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FOCUSED REVIEW

Using Post-graphene 2D Materials to Detect and Remove Pesticides: Recent Advances and Future Recommendations

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Abstract

Detection and removal of pesticides have become increasingly imperative as the widespread production and use of pesticides severely contaminate soil and groundwater and cause serious problems to non-target species such as human and animals. Recently, new two-dimensional materials beyond graphene (e.g., transition metal dichalcogenides, layered double hydroxides), called post-graphene two-dimensional materials (pg-2DMs), have exhibited promising potentials in detecting and removing pesticides due to their unique physiochemical attributes such as high photocatalytic activity and large specific surface area. This review summarizes the recent advances of utilizing pg-2DMs to detect, degrade and adsorb pesticides (e.g., thiobencarb, methyl parathion, paraquat). The current gaps and future prospects of this field are discussed as well.

Keywords Post-graphene 2D materials · Pesticide · Detection · Removal · Degradation

Abbreviations

2DMs	Two-dimensional materials
TMDs	Transition metal dichalcogenides
LDHs	Layered double hydroxides
pg-2DMs	Post-graphene two-dimensional materials
OPs	Organophosphate
GO	Graphene oxide
rGO	Reduced GO
g-C_3N_4	Graphitic carbon nitride
h-BN	Hexagonal boron nitride
MXene	Transition metal carbides and nitrides nanosheets
AuNPs	Gold nanoparticles
AChE	Acetylcholinesterase
ATCh	Acetylthiocholine
TBC	Thiobencarb
QDs	Quantum dots
SERS	Surface-Enhanced Raman Scattering
MP	Methyl parathion
BCBF	G-C_3N_4@$Bi_2O_2CO_3$@$CoFe_2O_4$ composites
PQT	Paraquat
RAPOP	G-C_3N_4@amine-rich porous organic polymer
2,4-DCP	2,4-Dichlorophenol
2,4-D	2,4-Dichlorophenoxyacetic acid
2, 4-DB	4-(2, 4-Dichlorophenoxy) butyric acid
4-NP	4-Nitropheno
MCPA	4-Chloro-2-methylphenoxyacetic acid
DNP	2,4-Dinitrophenol
DNOC	2-Methyl-4,6-dinitrocresol
2,4,5-T	2,4,5-Trichlorophenoxyacetic acid

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Pesticides are a group of chemicals, which are designed to kill and control the weeds and pests, and thus protect the agricultural production. According to their chemical compositions, pesticides can be classified into several categories, including organochlorines, organophosphate (OPs), substituted ureas, carbamate, biopesticides and miscellaneous pesticides (Rani and Shanker 2018). Many pesticides exhibit harmful effects on the human health and seriously threaten the environmental safety as well (Eddleston et al. 2008; Casida and Durkin 2013; Henry et al. 2015). Among the used pesticides, organochlorines are the most concerned persistent organic pollutants (POPs) (Rani et al. 2017). Yet, to boost up the worldwide crop production, lots of agrochemicals are still used extensively on crops every year without realizing their negative effects. For example, the consumption of atrazine (a kind of herbicide commonly used in the corn fields) reached 57.39 million pounds in 2005 (Zhang et al. 2011). Now, the largest pesticide consumer in the world is Europe, which is followed by Asia (Khalid et al. 2020).

Since pesticides and their metabolites are toxic and prevalent in the environment, there is an urgent need to develop effective, low-cost and easy-to-handle approaches for their detection and removal (Pitarch et al. 2010; Rani et al. 2017). To meet this demand, a lot of nanomaterials (e.g., zero-valent Fe nanoparticles (El-Temsah et al. 2016), Fe–Pd bimetallic nanoparticles (Joo and Zhao 2008), TiO_2 nanocomposites (Zaleska et al. 2000)) have been utilized on account of their interesting properties, such as large specific surface area, high adsorption capacity and excellent catalytic properties (more information can be found the related reviews (Rani et al. 2017; Rani and Shanker 2018)). These advances stimulate the scientists to explore the potentials of other materials in solving the pesticide issue.

