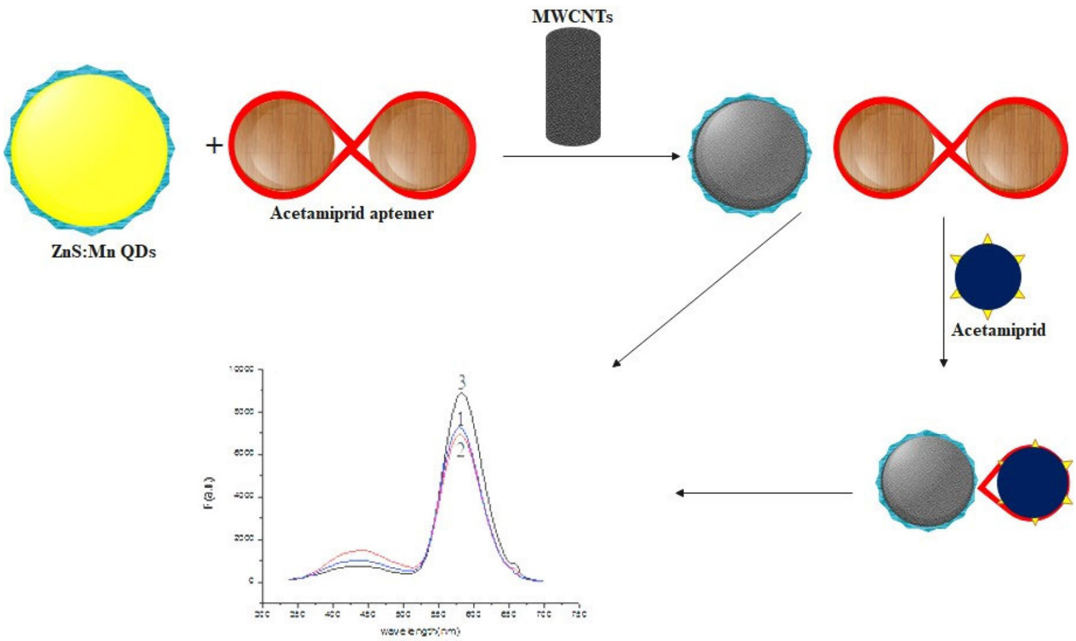
2.6. Enzyme-modified QDs

Enzymes, and antibodies can also be coupled to QDs and applied as recognizing elements in sensing applications.[70,71] For instance, a fluorescence sensor (ZnS-Mn/QDs) was successfully coated with DNA aptamers and used for the selective binding of acetamiprid.[72] The fluorescence probe (ZnS-Mn/QDs) is shown in Scheme 1.[72]

The limit of detection obtained for the ZnS-Mn/QDs fluorescence sensor applied for acetamiprid detection was 0.7 nM. Based on this same strategy, Guo *et al.*[73] modified the surface of QDs material with gold nanoparticles where the material was coupled with DNA aptamers and applied for the detection of insecticides. The choice of an appropriate aptamer for the detection of a target molecule in the fluid phase is clearly a challenging task. Enzyme-modified QD biosensors have been widely employed for pesticide detection, especially for the detection of organophosphorus pesticides. Enzymes are found to be particularly alluring as recognizing components in QD biosensors because of their specificity toward target pesticides. The ability of the enzyme to recognize pesticide molecules plays a crucial role in the efficiency of QD-based sensors when it comes to the detection of these target analytes. The selectivity of enzyme-based sensors can be improved via a combined application of other supporting materials such as graphene,[74] and multiwall carbon nanotubes.[75] Despite their excellent sensitivity and selectivity, these biosensors present some limitations in terms of stability and large-scale production. The stability of the sensors is severely influenced by temperature and pH conditions. In addition, the generation, separation, and refinement of these sensing devices can be time-consuming, and expensive. The reuse of enzyme-modified materials may reduce the sensitivity and stability of the sensor when applied toward the detection of pollutant compounds in environmental samples. To help overcome these constraints, researchers have introduced a new type of artificial receptors, which are referred to as molecularly imprinted polymer. These tailor-made materials have the potential to recognize the target compounds because of the specific cavity on their surface.

2.7. Synthesis of molecular imprinted polymers

One of the key factors that contribute toward the effective synthesis of MIP is the choice of an appropriate



Scheme 1. Representation of ZnS/Mn-QDs fluorescence sensor modified with acetamiprid aptamer for the recognition of insecticide (adapted from reference 72).

functional monomer which can interact with the template molecule to form a complex in the pre-polymerization process. The pore size and binding ability of the MIP are found to be considerably dependent on the mole fraction of the functional monomer in the pre-polymerization process.[76] A wide range of functional monomers have been employed for the synthesis of selective MIPs; these monomers can be classified based on their designing strategies, including covalent, non-covalent, and semi-covalent interactions.[77,78] Some functional monomers are developed based on covalent and non-covalent approaches. Some common functional monomers, cross-linkers, and initiators are presented in Figure 3.[79,80]

Some of the target compounds used as analytes/templates include pesticides, endocrine disruptors, drugs, steroids, hormones, and carbohydrates. The synthesis of biological molecularly imprinted polymers, such as proteins or viruses, is still a key challenge in electrochemical analysis because of the cavities that are poorly formed in cross-linked polymers.[81,82] Templates and solvents play a key role in the synthesis of MIPs because of their specific functionality in terms of the recognition of analytes and interaction with appropriate monomers,[83] polar solvents influence template interaction.[84] The noble nature of the template is an important property when it comes to the development of molecularly imprinted polymers. The noble nature of the template ensures that it will not affect the interaction of template with functional monomers or other components, resulting in a stable polymer structure. Selectivity and affinity of MIP are influenced by the intermolecular interactions between molecular templates and functional monomers.[85] It is possible to use computer simulation to examine these intermolecular interactions prior to polymerization. As *in silico*-based technology has advanced, it has become much simpler to choose MIP components like templates, functional monomers, and appropriate porogens.[86] It can also be used to ascertain the nature of the intermolecular interactions present in the precursor to polymerization.[87] Zeta potential, for instance, has been shown to be one of the most important variables affecting the surface reaction and behavior of polymer nanoparticles in sensor.[88] Because of their nanoscale size and high surface area to volume ratio, nanoparticles have a variety of intriguing features that make them useful materials for various applications, including sensors. In this way, the surface chemistry is crucial in deciding and regulating the necessary. For instance, Chao *et al.*[89] demonstrated the synthesis of molecularly imprinted polymer-wrapped with wrinkled silica-quantum dot hybrid particles for fluorescent determination of tetra bromo bisphenol A. Zeta potential pointed to variations in surface charge during the fabrication processes. After being modified with APTES (3-aminopropyltriethoxysilane), the wrinkled silica nanoparticles (WSN) values were changed from positive to negative when combined with negatively charged QDs. The wrapped molecular imprinted polymers with wrinkly silica-quantum dot hybrid particles showed positive charges, indicating the presence of amine groups from the impinging shells. The same zeta potential analysis has

