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Mini Review

Graphene-Based Composites for Electrochemical Sensor Fabrication and Their Application for Drug Detection

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Rapid detection of drugs is very important in clinical and food safety. Electrochemical detection technology is an analytical technology suitable for rapid detection in the field. However, the performance of commercial electrodes limits the application of electrochemical sensors. Graphene has excellent electrochemical performance, and its composite materials often show better performance. In this review, we summarize the electrochemical sensors fabricated from graphene composites for drug detection between 2010 and 2020. From the review, we found that graphene can greatly improve the performance of electrochemical sensors. The improvement of this performance greatly accelerates the practical application potential of electrochemical sensors in drug detection.

Keywords: Graphene; Electrochemistry; Drug detection; Sensor; Nanocomposite

1. INTRODUCTION

Graphene is a two-dimensional structure with single-layer sp^2 hybridized carbon atoms. It is a honeycomb structure film and the basic unit of graphite materials. It can be constructed into 0-dimensional fullerenes, rolled into one-dimensional carbon nanotubes, and stacked into three-dimensional graphite [1–4]. The extensive conjugation of graphene sheets can produce excellent thermal, mechanical and electronic properties and has become the most attractive field and has been widely studied. Graphene and its derivatives have excellent chemical and physical properties. The preparation of multifunctional composites based on graphene can be used in practical fields. To date, graphene and polymers, inorganic nanomaterials, organic crystals, metals, organic structures, biomaterials, carbon nanotubes and other materials have been successfully prepared. They are widely used in photovoltaics, fuel cells and other fields [5–10].

Sensors are the detection part of a tester system that has a direct relationship with the object to be measured, directly feel changes in the measured parameters, and turn the measured parameters into signals that are easy to transport, process and measure [11,12]. Electrochemical sensors are based on the electrochemical properties of the substance to be measured and change the chemical quantity of the substance to be measured into an electrical quantity for sensing detection [13,14]. The earliest electrochemical sensors can be traced back to the 1950s, when they were used for oxygen monitoring. In the mid-1980s, small electrochemical sensors began to be used to detect a variety of toxic gases in the PEL range and showed good sensitivity and selectivity [15–17]. Electrochemical sensors are classified by species: ion sensors include pH sensors, gas sensors, biosensors made of biological characteristics, etc. According to their principle, sensors can be classified depending on their electrochemical type, optical type, thermal type, mass type, etc. They can also be classified according to the amount of electricity converted: potential sensors, current (ampere or volt ampere) sensors and impedance (resistance type and capacitance type) sensors [18,19].

At present, various electrochemical sensors are widely used in many applications. With the further research and development of electrochemical sensors, research on electrochemical sensors will develop towards achieving high sensitivity, high stability, long service life, portable miniaturization and intellectualization.

1. Expand the research scope of electrochemical sensors and seek the combination of other technologies and electrochemical sensors;
2. Expand the application of electrochemical sensor technology in life medicine;
3. Screening of electrochemically active substances with high sensitivity and selectivity;
4. Looking for stable immobilization methods and exploring new detection principles;
5. Exploit the unique advantages of electrochemical sensors in drug analysis and drug screening.

Graphene has excellent electronic properties, and its application in electrode materials can promote the electron transfer of electroactive substances in solution [20–22]. Therefore, as an electrode material, graphene has better electrochemical behaviour than traditional carbon materials. Sensors based on graphene nanocomposites have been widely studied. Sensors based on graphene mainly include A. DNA and protein sensors; B. enzyme biosensors; C. electrochemical immunosensors; and D. electrochemical sensors based on the electrochemical catalysis of graphene materials for testing organic molecules, such as pollutants, drugs, and neurotransmitters. Sensors have also been used for the detection of metal ions, gases and other inorganic substrates [23,24]. In this review, we reviewed the electrochemical detection of drugs using graphene composites as electrode surface modifiers based on the literature published from 2010 to 2020.

2. GRAPHENE-METAL COMPOSITE BASED SENSOR

In the past few decades, much research has been done on the preparation of inorganic structural materials by controlling morphology, size, crystallinity and functionalization [25–28]. These materials are widely used in electronic, optical, electrochemical energy conversion and storage applications. To further enhance the properties of the materials, a large number of inorganic materials and graphene and

its derivatives were used to prepare composite materials containing Au, Ag, Pd, Pt, Ni, Cu, and metal oxides or hydroxides or metal alloys [29–35].

Er et al. [36] reported a fast, simple, high-sensitivity, high-selectivity electrochemical sensor based on a graphene nanosheet/gold nanoparticle/Nafion nanocomposite-modified electrode (GRP/AuNPs/NFN). GRP/AuNPs/NFN can be used for high-efficiency detection of siloxacillin. This work used adsorption and stripping differential pulse voltammetry to study the electrochemical oxidation behaviour of siloxillin. The research results prove that the electrode has a good electrochemical response to the electrochemical oxidation of siloxillin. Under the best detection limit conditions, the detection range is 10-330 nM. Therefore, the developed electrode can be conveniently used for the electrical analysis of siloxacillin in drugs or biological samples.