Graphene, the representative star of two-dimensional materials (2DMs), has been intensively studied in the treatment of pesticides due to its unique physiochemical properties. For instance, Zhang et al. prepared the cellulose/graphene composites, which exhibited strong adsorption behaviors towards six triazine pesticides (belonging to cationic pesticides) in the aqueous solutions. After six times of recycling, the adsorption efficiency of this composites was still over 85% (Zhang et al. 2015). Moreover, nanocomposites containing reduced GO (rGO) and Fe_3O_4 nanoparticles demonstrated a 93.61% adsorption efficiency towards triazine pesticides (belonging to cationic pesticides), which was aroused by the strong electrostatic interaction between the nanocomposites and the pesticides (Boruah et al. 2017). Suo et al. synthesized an activated carbon derived from sieve-like cellulose/graphene oxide composites (ACCE/G). They found the adsorption capacity of ACCE/G was up to 152.5 mg/g for chlorpyrifos (belonging to non-ionic pesticide) (Suo et al. 2018). It is believed that π–π stacking and van der Waals interactions are the major adsorption interactions between GO and pesticides (Wang et al. 2020). In fact, many other 2DMs beyond graphene, called post-graphene 2D materials (pg-2DMs), have already garnered increasing attentions in the detection and removal of pesticides. However, to the best of our knowledge, the advances in this field are not summarized yet. To fuel the development of this direction, the present review is motivated with emphasis on the pg-2DMs-based methods for pesticide detection and removal.

Basic Introduction of Post-graphene 2DMs

2DMs are layered materials that have large lateral size, but only one or few atoms in thickness (Sun and Wu 2018). The great success of graphene boosts the development of other 2DMs and they have displayed promising potentials in different fields, such as disinfection (Tian et al. 2019), catalysis, field effect transistors (Kong et al. 2017) and environmental remediation (Wang and Mi 2017). For example, few-layer MoS_2 with lots of atomically sharp edges and active sites can efficiently inactivate microbes (Zheng, et al. 2020). Meanwhile, because sulfur is a kind of soft Lewis base and shows a high affinity to heavy metal ions (i.e., Ag^+ and Hg^{2+}), few-layer MoS_2 nanosheets with numerous sulfur atoms on their surfaces exhibit high adsorption capacities towards heavy metals (Ai, et al. 2016; Gash et al. 1998). Until now, around 30,000 2DMs related articles are published annually, suggesting 2DMs are the hot spot in scientific research (Yin and Tang 2016). Some members of pg-2DMs, like transition metal dichalcogenides (TMDs) (Liang et al. 2017), graphitic carbon nitride (g-C_3N_4) (Kumar et al. 2018), layered double hydroxides (LDHs) (Otero et al. 2012), hexagonal boron nitride (h-BN) (Atar and Yola 2018) and transition metal carbides and nitrides nanosheets (MXene) (Jiang et al. 2018), have been used for the detection and removal of pesticides. The structures of these materials are shown in Fig. S1. To date, various methods have been developed to prepare pg-2DMs. These methods can be generally divided into two categories: top-down (e.g., mechanical exfoliation and liquid phase exfoliation) and bottom-up (e.g., chemical vapor deposition) methods (Agarwal and Chatterjee 2018).

Applications of pg-2DMs in the Detection and Removal of Pesticides

TMDs

TMDs are MX_2-type compounds, in which X is a chalcogen (e.g., S, Se, and Te) and M is the transition metal from groups IV, V and VI (Chhowalla et al. 2013; Tang and Zhou 2013; Xu et al. 2013). There are quite strong intralayer covalent M–X bonds within bulk layered TMDs. Yet, the adjacent layers are connected by the weak van der Waals interactions.