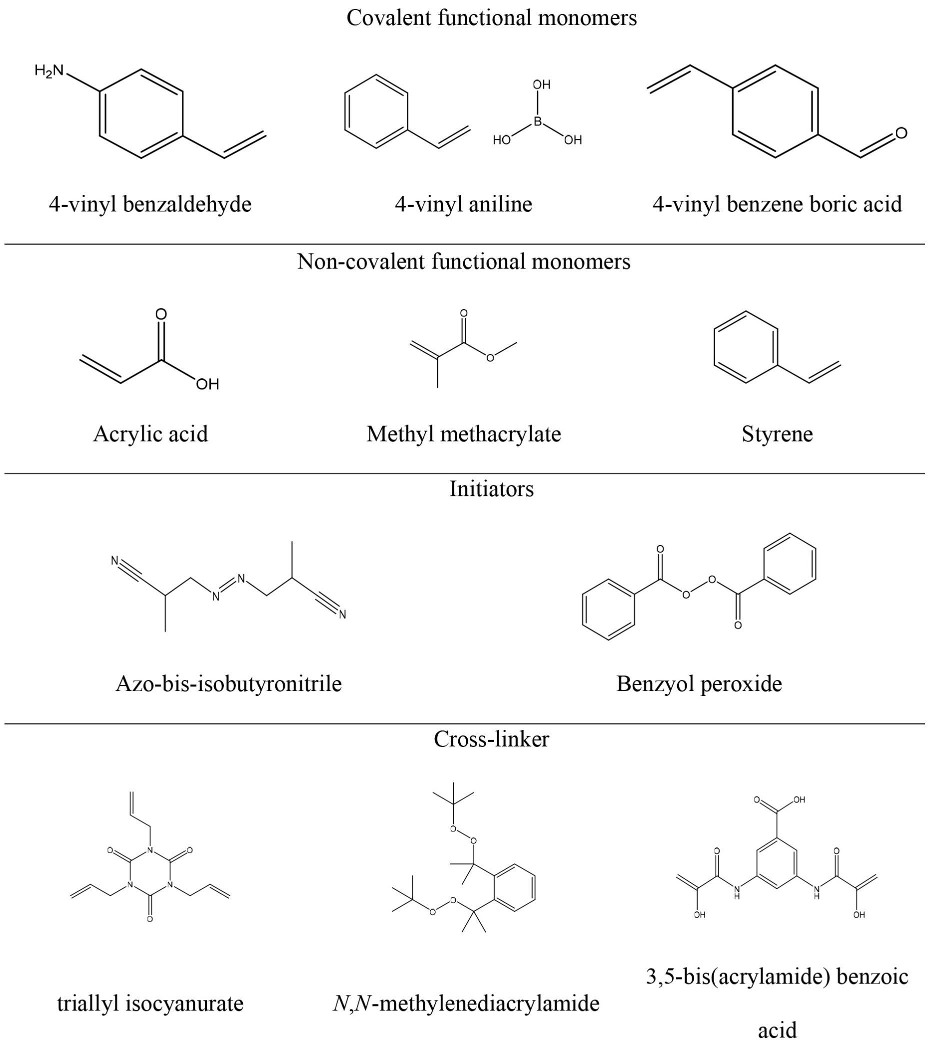


Figure 3. A wide range of functional monomers, radical initiators and cross-linkers.

been extended to determine tannic acid in food samples using a molecularly imprinted polymer on amine-modified carbon quantum dots.[90] The zeta potential of amine-modified carbon quantum dots (a-CQDs) was determined to be 1.5 ± 0.5. The presence of carboxyl functional groups on the surface of a-CQDs is shown by the negative charge. The presence of amine groups on the surface, however, is responsible for the low negative charge.[91] Similarly, another study found that MIP could be used to couple the core support material, such as QDs, and that the carboxyl group on the surface of CdTe QDs could improve their performance.[92] As a means of stabilization, 2-aminoethyl methacrylate hydrochloride (AMA) was incorporated. As revealed by the significant increase in zeta potential from -30.1 to -19.3 mV, the negatively charged CdTe QDs readily interact with the positively charged AMA to form the desired structure via electrostatic attraction. This is clearly conceivable if the template molecule does not have any functional group which interferes with the radical polymerization. Functional groups, such as amino acids, hydroxyl, ester, amide, and carboxyl groups, tend to hinder the performance and selectivity of molecularly imprinted polymer. In fact, the template in the hybrid MIP is close to the surface, and this makes it easy to be removed from the MIP. [93,94]

Cross-linking agents are used to bind the functional monomer around the template molecule.[95] The cross-linker agent is used as a shield to protect the structure of the polymer from demolition after washing the template molecule.[96] The use of a considerably high amount of cross-linker leads to a decrease in the number of cavities formed in the polymer matrix, while the use of a markedly low amount of cross-linker leads to insecurity in the mechanical strength of the material.[97] The existence of a limited number of suitable cross-linkers clearly derails advancement in MIP innovation.[80-98]

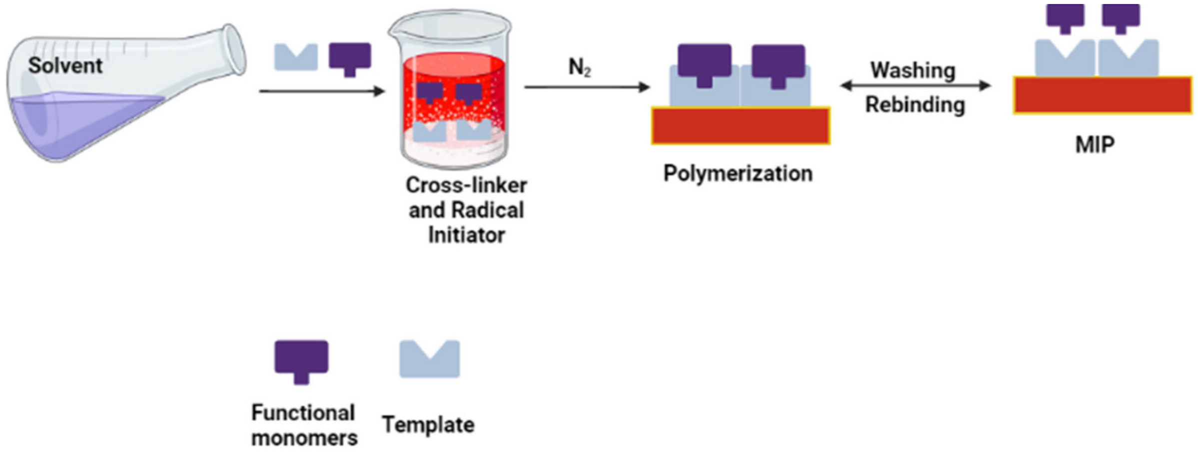


Figure 4. Process flow diagram for developing molecularly imprinted polymers.

2.8. Methods to synthesize MIP@QDs

MIPs can be generated in general by a variety of polymerization techniques,[99] including bulk imprinting (3D) and surface imprinting (2D). When using surface imprinting technology, templates are placed on the material's outermost layer, whereas when using bulk imprinting, the template is incorporated into the material's cross-linked structure to create a single solid piece. The schematic for MIP synthesis is shown in Figure 4.

Surface imprinting is still preferable for the manufacturing of MIPs for (bio)macromolecules because the diffusion to and binding of the template/analyte at the polymer surface is more favorable and faster than inside its porous structure. Soft lithography,[100,101] self-assembled monolayers,[102] core-shell particle preparation,[103,104] and miniemulsion polymerization[105,106] are some of the most widely used surface imprinting methods. For the fabrication of molecular imprinted polymers, see Figure 5, which depicts surface imprinting techniques.

Fabrication methods of QDs have been reported in the literature such as top-down and bottom-up.[107] Top-down methods, such as laser ablation, electrochemical etching, liquid-phase exfoliation, electron beam lithography, etc., entail the direct cleaving of bulk materials into nanoscale QDs. Bottom-up methods, such as hydrothermal/solvothermal, microwave irradiation, template, pyrolysis, and wet-chemical reactions, involve the conversion of appropriate molecular precursors into QDs under controlled conditions. Top-down and bottom-up approaches to GQD synthesis are shown in Figure 6. The relative benefits and drawbacks of the various approaches are outlined in Table 2.[108]

Nonetheless, in this section, we will discuss about the documented literature on synthesis approach of molecular imprinted polymers coated quantum dots.

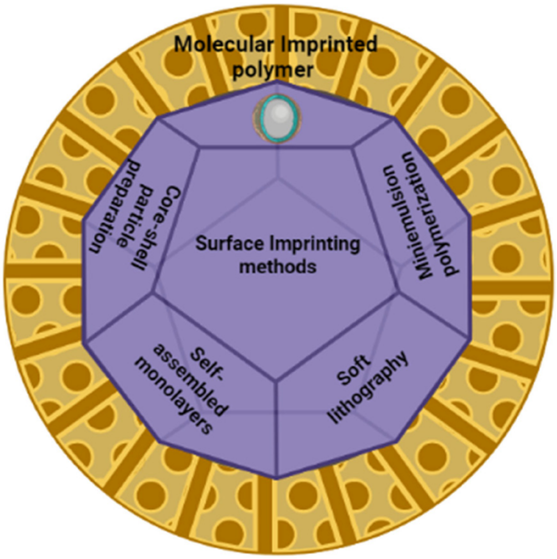


Figure 5. Surface imprinting techniques for the synthesis of molecular imprinted polymers.

One-pot synthesis is a technique used in materials chemistry to increase the throughput of a chemical reaction by performing multiple reactions on the same reactant in a single reactor. Researchers appreciate the simplified procedure because it eliminates the need for time-consuming steps like separating and purifying the chemical compounds in the intermediate stages.[110] So, the advantages of one-pot synthesis include reduced expenses, enhanced reaction efficiency, and increased product purity. One-pot synthesis is illustrated by the creation of MIP-based QDs.[92] In the first step, the author followed a previously reported method,[111] to synthesize green-emitting TGA (thioglycollic acid) modified CdTe QDs in an aqueous phase. To summarize, a mixture of calculated amount of $NaBH_4$ (sodium borohydride) and tellurium powder were added to 1.5 mL of absolute ethanol and 0.5 mL of ultrapure water, and the mixture was then heated to 40 °C for 4 h. After

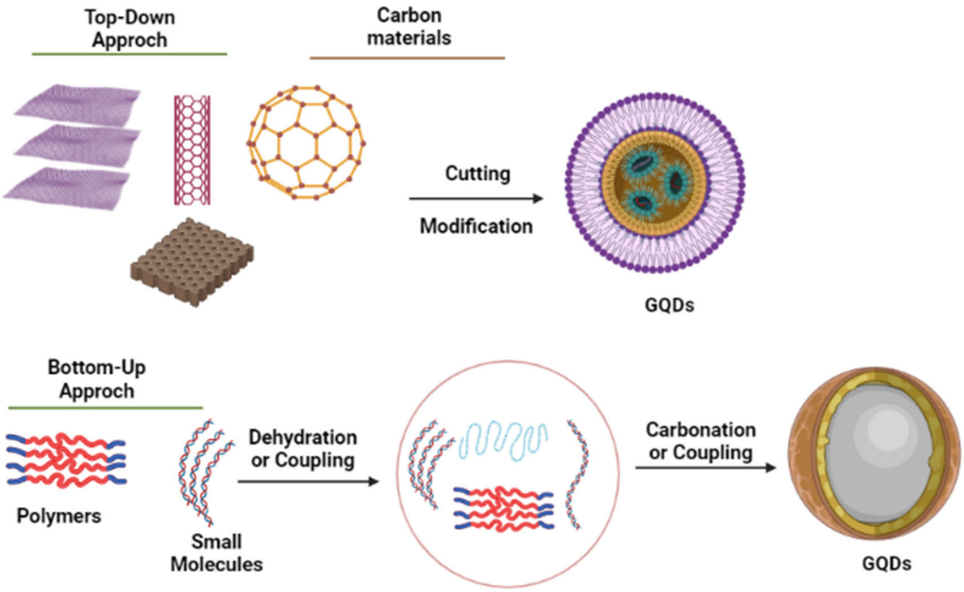


Figure 6. Fluorescent GQDs were synthesized using two primary methods: the "top-down" splitting technique, whereby various carbon sources are used, and the "bottom-up" method, wherein small molecules or polymers are used as building blocks (adapted from reference 109).

Table 2. Comparison synthesis methods of QDs

Synthesis method	Advantages	Disadvantages
Carbonization	it is easy and green method	polydisperse GQDs are challenging to manage in size and structure
Controllable synthesis	as-prepared GQDs have exact carbon atom count, homogeneous size and shape	multi-step chemical processes are involved; lengthy and low-yielding process
Hydrothermal/ solvothermal	simple and fast technique	before reactions occur, carbon must be oxidized
Electrochemical oxidation	high stability and consistent size distribution	low yield prevents mass fabrication
Microwave-assisted/ ultrasonic-assisted	it reduces reaction time and boosts productivity	industrial production of microwave/ultrasonic reactors is limited and expensive
Cleavage oxidative	simple and effective, it is the most viable method for mass production	strong oxidizers may produce fire or explosion, and postprocessing is difficult

Obtained from publication of Chen *et al.*[108]

dissolving 92.4 mg of Cd $(NO_3)_2.4H_2O$ (cadmium nitrate) and 6.3 µL of TGA in 75 mL of ultrapure water, the pH was adjusted to 9 with 1.0 M NaOH (sodium hydroxide). For at least 30 min, oxygen was removed from the solution by purging the mixture with nitrogen. Under constant stirring, 1 mL of a freshly made NaHTe aqueous solution was added to the mixture. TGA-modified CdTe QDs with a green emission were obtained after the material was subjected to 2 h of boiling and refluxing. The QDs@MIP were then synthesized using a simple free radical polymerization process. A pre-polymerization solvent was prepared by adding the appropriate amounts of a template, functional monomer, QDs nano-materials, cross-linker, and radical initiator. After 30 min in the presence of nitrogen, 4 mg of potassium persulfate was added, and the reaction was allowed to proceed at 40 °C while being stirred in the dark for an additional 12 h. In the end, the products were centrifuged and washed with ethanol/acetonitrile. Synthesis of MIP@QDs nanomaterial is shown in Figure 7.

The sol-gel method is a technique for the manufacture of materials that involves first solidifying a compound that contains a highly chemically active component by means of a solution, gel, or sol, and then treating an oxide or another compound with heat.[112] This technique is used for material preparation in relatively moderate conditions. In comparison to other synthesis methods, this one allows for greater modification of material texture, size, and surface qualities; it is also easy to apply; it is inexpensive; it yields high-quality; and it can produce materials with huge surface areas.[113] Preparation of molecular imprinted polymers on the surface of silica-coated graphene quantum dots by sol-gel polymerization was reported by Zhou *et al.*[114] Beaker having 20 mg of 4-nitrophenol (as a template), 20 mL of ethanol, and 50 µL of 3-aminopropyltriethoxysilane (APTS) was agitated for 30 min. After injecting 1.6 mL of acetic acid (1 M), 200 mL of TEOS (tetraethoxy-silane), and 4 mL of silica-coated GQDs colloidal solution, the mixture was stirred for 15 h. The resulting GQDs were coated with MIP,

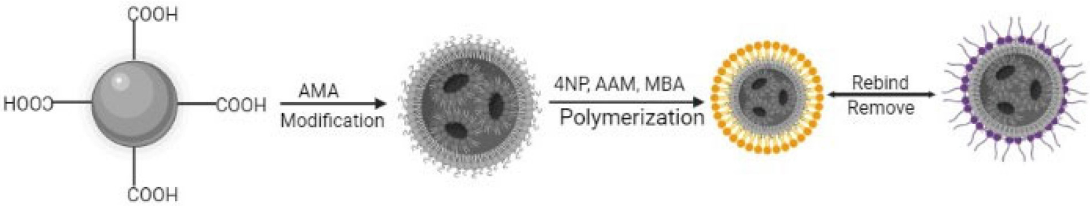


Figure 7. Proposed fabrication procedure for the QD@MIPs nanosensor are depicted schematically.

centrifuged, and then washed twice with anhydrous ethanol and distilled water. To prepare the MIP-coated GQDs for use, the templates were removed, rinsed in 8 mL water using ultrasonication, and stored in the refrigerator until use. The main procedures of manufacturing MIP-coated GQDs are shown in Figure 8.

Precipitation polymerization method can be used to separate and purify polymers. A "good" organic solvent is used to dissolve the polymer sample, and this solution is subsequently transferred to a "poor" solvent. When the polymer is introduced into the insufficient solvent, the polymer chains break apart, clump together, and precipitate out of solution. Because of its high efficiency, low cost, and lack of need for a surfactant, precipitation polymerization is a useful method for making a wide range of homogeneous polymer particles. Novel molecularly imprinted polymers (MIPs) based on Mn doped ZnS quantum dots (QDs) with molecular recognition capacity were effectively synthesized by precipitation polymerization utilizing 2,6-dichlorophenol (2,6-DCP) as template, as reported by Wei *et al.*[115] The conventional synthesis involved producing Mn-doped ZnS QDs in aqueous solution in a way similar to that described in previous literatures with a minor modification.[116,117] Methacrylic acid (MAA) and ethylene glycol dimethacrylate (EGDMA) were utilized as a functional monomer and cross-linking agent, respectively, to create the 2,6-DCP-imprinted polymer on the surface of ZnS:Mn QDs. After adding all of the synthesis materials to the acetonitrile solvent, the 50 mg of ZnS:Mn QDs were dispersed using ultrasonication. After that, a quantity of 10 mg of the initiator AIBN (2,2-azobis(2-methylpropionitrile)) was added, and the mixture was purged with nitrogen for a period of thirty minutes. The general procedure for synthesizing MIPs-ZnS:Mn QDs is depicted in Figure 9. The polymerization reaction yielded MIPs-ZnS:Mn QDs, which were then centrifuged and washed multiple times with acetonitrile and ethanol to get rid of any residual monomers. The non-imprinted polymer (NIPs-ZnS:Mn QDs) was likewise synthesized in the same way without 2,6-DCP analyte.

3. Application of MIP@QDs in Wastewater

In MIP-based QDs sensors, the MIP serves as both the recognition unit to interface and bind specifically with the desired target compound and the transducer component to signal the binding event. Their sensing technique for sensitive pollutant detection is based on the measurement of the changes in optical responses of the transducer caused by the creation of a complex between the desired compound and MIP, and it is simple, fast, and inexpensive. Recent years have seen a rise in the popularity of MIP based QDs sensors, the optical sensing platform for detecting trace amounts of a wide range of analytes because of their high

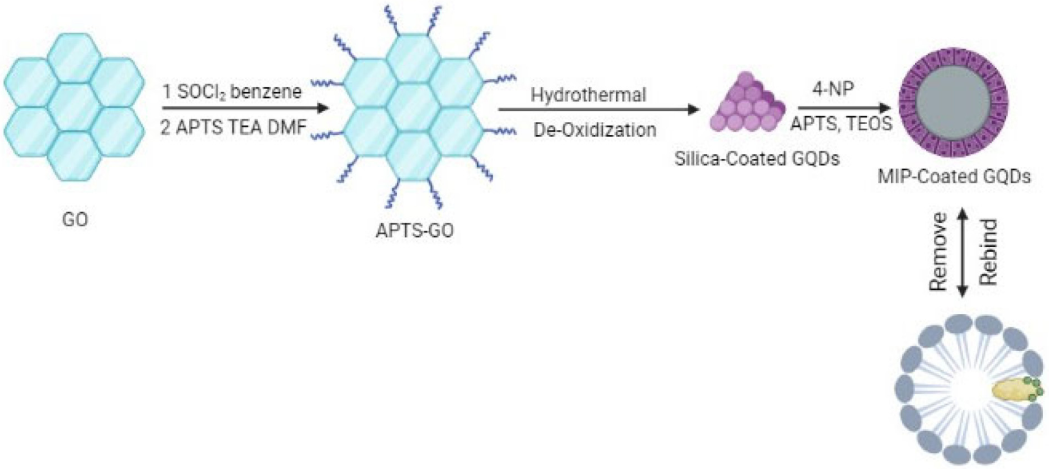


Figure 8. A graphical image of the process of coating GQDs with MIP.