Owing to their catalytic activities, sensitive surface states and large junction areas toward the electrode or electrolyte, some TMDs (i.e., MoS_2 and WS_2) have received considerable attention in fabricating electrochemical sensors for pesticides. The inherent properties of these TMDs suggest high catalytic activities of edge planes, which can be attributed to their high surface energy (Sinha et al. 2019). However, to boost their inherent electrochemical capacities, some strategies (e.g., doping of non-metal (Song et al. 2018b) or rare earth (Sakthivel et al. 2018), synthesizing metallic 1T-polymorph (Nasir et al. 2017), fabricating 3D architecture (Sinha et al. 2019)) are often needed. Decorating TMDs with conducting nanomaterials also significantly enhances their electrocatalytic capacities and thus improves the accuracies and limits of detection (Govindasamy et al. 2017; Qi et al. 2018; Song et al. 2018a; Zhao et al. 2018; Jia et al. 2020) (Table S1). For example, Zhao et al. prepared metallic 1T-phase MoS_2 nanosheets using the chemical lithium-intercalation method. 1T phase MoS_2 nanosheets were further modified with gold nanoparticles (AuNPs) to fabricate an acetylcholinesterase (AChE) based biosensor for the paraoxon detection (Fig. S2a). The biosensor exhibited a wide linear range (1.0–1000 μg/L) with a detection limit of 0.013 μg/L (Fig. S2b, c) (Zhao et al. 2018).

MoS_2 with single or fewer layer or small planar dimensions (also known as quantum dots (QDs)) shows intriguing optical properties and/or redox activities (Gopalakrishnan et al. 2015; Xiao et al. 2016). Because of this, MoS_2 also demonstrates promising potentials in detecting pesticides by colorimetric sensing (Chen et al. 2017), fluorescence sensing (Fahimi-Kashani et al. 2017), electrochemiluminescence sensing (Yang et al. 2017) and SERS sensor (Liang et al. 2017). For example, Fahimi–Kashani and co-workers utilized a simple hydrothermal method to synthesize MoS_2 QDs. They found that p-nitrophenol, the alkaline hydrolysis product of methyl parathion (MP), can induce the photoluminescence quenching of MoS_2 QDs, by which they developed a sensitive fluorescence method to detect MP. This method exhibited a detection limit of 0.085 μg/mL (Fahimi-Kashani et al. 2017).

Apart from the detection, TMDs can be utilized for the photocatalytic degradation of pesticides as well (Table S1). For instance, MoS_2 has a strong absorption in the visible region of solar spectrum and is possible to use visible light for photocatalytic reactions due to its relatively small bandgap (Han and Hu 2016). Furthermore, some favorable properties of MoS_2, such as atomic thickness, large distance between stacked layers and satisfying stability in both alkaline and acidic media, let MoS_2 be a promising photocatalyst (Anjum et al. 2018; Susarla et al. 2018). Up to now, many approaches, such as synthesizing multiphasic MoS_2 heterostructure (Chen et al. 2019) or MoS_2 microsphere (Huang et al. 2018), incorporating MoS_2 with other materials (Jo et al. 2016a, b; Kumar et al. 2016; Long et al. 2016; Luo et al. 2018; Ahamad et al. 2019; He et al. 2019), have been developed to prepare MoS_2-based hetrostructures for the photocatalytic degradation of pesticides. For instance, Huang et al. used sodium molybdate and L-cysteine as raw materials to prepared MoS_2 microsphere by a hydrothermal method (Fig. S2d). Under visible light irradiation, without adding H_2O_2, thiocarbon can be successfully degraded in MoS_2 microsphere suspension. Its degradation efficiency can reach 95% within 12 h (Fig. S2e) (Huang et al. 2018). Besides, MoS_2 can also degrade the pesticides via the electrocatalytic oxygen-reduction reaction (Qu et al. 2018), or serve as an efficient support for other materials and enrich the pesticides nearby, leading to a higher removal rate (Lu et al. 2017).