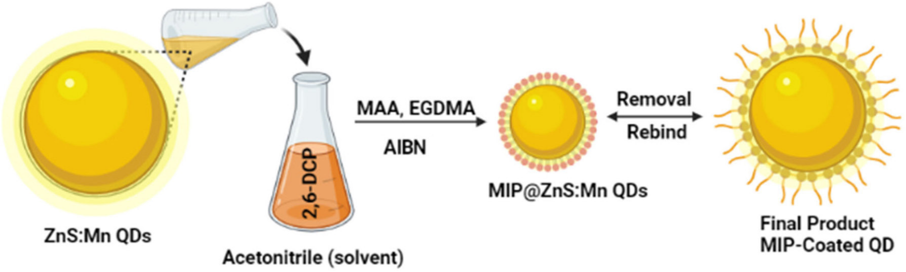


Figure 9. Flowchart illustrating the synthesis of MIPs-ZnS:Mn QDs.

sensitivity, simplicity, and practicality. Table 3 provides an evaluation of various sensing materials for determining the significant pollutants.

Table 3 is a comparison that was made between the reported methods for pollutants. The MIP@QDs are comparable to or even better than the described methods. In comparison to previous nano-probes, the results show that the MIP@QDs nano-probe is both more sensitive and has a more desirable limited detection range. This extraordinary performance can be due to the outstanding interaction that existed between the nano-sensor material (MIP@QDs) and the analyte that was present in the solution. The selective polymer-based QDs nano-sensor displayed an excellent level of performance. Conversely, the performance of the non-selective materials based QDs nano-sensor was clearly lower to that of the selective polymer QDs nano-sensor, since the QDs depends on non-selective materials, its results are consistent with low analyte detection in the solution.

3.1. MIP@QDs for the detection of antibiotics

There has been a dramatic rise in the use of pharmaceutical products, such as antibiotics, for the treatment of diseases. The pharmaceutical manufacturing industries discharge a large amount of wastewater containing pharmaceutical substances into the environment. The mishandling of these products provokes huge environmental challenges and poses serious health risks to both humans and animals.[126] Most often, the exposure to harmful compounds present in wastewater discharged by the pharmaceutical manufacturing industries leads to the emergence of different kinds of diseases in living organisms.[127] According to Klein *et al.*[128] there has been a sharp rise in the consumption of antibiotics; precisely, there has been an increase of 65% in antibiotics use between 2000 and 2015. A global survey conducted by the World Health Organization (WHO) in 2018 showed that there has been an increase of 14,272 tons in antibiotics consumption worldwide.[129] Most of the antibiotics consumed worldwide, including tetracycline, are found in dairy products; these drugs/compounds are used as chemical preservatives and their excessive consumption may lead to vision and skin problems, as well as teeth discoloration in human beings.[130,131] Because of the negative impact of these compounds on human health, it is essentially important to develop efficient techniques for the detection of these substances in aqueous systems. Zhou *et al.*[132] employed graphene quantum dots with molecularly imprinted polymer (MIP@GQD) recognition material for the detection of tetracycline antibiotics in milk samples. The MIP-based GQD sensor exhibited fluorescence emission at 410 nm and the fluorescence intensity of the sensor was found to decrease as the concentration of the analyte

Table 3. Evaluation of various sensing materials for identifying major contaminants

Analyte	Method	Sensing materials	LOD	Reference
Acetamiprid	fluorometric	CQD@MIPs	110 pM	118
Acetamiprid	fluorometric	FRET between Cy5.5 and QDs	0.02 µM	119
Diazinon	fluorometric	CdTe/CdS QDs	0.16 mg kg^{-1}	120
Diazinon	fluorometric	MIP@ZnS:Mn^{2+}	164 nM	121
Sulfadiazine	fluorometric	CdS quantum dots	8.0 µM	122
Sulfadiazine	fluorometric	MIP@QDs	0.7 µM	123
Tetracycline	fluorometric	MIP mesoporous silica @QDs	15.0 ng mL^{-1}	124
Tetracycline	fluorometric	MoS$_2$ QDs	50 µM	125

LOD: limit of detection; QDs: quantum dots; CQDs: carbon QDs; MIP: molecularly imprinted polymers; FRET: Forster resonance energy transfer; Cy5.5: cyanine5.5.

was increased; the proposed sensor exhibited a limit of detection of 1 µg L^{-1}. Molecular imprinting technology is used to create cavities in the polymer matrix based on the shape of the analyte molecule (used as template). The combined application of molecularly imprinted polymer and QDs material in the construction of fluorescent sensors has led to a significant improvement in the selectivity of the fluorescent material. Shi *et al*.[123] employed molecularly imprinted polymer coated with CdTe quantum dots for the determination of sulfadiazine in aqueous medium. The three-dimensional structure was synthesized in the presence of sulfadiazine-which was used as a template, 3-aminopropyltriethoxysilane (APTES)-used as functional monomer, and tetraethyl orthosilicate (TEOS) used as cross-linking agent. The analysis of the interaction between the analyte and the fluorescence sensor was conducted by fluorescence spectroscopy. Under optimized conditions, the fluorescent sensor exhibited good results with limit of detection of 0.67 µM and recovery percentages ranging between 91.8 and 109.4%. Through the application of the sol-gel method, Chullasat *et al*.[133] employed molecularly imprinted polymer coated on CdTe quantum dots for the detection of amoxicillin in real samples where they reported significant improvements in the selectivity of the fluorescent sensor. The fluorescent sensor was successfully applied for the detection of amoxicillin in egg, milk and honey samples with satisfactory recovery percentages of 85-102%; the limits of detection and quantitation obtained were 0.14 and 0.46 µg L^{-1}, respectively. Figure 10 shows a schematic representation of the mechanism involving the fabrication of the fluorescent sensor used for the determination of amoxicillin.[133]

In another recent study related to the use of quantum dots for analytes detection, Yu *et al*.[134] employed an interesting technique which involved the functionalization of the surface shell of carbon quantum dot embedded SiO_2 with tetracycline templated molecularly imprinted polymer (MIP) to boost the efficiency of the fluorescent sensor when applied toward the detection of tetracycline in river water. The method applied by the authors yielded satisfactory recovery percentages in the range of 101.2 to 104.1%, with detection sensitivity of 9.7 mg L^{-1}. As pointed out in previous studies, one can obtain better detection performance and selectivity when highly sensitive materials such as artificial receptors (MIP) are employed in the construction of sensors; clearly, quantum dot nanoscale materials have proven to possess good optical properties and excellent stability which make them suitable for application in this regard.[135,136] At this juncture, it has now become a general consensus that the use of a hybrid nanocomposite material in the fabrication of fluorescent sensors contributes effectively toward enhancing the detection and selectivity of the sensing devices when applied for the detection of analytes of interest in wastewater. Sa-nguanprang *et al*.[137] used a hybrid nanocomposite fluorescent sensor to monitor thiamphenicol in milk samples. The hybrid fluorescent sensor was synthesized using mesoporous carbon and CdTe/CdS/ZnS quantum dots coupled with a specific memory polymer (MIP). The fluorescent sensor was able to successfully detect the presence of thiamphenicol in milk samples with satisfactory recoveries ranging from 93.1 to 100.1% and limit of detection of 0.04 µg L^{-1}. Virginiamycin is a streptogramin antibiotic, which has anti-microbial properties; this compound is widely used for the treatment of bacterial contaminations in animals because of its efficient antimicrobial activity against both Gram-positive and Gram-negative microbes.[138] After its application for the treatment of bacterial contamination