g-C_3N_4

Graphite-like carbon nitride (g-C_3N_4) is a kind of organic and metal-free semiconductors, which consists of tri-*s*-triazines and has a well crystallized layered structure (Liu et al. 2015). Based on its unique electrocatalytic and optical properties, it has been used alone or coupled with other materials to detect pesticides (Wang et al. 2016a, 2016b, 2017; Xie et al. 2018; Ouyang et al. 2018; Cao et al. 2019; Shetti et al. 2019; Yin et al. 2019) (Table S2). For instance, using g-C_3N_4 as the fluorescent probe and AuNPs as the colorimetric probe, Xie et al. developed a dual-signaling detection approach for sensing organophosphorus pesticides (Fig. S3a).The method displayed a wide linear range (2.0×10^{-11}–6.0×10^{-9} M) with a detection limit of 6.9×10^{-12} M (Fig. S3b, c) (Xie et al. 2018). Ouyang reported that g-C_3N_4@$BiFeO_3$ nanocomposites with photocatalytic activity can be used as a single peroxidase-like catalyst to fabricate the colorimetric/chemiluminescent dual-readout immunochromatographic assay for detecting multiple pesticide residues (Ouyang et al. 2018).

g-C_3N_4 with narrow bandgap energy (2.7 eV) can utilize visible light efficiently (Wang et al. 2009). Because of this, g-C_3N_4 is used for not only detection but also photodegradation of pesticides (Desipio et al. 2018; Pang et al. 2019). Yet, pure g-C_3N_4 has some disadvantages, such as low surface area and high recombination rate of photoelectron-hole pair (Jiang et al. 2016; Muhmood et al. 2017; Zhao et al. 2017). Therefore, many researchers prepared numerous g-C_3N_4-based composites by various methods, such as doping metal/non-metal elements (Kesarla et al. 2019; Vigneshwaran et al. 2019), combining with carbon materials (Chu et al. 2019; Dikdim et al. 2019) or semiconductors (Kumar et al. 2018; Abazari et al. 2019; Ayodhya and Veerabhadram 2019; Balasubramanian et al. 2019; Humayun et al. 2019; Yasmeen et al. 2019). For example, Kumar et al. fabricated the biochar supported ternary g-C_3N_4@$Bi_2O_2CO_3$@$CoFe_2O_4$ composites (BCBF), which was magnetically recoverable and showed a high catalytic activity under visible light. After visible radiation for 90 min, 99.3% of paraquat (PQT) was degraded by BCBF (Fig. S3d, e) (Kumar et al. 2018).

Vigneshwaran et al. prepared a novel catalyst based on the g-C_3N_4/chitosan composites. Owing to their efficient separation of electron hole pairs, these composites showed excellent photodegradation abilities towards chlorpyrifos (Vigneshwaran et al. 2019). Humayun et al. coupled cerium oxide (CeO_2) with g-C_3N_4 to form CeO_2/g-C_3N_4 (CeO/CN) composites with suitable band alignments. Under the visible-light irradiation, 57% of 2,4-dichlorophenol (2,4-DCP) was degraded by the CeO/CN composites after 2 h. The authors further proposed the photodegradation mechanism of 2,4-DCP based on the liquid chromatography tandem mass spectrometry analysis (Fig. S4a, b) (Humayun et al. 2019).

g-C_3N_4 has excellent stability and high reactivity due to its special layered structure. These properties make it possible to serve as a skeleton material to improve the adsorption capacities of other materials (Liu et al. 2015; Ou et al. 2018). For instance, Ou et al. prepared g-C_3N_4/amine-rich porous organic polymer composites (g-C_3N_4/RAPOP) via one-pot polymerization, which was then used as an adsorbent towards 2,4-DCP. When 2,4-DCP got close to the surface of g-C_3N_4/RAPOP, the hydrogen bond interactions and π-π interactions caused the adsorption of 2,4-DCP (pH 2–7). Within 40 s, the adsorption equilibrium of g-C_3N_4/RAPOP was reached. The maximum adsorption amount was 270.27 mg/g. In alkaline environment, both 2,4-DCP and g-C_3N_4/RAPOP were negatively charged and 2,4-DCP was desorbed from g-C_3N_4/RAPOP because of the strong electrostatic repulsion. The authors conducted the adsorption–desorption cycles for five times, and they believed that g-C_3N_4/RAPOP could be effectively regenerated via the alkali treatment (Fig. S4c, d) (Ou et al. 2018).