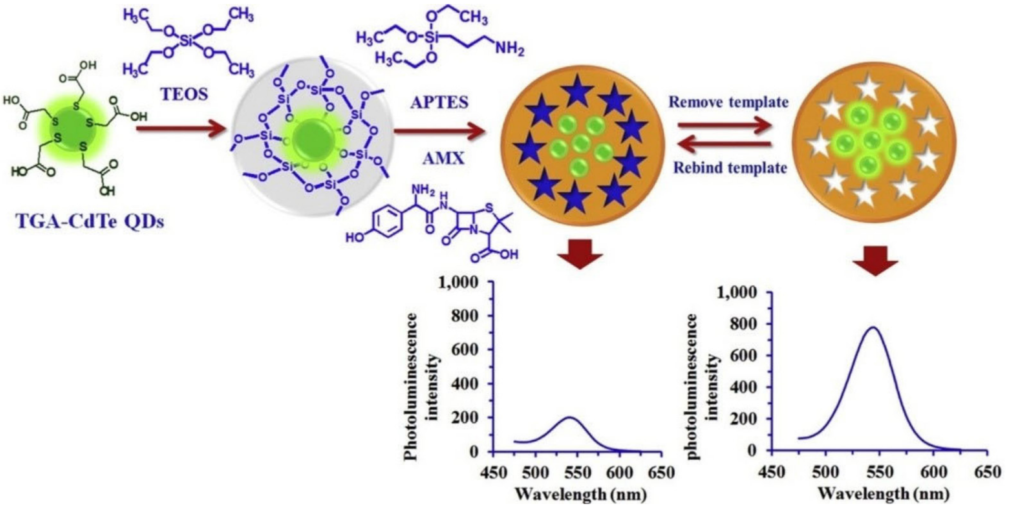


Figure 10. Schematic representation of the mechanism involving the construction of MIP@CdTe QD fluorescent sensor (reproduced from reference 133 with copyright permission 2018 from Elsevier).

in animals, the residues of virginiamycin in meat products may cause hazardous effects on the health of humans and animals.[139] Graphene is a two-dimensional honeycomb lattice nanostructure made up of a single layer of atoms which has certain physical properties, including uniquely large surface area, good thermal stability, and exceptional optical properties,[140] in view of its excellent properties, this carbon allotrobe is used as the base material for the enhancement of signal/detection in fluorescence sensors. The use of graphene coated carbon quantum dots nanomaterials in the construction of fluorescent sensors contributes effectively toward improving the selectivity and strength of these sensors. Li *et al.*[139] employed a MIP-based fluorescence sensor based on graphene oxide and carbon quantum dot (GO/C-dot) for the detection of virginiamycin; the fluorescence intensity of the GO/C-dots in the MIP can be turned "ON" and "OFF" by removing and adsorbing virginiamycin in the MIP cavities, see Figure 11 for an illustrative representation of the synthesis scheme of the fluorescence sensor.[139]

The following is a brief description of the synthesis. Firstly, the hydrothermal strategy was used to synthesize the GO/C-dots, and the material was coated on indium tin oxide (ITO) electrodes. The material was then subjected to electropolymerization in the presence of *o*-aminophenol used as functional monomer, and virginiamycin used as template molecule. The fluorescence sensor exhibited excellent stability, good selectivity, and high fluorescence intensity, with limit of detection of 1.56×10^{-11} mol L^{-1}.

3.2. MIP@QDs for the detection of pesticides

Pesticides are chemicals or mixtures which are used to prevent the emergence and spread of plant diseases, weeds, and bugs, as well as to improve the quality of food products.[125] Agriculture and forestry are the two major sectors that account for the largest amount of pesticides consumed in the economy; these sectors contribute immensely toward the pollution of the ecosystem.[141] The sharp rise in global population has led to the growing need to produce crops and other food products to feed people worldwide. Pesticides kill the bugs that regulate the effects of weed species on the growing plants. The rampant utilization of pesticides that contain harmful compounds in agricultural production causes negative effects on the quality of plants/crops and the whole ecological structure of the area and leads to the pollution of water bodies. According to the data reported in the literature, the global consumption of crop-protecting chemicals is distributed as follows: nearly 47.5% of herbicides, 29.5% of pesticides, and 17.5% of fungicides.[142] Based on the report published by the US Environmental Protection Agency,[143] 50% of water pollution worldwide is caused by the direct discharge of pesticides used in agricultural production. Water pollution, which is caused by the spread of pesticides into the environment and water bodies, has been found to pose serious risks to the health of humans (vital organs in particular) and other living beings (animals and aquatic species).[144] In view of that, it is essentially crucial to develop rapid, cost-effective and efficient techniques that are capable of effectively determining the presence of pesticides in biological and environmental matrices. Fluorescence-based detection with high sensitivity is considered a useful technique for monitoring pesticides in fluid phase. Yu *et al.*[92] employed a fluorescence nanosensor for the successful detection of 4-nitrophenol (4-NP) in water samples. Silica (SiO_2) has also been applied as a substrate for the construction of MIP-based QD nanosensor, where it functions as a core material or as a shell around the QDs material; however, it should be noted that the use of silica in this technique may reduce the QDs fluorescence and diminish the sensing sensitivity.[145] Bearing that in mind, in the aforementioned article, the authors employed the one-spot synthesis approach to fabricate MIP-based QDs sensor without using any matrices such as SiO_2 or the embedding technique. The MIP was polymerized on the surface of QDs material-QD@MIP. The application of the QD@MIP nanosensor in aqueous samples yielded

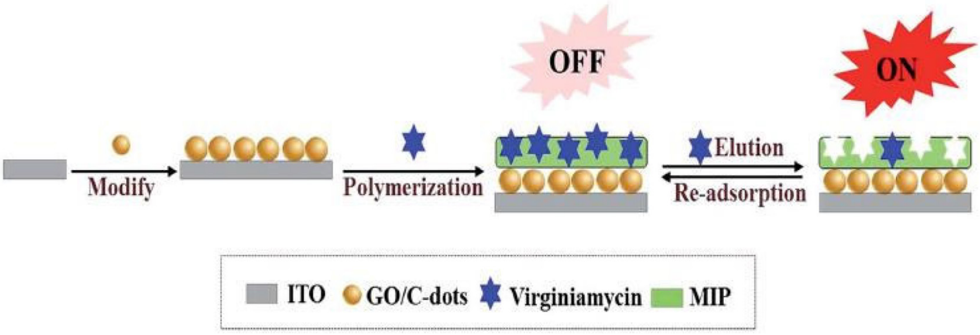


Figure 11. Graphene-oxide coated carbon quantum dot (GO/C-dot)-MIP fluorescence sensor detection of virginiamycin (figure from reference 139 with CC-BY attribution).

excellent results, with sensitivity of 0.051 μM and recovery percentages in the range of 92.7-109.2%. As pointed out in the literature, perovskite-based QDs materials have been found to exhibit higher photoluminescence properties compared to most conventional CdSe-based QDs; this improvement in photoluminescence properties is derived from the surface alterations in the CdSe-based QDs material.[146] Hybrid organic-inorganic lead halides, such as $MAPbX_3$, where MA = $CH_3NH_3^+$ and X = Cl^-, Br^- and I^-, have become a massive part of perovskite-based QDs materials because of the significantly rapid developments made in the energy conversion efficiencies of perovskite solar cells.[147,148] Anion-exchange reaction causes instability in halide QDs materials, and this makes these types of QDs materials unsuitable for application in analytes detection.[149,150] It should be noted, however, that the use of inorganic matrices (SiO_2) as substrate has been found to improve the stability of perovskite-based QDs materials.[151] Due to its excellent photoluminescence properties, the use of perovskite QDs material for fluorescence detection has sparked considerable interest among researchers.[152] Recently, Tan *et al.*[153] employed perovskite ($CsPbBr_3$)-based QDs material coupled with molecularly imprinted polymer (MIP@$CsPbBr_3/SiO_2$) for the sensitive fluorescence detection of phoxim in real samples. The fluorescence nanosensor exhibited an excellent limit of detection of 1.45 ng mL^{-1} with satisfactory recovery percentages in the range of 86.8-98.2%. Shirani *et al.*[154] also employed silica-based QDs (SiO_2-CdS) coupled with molecularly imprinted polymer for the selective fluorescence sensing of indoxacarb in environmental and biological samples; the proposed alternative approach exhibited satisfactory results with five replicate detections conducted successfully at 21 and 60 nM, respectively, and recovery percentages in the range of 95-106%. Other studies reported in the literature[155,156] have shown that the application of ZnO quantum dots materials in the construction of chemo-sensors led to high quantum yield, wide excitation spectrum, and narrow emission spectrum. Other advantages of the application of ZnO QDs under the fluorescence sensing approach include the following: less toxicity, good bio-compatibility, cost-effectiveness, environmental friendliness, and ease of synthesis.[157,158] Vahid *et al.*[159] enhanced the sensing functionality of fluorescence nanosensor, which was constructed using MIP-coated ZnO QD materials and applied for the specific detection of dimethoate (DM); the sensing device was successfully applied for the detection of DM in water samples where it exhibited a limit of detection of 0.006 mg L^{-1}. Ge *et al.*[160] also employed CdTe quantum dots with deltamethrin template MIP for the detection of the pesticides. The deltamethrin target compound has caused the mechanism that stops the reaction from happening. In the ideal conditions, a Stern-Volmer type equation showed that the relative fluorescence intensity of CdTe-SiO_2-MIPs decreased with increasing DM concentration in the range of 0.5-35.0 μg mL^{-1}. As a result, 0.16 μg mL^{-1} is the lowest detectable concentration. CdTe-SiO_2-MIPs sensor was used to analyze food samples to quantify the concentration of deltamethrin (DM). 4-Nitrophenol (4-NP) compound is used in the production of pharmaceuticals, fungicides, pesticides, and colors. So, using 4-NP requires close monitoring and supervision. The maximum concentration of 4-NP in drinking water is 60 ng mL^{-1}, according to the Environmental Protection Agency (EPA) in the United States.[161] Zhou *et al.*[114] used graphene quantum dot combined with MIP to monitor *para*-nitrophenol (4-NP) in liquid matrix. The use of MIP-based fluorescence sensors for the detection of analytes has gained attraction among researchers due to their outstanding advantages; these sensors have been found to be environmentally friendly, in addition to exhibiting stable fluorescence, good screening results, excellent selectivity, and speedy response. Ren and Chen[162] employed manganese-doped ZnS QDs coupled with MIP for the detection of cyphenothrin (see Figure 12). Cyphenothrin concentration in water samples were determined using this material, with recoveries ranging from 88.5 to 97.1%.