LDHs

Layered double hydroxides (LDHs) are a kind of inorganic layered materials, whose generic formula is $[M^{II}_{1-x}M^{III}_{x}(OH)_2]^{z+}(A^{n-})_{z/n}\cdot yH_2O$. M^{II} and M^{III} are divalent and trivalent metal ions, respectively. A^{n-} is interlayer anions in the brucite-like layers (Wu et al. 2013).

LDHs usually have a large surface area and the interlayer spacing of LDHs can vary, which depends on the size and geometry of the interlayer anions. In the electrochemical detection of pesticides, LDHs have been used as host materials to construct new functional host–guest materials. For example, Gong et al. built a series of electrochemical sensors using LDHs and some guest materials (including AChE (Gong et al. 2013), graphene (Liang et al. 2012), Ni/Al (Gong et al. 2009), NanoPt (Gong et al. 2010)) (Table S3).

Due to their high anion exchange capacities, high specific surface areas and flexible interlayer space, a variety of LDHs have long been used to fabricate efficient adsorbents for removing negatively charged contaminants in water such as anionic pesticides (Cornejo et al. 2008) and anionic dyes (Morimoto et al. 2011). Until now, many pesticides, such as 4-chloro-2-methylphenoxyacetic acid and 3,6-dichloro-2-methoxy benzoic acid can be effectively adsorbed by LDHs (Inacio et al. 2001; You et al. 2002; Legrouri et al. 2005; Li et al. 2005; Chaara et al. 2010, 2011; Otero et al. 2012; Nejati et al. 2013; Pavlovic et al. 2013). For example, Otero et al. investigated the adsorption behaviors of *S*-Metolachlor on the LDHs, which were intercalated with dodecylsulfate (HT-DDS) and tetradecanedioate (HT-TDD), respectively. They found that HT-TDD adsorbed more *S*-Metolachlor compared with HT-DDS. The amount of the adsorbed S-Metolachlor increased with temperature (Otero et al. 2012). Pavlovic et al. prepared the Mg–Al layered double hydroxide with caprylate (LDH-Cap) and studied its adsorption performances towards three pesticides, linuron, 2,4-DB and metamitron. They found that ~90% of initial linuron and 2,4-DB were absorbed by LDH-Cap within the first 30 min. However, metamitron was absorbed in a more gradual manner. The adsorbed 2,4-DB and metamitron were probably intercalated between caprylate chains and the brucite layers. Linuron may be adsorbed on the external surface of LDH-Cap. (Pavlovic et al. 2013).

In addition, LDHs also can be used as photocatalyst (Nguyen Thi Kim et al. 2016) or microbial immobilization matrix (Alekseeva et al. 2011) to mineralize pesticides via photodegradation or biodegradation. For instance, Phuong et al. prepared the calcined and Ti-doped LDHs (Mg@Fe@Ti@LDH) by the co-precipitation method. They found that Mg@Fe@Ti@LDH exhibited perfect photo-Fenton-like activity, causing 80–95% degradation of 2,4,5-trichlorophenoxyacetic acid in 360 min (Nguyen Thi Kim et al. 2016).

Hexagonal Boron Nitride and MXene

Hexagonal boron nitride (h-BN) has graphene-like layered crystal structures. It contains equal numbers of boron and nitrogen atoms, which are arranged in a hexagonal structure. In each layer, the boron and nitrogen atoms are bonded by covalent bonds, whereas these layers stack together by the van der Waals force. A single-layer h-BN nanosheet can be regarded as a graphene analogue and thus it is also known as "white graphene".

Owing to its unique properties like high mechanical strength and thermal conductivity, large surface area and high temperature stability, h-BN has been combined with other materials like metal nanoparticles to form nanocomposites for sensing pesticides (Atar and Yola 2018; Kiran et al. 2019; Tan et al. 2019; Zhan et al. 2019; Zhang et al. 2019). For instance, Atar and co-workers built an electrochemical sensor based on Fe@AuNPs@h-BN nanocomposites for the determination of cypermethrin in waste water samples. This electrochemical sensor exhibited a linear range of 1.0×10^{-13}–1.0×10^{-8} M and a detection limit of 3.0×10^{-14} M (Atar and Yola 2018).

As another popular member in pg-2DMs, transition metal carbides and nitrides nanosheets (MXene) has garnered increasing attentions in various fields (Naguib et al. 2014). It can be prepared using the HF-based chemical method to selectively etch the A element in its raw $M_{n+1}AX_n$ (MAX). M represents an early transition metal and A is mainly an element coming from group IIIA or IVA. X represents N or C element (Naguib et al. 2011). Due to its intriguing attributes like large specific surface area and excellent electrochemical activity, it has been utilized in sensing pesticides (Jiang et al. 2018; Xie et al. 2019; Zhao et al. 2020) (Table S3). Zhao et al. used MXene@Au@Pd nanocomposites to fabricate an enzyme-based pesticide biosensor. They chose paraoxon as the model pesticide and found the as-prepared biosensor showed a detection limit of 1.75 ng/L and a linearity range of 0.1–1000 µg/L (Fig. S5a) (Zhao et al. 2020). To detect malathion, Jiang et al. modified MXene with silver nanoparticles to prepare an AChE sensor (Fig. S5b), which showed a detection limit of 3.27×10^{-15} M and a linearity range of 1.0×10^{-14}–1.0×10^{-8} M. This biosensor utilized the unique synergistic effects and electrocatalytic properties between $Ti_3C_2T_x$ nanosheets and silver nanoparticles, which not only improved the electron transfer, but also enlarged the surface area for malathion detection (Jiang et al. 2018).

Conclusion and Perspective

The rise of global population generates a need to increase crop productivity and yield, which causes a large amount of pesticides to be produced and consumed annually. The released pesticides go through many transformations such as biodegradation, oxidation and photodegradation (Martinez Vidal et al. 2009). Photodegradation is an important abiotic process in the dissipation of pesticides, however, its efficiency is too low when it occurs naturally (Katagi 2004) To solve this problem, various nanomaterials have been used as photocatalysts to degrade pesticides in soil (Zeng et al. 2010) and water (Liu et al. 2016; Keihan et al. 2016) in the past decade. Recently, many pg-2DMs with unique properties has also been utilized in the treatment of pesticide. Although still in their infancies, the researches on the use of pg-2DMs have already demonstrated promising in, for example, the sensing, photodegradation and adsorption of pesticides. We believe more exciting results will be obtained in the future by investigating other newly emerged pg-2DMs with intriguing properties, such as black phosphorus and SnS_2. However, there are also some unsolved questions in the current research: (1) Many factors, such as ionic strength, pH and natural organic matters will influence the adsorption capacities of pg-2DMs based adsorbents toward pesticides. Yet, such effects are rarely investigated; (2) The products of pg-2DMs catalyzing decontamination of pesticides need to be explored; (3) Although some pg-2DMs, such as $g\text{-}C_3N_4$ (Vigneshwaran et al. 2019) and MoS_2 (Xu et al. 2013), already show high thermal and chemical stabilities in the photodegradation process, their stabilities still need to be confirmed at industrial scale; (4) Since increasing reports prove that graphene and other 2DMs exhibit toxicities to some extend (Tian et al. 2016; Li et al. 2018; Yu et al. 2019; Guiney et al. 2018), more efforts are needed to explore the environmental toxicities of these pg-2DMs when they are used for environmental remediation.

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