Agrochemical manufacturers employ levulinic acid to create diphenolic acid, which is then used in the formulation of fertilizers and insecticides.[163] Recent research[164] has identified ties between the compound and major human health issues. Since this is the case, aqueous solution tracing of the component is essential. Chen *et al.*[163] used dummy-template molecularly imprinted polymer in the presence of diphenolic acid (DPA) in combination with Mn-doped ZnS quantum dots to enhance the detection ability of the sensor.

3.3. Using MIP@QD for heavy metals detection

The term heavy metals is used to refer to a group of elements that have atomic weight in the range of 63.5-200.6 g mol^{-1} and atomic density of more than 4000 kg m^{-3}.[165,166] Studies reported in the literature[167,168] have shown that the exposure of living creatures to even low metal ion concentration in aqueous systems leads to toxicity. A few examples of heavy metals include copper, cadmium, zinc, chromium, arsenic, boron, cobalt, titanium, strontium, tin, vanadium, nickel, molybdenum, mercury, lead, to name a few. Heavy metals like copper, zinc, nickel, boron, iron, and molybdenum are beneficial when applied in the soil for the enhancement of flora growth; however,

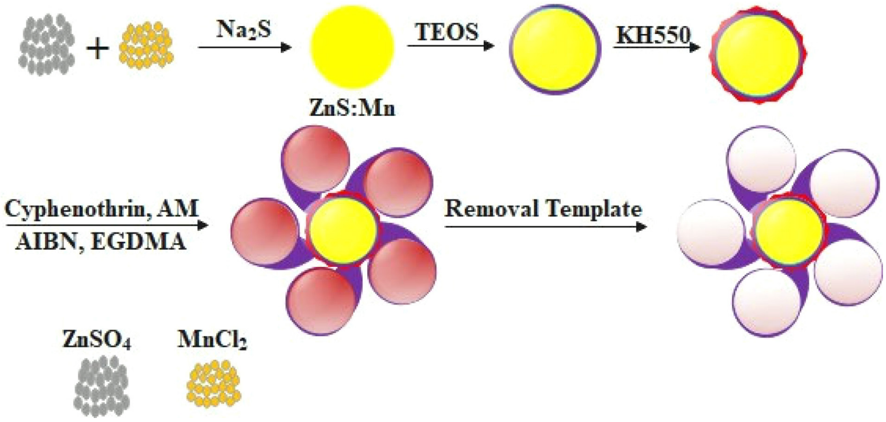


Figure 12. The step-by-step construction of MIP-coated QDs (adapted from reference 162).

the exposure of living organisms, including human beings, aquatic creatures and flora, to these metals at concentrations above the permissible threshold has been found to be extremely dangerous. Other heavy metals such as lead, mercury, cadmium, and arsenic do not play such a vital role in the growth of vegetation and animals. The soil is subject to pollution because of the heavy metals present in industrial wastewater, sewage sludge, fertilizers and treated wastewater which find their way into it through land use, crop production, leeching, and weathering of soil minerals.[169] Apart from contributing massively toward soil pollution, the presence of some heavy metals in the soil can diminish soil fertility and affect the natural nutrients present in it, thus undermining crop production and causing serious health problems in humans-damages to the vital organs of humans and other living beings.[170] Despite the fact that fluorescent QD sensors have been employed for the analysis and detection of harmful compounds in wastewater, the low detection efficiency of the sensing devices is still a key challenge for researchers in the field.[171] Like molecularly imprinted polymer, ion-imprinted polymer (IIP) is an artificial receptor material that possesses specific metal ion cavities and has the ability to detect metal ions in aqueous media. Wang *et al.*[172] showed that the combined application of Cu-ion-imprinted polymer with carbon quantum dots nanoscale led to an improvement in the fluorescent sensing efficiency of the material; according to the authors, the nano-sensor (CQDs@Cu-IIP) was successfully applied for the detection of copper(II) ion in water samples, where it exhibited excellent recovery rates ranging from 99.29-105.42%. The use of doped QDs as a new class of luminescent materials has drawn considerable attention among researchers since the dopants can influence the optical behavior of QDs. Zhang *et al.*[173] used QDs material functionalized with ion-imprinted polymer for the successful detection of Cr^{VI} in water samples. Other studies reported in the literature have also shown that the addition of Mn ion to ZnS quantum dots enhances the fluorescent intensity of the nanosensor,[174] while the use of ion-imprinted polymer improves the sensor specificity toward the target model pollutant. With the improvement of miniaturization and integration, the use of 3-D microfluidic paper-based analytical devices (µPADs) has sparked considerable interest among researchers.[175,176] Compared to silicon wafers and glass, paper chips are inexpensive and easy to fabricate. In addition, papers are tiny materials that are easy to hold and transport and are ecologically user-friendly once they can be burned after use; also, paper chips can be employed in combination with fluorescence materials in a wide detection range.[177,178] Using the paper chip sensing platform technique, Zhou *et al.*[179] employed ion-imprinted polymer coated with fluorescent ZnSe quantum dots on 3D rotary microfluidic chip (µPADs) for the recognition of Cd^{2+} and Pb^{2+} ions in aqueous samples. Under optimized conditions, the applied technique yielded low limits of detection of 0.245 and 0.335 µg L^{-1}, for Cd^{2+} and Pb^{2+}, respectively. In another study reported in the literature, Shao *et al.*[180] successfully employed carbon quantum dots embedded ion-imprinted polymer for the fluorometric determination of lithium ion in water samples; the fluorescence nanoscale sensor exhibited satisfactory recovery percentages ranging from 102 to 111.6% and a low limit of detection of 16 µmol L^{-1}.

3.4. Using MIP@QD for detection of other emerging pollutants

Emerging contamination is a standard terminology used to refer to the environmental hazards related to the release of new waste products that have unintended consequences, but which are likely to cause damages to the environment.[181,182] More than 700 emerging contaminants, including metabolites and transformation products, have been detected in European aquatic ecosystems.[183]

Synthetic or naturally occurring compounds that have the potential to infiltrate the environment and produce known or suspected detrimental ecological and (or) human health consequences are referred to as "emerging pollutants" (Eps).[183] The harmful effects caused by the discharge of emerging pollutants into the environment have become an issue of great concern to researchers worldwide.[184] Emerging contaminants consist of products manufactured by pharmaceutical companies, beauty products, endocrine-disrupting chemicals, fire retardants, fertilizers, among others.[185] Pharmaceutical manufacturing industries have been reported to discharge a wide range of emerging pollutants into aquatic systems with no treatments, posing serious risks to living organisms.[186] Diethylhexyl phthalate also referred to as phthalate acid esters, is an organic compound which is considered an emerging pollutant; this pollutant is generally used in the preparation of plastic and other consumer items.[187] The use and disposal of these items may lead to the discharge of diethylhexyl phthalate into the nourishment chain, water bodies, environment,[188] and in human organisms after or amid their life cycle.[189] The exposure of diethylhexyl phthalate to the endocrine system of living beings causes cancer, growth and neurodevelopment disorders, and genetic diseases.[189,190] Several alternatives and lab-made methods have been proposed for the determination of emerging pollutants in wastewater. Wang *et al*.[191] developed a fluorescent sensing approach for the effective monitoring of diethylhexyl phthalate in aquatic samples. Under this approach, the MIP was coated with fluorescence ZnO quantum dot nanoparticles and synthesized using the polymerization method in the presence of diethylhexyl phthalate (used as template), methacrylic acid (used as functional monomer), ethylene glycol dimethacrylate (employed as cross-linker), and 2,2'-azobis(2-methylpropionitrile) (used as an initiator). The proposed technique yielded good results, with satisfactory recovery percentages ranging from 97.50 to 106.10% and relative standard deviation (RSD) below 3.86% based on the application of three diethylhexyl phthalate (DEHP) concentration levels in real water.

The fluorescence intensity of graphene-based quantum dot (GQD) sensor can be enhanced significantly by doping the material with other hetero-atoms; in addition, this can improve the intrinsic properties and the quantum yield of GQDs. For instance, the incorporation of nitrogen into graphene quantum dots (N-GQD) leads to the development of a sensor with high quantum yield, greater fluorescent strength, and stable photoluminescence properties. Liu *et al*.[192] developed a strip-based sensor using poly-dopamine (PDA) molecularly imprinted polymer (MIP) and nitrogen-doped graphene quantum dots (N-GQD) where the device was applied for the quantitative detection of thiacloprid. Under optimized parameters, the proposed strip-based sensor exhibited a limit of detection of 0.03 mg L^{-1}. Shirani *et al*.[193] also employed a MIP coated silane-functionalized carbon dots (MIP@Si-CD) fluorescence sensor for the detection of acetamiprid specific target molecule in wastewater. The authors used the hydrothermal method in the presence of citric acid and 3-aminopropyltriethoxysilane (APTES) to produce organosilane functionalized carbon dots. Through the application of the sol-gel technique, the surface chemistry of Si-CD was modified using MIP. The fluorescence signal of the proposed MIP@Si-CDs showed linear response with acetamiprid (ACT) concentration in the 7-107 nM range and a limit of detection of 2 nM. Kim *et al*.[194] employed silica imprinting polymer coated with QDs material (CdSe) for the detection of bisphenol A (BPA). The quantum dot (CdSe) material was applied as a signal transducer in the proposed sensor. Several approaches have been used to modify the surface of the imprinting polymer with fluorescence materials; fluorescence materials are used as modifiers in MIP-based sensors in order to enhance the detection efficiency of the imprinting polymer. The tetrabromobisphenol A (TBBPA) compound may be included in the list of emerging pollutants. There is evidence that TBBPA is an endocrine disruptor that leads to neurobehavioral and immunotoxic consequences, oxidative stress, and apoptosis.[163] The findings of the study showed that the proposed MIP-based fluorescence sensor exhibited high affinity toward TBBPA and good recovery percentages (ranging between 80.2 and 96.5%), with relative standard deviation below 8.0%. In another study,[195] the authors employed a MIP-based chemo-sensor for the selective recognition of adenosine 3':5'-cyclic monophosphate where they used fluorescent dye material in the selective binding sites of the MIP, the modification of the sensor with fluorescent dye material helped enhance the fluorescence recognition of the analyte in fluid phase. The findings of the study showed that the colorant material was quenched upon the rebinding of the analyte molecule to the MIP.

3.5. MIP@QDs for detection of dyes

Dyes are natural or manufactured organic substances that are used for coloring items/objects of interest. Dyes get stuck with the substrate via chemical bonding. There are two different groups of dyes: chromophores and auxochromes. Chromophores can absorb and reflect a given color while auxochromes influence the coloring capacity of dyes (chromophores). Some organic dyes are soluble in water while others are insoluble in water. Natural dyes are derived from natural sources such as tree barks, leaves, fungi, wood, and roots, whereas synthetic dyes are produced from man-made petrochemical substances. In the dyeing process, a variety of colors are released into the water during the coloring and completing of textures. Dyes are composed of organic compounds and can be found in a wide range of things, from human hair to clothes; human exposure to dyes has been found to cause numerous diseases and can pose serious health risks to the indispensable organs of the human body.[196] Reports in the literature show that approximately 100,000 dyes are accessible commercially and around 7×10^5 tons of dyes are manufactured annually (worldwide).[197] Organic synthetic dyes are highly soluble and are directly released from dyeing industries into aqueous environments.[198] Organic dyes are made up of only one azo group which connects two chemical groups (for example, aromatic systems like the phenyl group), resulting in water pollution. Studies reported in the literature[199,200] have shown that, despite its carcinogenic and mutagenic effects on humans, most countries illegally employ the Malachite Green dye (MG) as antifungal and antibacterial in aquaculture. Concerned about the negative effects of this pollutant, Wu *et al.*[201] employed a fluorescent sensor based on MIP coated QDs (CdTe) materials for the rapid monitoring of MG dye in water samples. The authors employed the precipitation technique to synthesize the MIP coated QDs material (MIP@QDs). The fluorescent sensor was used to construct calibration curves in the concentration range of 0.1-20 µmol L^{-1}. The proposed sensor exhibited a limit of detection of 0.059 µmol L^{-1} and excellent recovery percentages of 94.8 and 98.1% when applied for MG detection in water samples. All the optimized experiments showed that the proposed MIP@QDs material can be reused in further analyses. Figure 13 shows the proposed fluorescent sensor (MIP@QDs) used for the detection of MG dye.[201]

Currently, the attention of researchers has been drawn toward the use of dual-emission molecularly imprinted sensors for the detection of compounds in environmental samples.[202] In this sensing mechanism, carbon dot doped silica core is employed as reference signal and the application of the technique leads to an integrated remediation of environmental effects.[192] Gui *et al.*[203] used dual emission molecularly imprinted mesoporous microsphere for the rapid and selective detection of MG dye in river and lake water samples. For the development of this fluorescent sensor, the green fluorescent QDs (CdTe) material was incorporated into the core of silica nanoparticles, while the other red fluorescent QDs (CdTe) material was implanted around the silica core; the combined application of MIP with the fluorescence sensor led to excellent selectivity of the device. The authors analyzed the sensor response in the concentration range of 27.4 nM-13 µM, where they obtained a limit of detection of 17.0 nM and recovery percentages ranging from 95.8-106.8% in river and lake water samples.

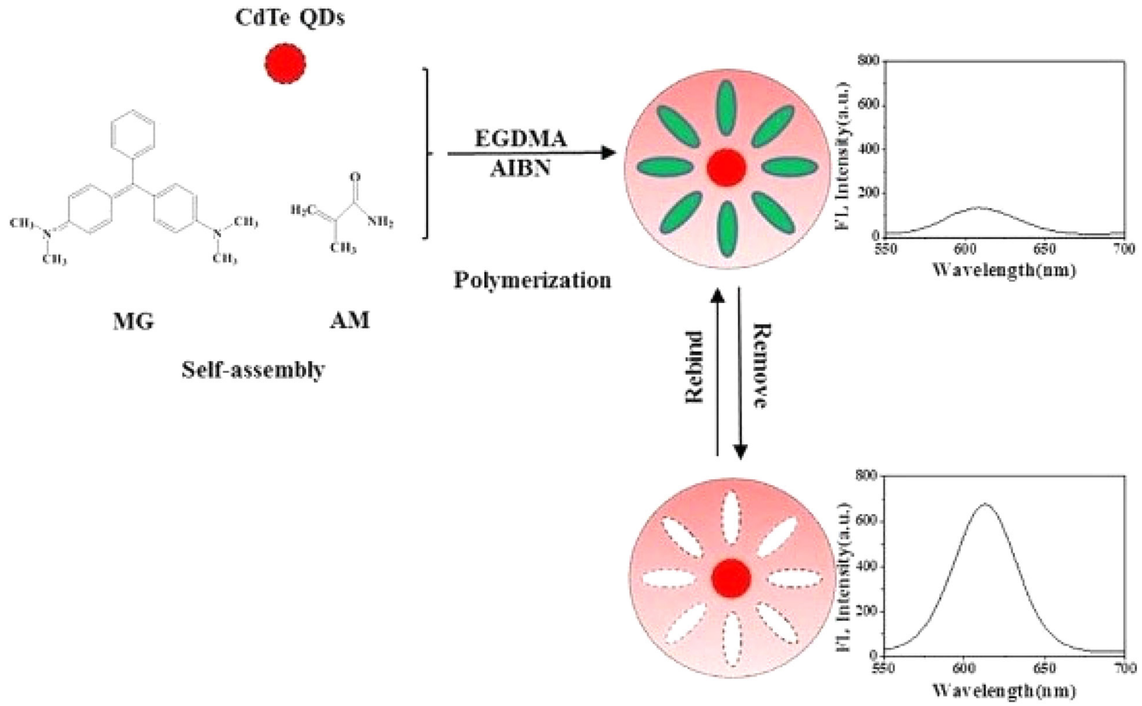


Figure 13. Graphical representation of the MIP@QDs-based sensor. Molecular imprinted polymer synthesized using various reagents in polymerization process (reproduced from reference 201 with copyright permission 2017 from Elsevier).

Shi *et al.*[204] also employed the dual emission fluorescence technique for the determination of MG dye in fish farming water. The sensor was constructed using mesoporous molecularly imprinted polymer combined with C_3N_4 and CdTe fluorescent materials. The red fluorescence of CdTe QDs with excitation/emission wavelengths of 350/680 nm is quenched by MG, while the blue fluorescence of C_3N_4 with excitation/emission wavelengths of 350/458 nm is unaffected. The intensity of the fluorescent sensor increased linearly in the range of 50-1000 ng mL^{-1} and the limit of detection obtained was 10 ngm L^{-1}. The recovery percentages obtained for the fish farming water samples ranged from 92.5-97.8%.

4. Practical Implementation of MIP@QDs Sensors

4.1. Importance of the development and application of MIP@QDs sensors

A nation's quest for economic, scientific and technological advancement inevitably leads to the development and expansion of its industrial sectors. As the number of industrial sectors increases, the quality of the country's freshwater declines due to the widespread disposal of hazardous substances in its water bodies, causing environmental degradation and serious health problems for the population. This appalling phenomenon has raised serious concerns among researchers, and numerous studies are being conducted targeted at devising efficient, eco-friendly and low-cost sensing techniques that are capable of determining the presence and occurrence of harmful pollutants in wastewater to help tackle this global catastrophe. One of the pollutants determination techniques which have gained ample traction among researchers is the molecularly imprinted polymer technique; this technique has been progressively utilized over the last ten years and has been successfully applied for the detection of hazardous pollutants in environmental matrices. MIPs are artificially engineered materials which are used as recognition components in the fabrication of sensors due to their relatively higher thermal stability, great reusability potential, and better selectivity compared to biological recognition materials. The use of these polymeric materials leads to positive changes in the physical parameters (including absorbance, refractive index, and functionality of the target-specific molecule) of the sensing technique. In this sense, the combined application of MIP and QD material in the construction of sensors leads to significant improvements in the detection efficiency and signal intensity at low concentrations. The use of MIPs in detection strategies can effectively help to remove the organic and inorganic toxic compounds from the fluid phase. MIPs are stable materials which are capable of resisting variations in experimental parameters, including pH, temperature, and organic solvent. MIP-based sensing techniques have several other advantages over conventional techniques; among these advantages include ease of fabrication, low cost, and long-term durability. MIP-based techniques have also become more appealing for future industrial requirements due to their high recovery rates of target metal ions (analyte) in real samples and the selective elimination of analytes from complex effluents. The basic foundation for the synthesis of MIPs was laid out by Wulf[205] and Takagishi and Klotz[206] in 1972; since then, significant advances have been made in the area with the development of a wide range of MIP-based techniques using a variety of modifiers targeted at ensuring the effective recognition of analytes of interest and the effective elimination of organic and inorganic toxic substances from wastewater. Table 4 presents an outline of the findings of numerous studies reported in the literature based on the application of QDs material coated with molecularly imprinted polymer (MIP@QDs), used as recognition layer, for the detection of different contaminants in real environmental samples; the table also shows the limit of detection and recovery percentages obtained in each study.

4.2. Final remarks and future perspectives

This review has shed light on the development and application of MIP@QDs nanomaterials, which have become suitable platforms for the construction of highly efficient, sensitive fluorescence-based sensors with special optical properties. MIP-based QDs sensors have been successfully employed for the determination of antibiotics, heavy metals, pesticides, dyes, and other harmful emerging pollutants. The review also provided in-depth analyses regarding the surface chemistry of QDs materials, as well as their modification with supporting materials and the detection pathways involving their application. Silica-based MIP-QDs sensors were reported to have gained considerable attraction among researchers because the incorporation of silica into the sensing platform safeguards the unique optical properties of QDs and contributes toward enhancing the thermal and mechanical properties of the imprinted polymer. The use of hybrid organic and inorganic MIP-based QDs sensors has also been found to beef up the optical properties of the sensing devices apart from improving the sensors detection efficiency and recovery ability. According to some studies reported in the literature,[216] the incorporation of silica nanoparticles in QDs

Table 4. QDs material with recognition layer (MIP) for the detection of effluents in environmental samples

	Contaminant	Limit of detection	Matrix	Recovery / %	Reference
Antibiotics	sulfadiazine	0.004 μg L^{-1}	seawater	90.4-99.5	207
	sulfadiazine	0.042 μmol L^{-1}	tape water	91.7-101.2	208
	norfloxcacin	0.18 μM	seawater	96.2-106	209
	ceftazidime	0.11nmol L^{-1}	urine	97.5-107.5	210
	thiamphenicol	0.04 μg L^{-1}	milk	93.5-100.1	157
Pesticides	chlorpyrifos	17 nmol L^{-1}	water	87.1-94.5	211
	pentachlorophenol	86 nM	river water	93-106	116
	diniconazole	6.4 μg L^{-1}	river water	99-102.1	202
	thiacloprid	0.03 mg L^{-1}	lake water	101.1-110	192
	4-nitrophenol	0.051 μM	lake water	92.7-109.2	92
Heavy metals	Cd^{II} ions	25 nM	lake water	86.5-94.8	212
	Cd^{II} ions	0.016, 0.028 mg L^{-1}	real water	99.2-105.4	172
	Cr^{VI} ions	5.48 μg L^{-1}	real water	94.4-106.4	173
	Cd^{II} ions and Pb^{II} ions	0.245, 0.335 μg L^{-1}	sea, lake water	95-101.7, 95-100.7	179
Emerging pollutants	dimethoate	0.006 mg L^{-1}	river water	96.5-99.6	
	p-aminophenol	0.02 μM	lake water	97.3-103.3	213
	diethylstilbestrol	5.9×10^{-5} mg L^{-1}	river water	95.5-107.5	214
	cypermethrin	0.3 μg L^{-1}	sea water	95.6-98.5	215
	atrazine	0.80×10^{-7} mol L^{-1}	tape, river water	92-118	6
Dyes	Malachite Green	0.059 μmol L^{-1}	aquaculture water	94.8-98.1	201
	Malachite Green	17.0 nM	river, lake water	95.8-107.3	203

nanomaterials enhances the fluorescence intensity of these materials. Finally, based on this literature review, one can conclude that the MIP@QDs sensor is a highly promising tool which has proven to be efficient when applied toward the recognition of target molecules in wastewater.

While the use of MIP as a receptor material in QDs sensors significantly enhances the detection efficiency, further studies are needed in order to have a fully comprehensive understanding regarding the synthesis of the QDs/MIP-based materials and the devising of suitable mechanisms for the efficient removal of the templates from the polymeric materials. In addition, further research is needed in order to discover more effective supporting materials for application in QDs with a view to improving the fluorescence intensity of the sensing platform. Based on the published literature, the usage of recognition materials (MIP@QDs) exhibits a higher limit of detection (LOD) in comparison to other sensing materials. The focus materials of this review are simple, rapid, and also acceptable for the quantification of target pollutant chemicals. MIPs are advantageous components that have the potential to be linked to nanomaterials such as QDs in order to construct various sensitive and selective sensors for the detection of hazardous contaminants. If the same QD materials are used as signal transducers, then a different MIP can be coated on the surface of the QD to make it selective for a variety of different pollutant compounds and thus led to enhancing the selectivity of the fabricated sensor. Future research activities should also be targeted at exploring less hazardous QDs materials for the development of sensing platforms as opposed to cadmium-based QDs materials. Clearly, carbon quantum dot is a promising candidate in this regard. Most importantly, future research should be devoted toward developing fluorescence sensors that go hand in hand with green chemistry where these sensing devices are environmentally friendly, cheap to produce, and are able to be reused for consecutive detection analyses so as to prevent the generation of excessive waste. Solution-based sensors can usually be employed only once, and this may lead to the generation of huge amounts of poisonous wastewater. The obstacles and prospective changes should be made in the following areas. As artificial receptors, MIPs in fluorescence detections offer a number of benefits. Some modifications are still required in coupling with QDs for fluorescence detections, their fabrication, and extraction of template molecules in order to decrease non-selective binding and advance the biocompatibility factor in fluorescence measurement. The ability to synthesize QDs with a wide range of visible emission is crucial for future scientific endeavors. The prepared sensor has low sensitivity is a major obstacle in many sensing applications. Because of this, it is possible to consider about increasing both sensitivity and selectivity at the same time. MIP/QDs-based probing is anticipated to make use of not just fluorescence techniques (off and on approaches), but also lifespan changes, spectra shift, bi-modular detection, and ratiometric sensing. One-time use and toxic waste from solution-based probes motivates research on green chemistry techniques and reusable devices that save cost. The development of MIP/QDs-based fluorescence sensors for the detection of contaminants in an aqueous solution based on imprinting procedures is an option that may be taken into consideration.

Author Contributions

Shakeel Zeb was responsible for collection of material, writing original draft, graphical sketching, formatting, revision of final draft; Javier E. L. Villa for original draft, corrections and suggestions; Ademar Wong for original draft, corrections and suggestions; Sabir khan for writing review and editing; Sajjad Hussain for writing review and editing; Maria D. P. T. Sotomayor for conceptualization, supervision, and administration of the team.

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