Ansari et al. [37] proposed an electrochemical sensor for the determination of ranolazine using a glassy carbon electrode made of WO_3-modified graphene nanocomposites. Ranolazine is an anti-angina pectoris drug that is widely used in patients with chronic angina pectoris. Because WO_3 nanoparticles and graphene nanosheets have high conductivity and a large effective surface area, the developed WO_3/graphene/GCE sensor can detect the oxidation of ranolazine at a low potential. Under optimal conditions, the sensor can respond within a concentration range of 0.2-1.4 μM and 1.4-14 μM. The lowest detection limit was calculated to be 0.13 μM. Experimental results show that this electrochemical sensor can be used for drug quality control, clinical analysis and pharmacokinetic research. Bagheri et al. [38] synthesized a Zn_2SnO_4-graphene nanocomposite, which was then used for the simultaneous determination of morphine and codeine. Compared with graphene and Zn_2SnO_4, the as-prepared Zn_2SnO_4-graphene nanocomposite has a significantly enhanced oxidation peak current during detection. The proposed electrochemical sensor can linearly detect morphine and codeine in the concentration range from -0.02 to 1.0 μM. The detection limits of morphine and codeine were 0.011 and 0.009 μM, respectively. Some interfering substances, such as Ca^{2+}, glucose, lactose, sucrose, ascorbic acid, acetaminophen, ethanol, and norspin, did not significantly interfere with the detection of analytes.

Afkhami et al. [39] proposed a simple preparation method for the synthesis of nanocomposites of graphene and $CoFe_2O_4$ nanoparticles. Using the synergistic effect of graphene and $CoFe_2O_4$ nanoparticles, they successfully prepared an ultrasensitive electrochemical sensor for acetaminophen and codeine detection. Under optimal conditions, the linear detection range of the electrochemical sensor for both paracetamol and codeine is 0.03 to 12.0 μM. The detection limits of acetaminophen and codeine were 0.025 μM and 0.011 μM, respectively. This method does not interfere with many common small molecules, such as glucose, ascorbic acid, caffeine, naproxen, and alanine.

Lee et al. [40] reported an iron oxide (Fe_2O_3)/graphene nanocomposite electrode used as an electrochemical sensor for the determination of trace amounts of Zn^{2+}, Cd^{2+} and Pb^{2+}. The study used differential pulse anodic stripping voltammetry to determine metal ions. Due to the synergistic effect of graphene and nano-Fe_2O_3, the modified electrode exhibits electrochemical catalytic activity, with high sensitivity to trace heavy metal ions. Under optimal conditions, the sensor can detect Zn^{2+}, Cd^{2+} and Pb^{2+} in the range of 1-100 μg/L. The detection limits of Zn^{2+}, Cd^{2+} and Pb^{2+} are 0.11 μg/L, 0.08 μg/L and 0.07 μg/L, respectively. The assembly of the sensor uses a solvent-free thermal decomposition method, which can be applied to the simple synthesis of nanocomposite electrode materials.

Table 1 summarizes recently developed graphene-metal composited based electrochemical sensors for drug determination.

Table 1. Recent developed graphene-metal composited based electrochemical sensors for drug determination.

Materials	Method	Target	Reference
CeM/GO	DPV	Chloramphenicol	[41]
β-cyclodextrin, Au NPs and GO	DPV	Nilutamide	[42]
GO/$CaTiO_3$	I-T	Chemotherapeutic drugs	[43]
Pd–Ag/reduced graphene oxide	DPV	Acetaminophen	[44]
Fe_3O_4 NPs-GO	CV	Rutin	[45]
Pd/rGO	CV	Acetaminophen and 4-aminophenol	[46]
$NiFe_2O_4$/graphene	DPV	Tramadol and acetaminophen	[47]
GO-Fe/ZnO	DPV	Chlorpromazine	[48]
Ni-CoS/GQDs	DPV	Tenofovir disoproxil fumarate and lamivudine	[49]
$CeBiO_x$/rGO	DPV	Acetaminophen	[50]
NiO–CuO/graphene	CV	Dopamine, acetaminophen and tryptophan	[51]
CuO–graphene	CV	Acetaminophen, caffeine and ascorbic acid	[52]
Graphene-ZnO	CV	Acetaminophen and phenacetin	[53]
Fe_3O_4-graphene oxide-gold nanoparticle	CV	Catechol and hydroquinone	[54]
Graphene-MoS_2	CV	Eugenol	[55]
Au-Pd/RGO	CV	Ascorbic acid, acetaminophen and tyrosine	[56]
RGO/$CuFe_2O_4$	CV	Hydrogen peroxide	[57]
Cu_2O/Graphene	DPV	Dopamine	[58]
MoS_2–graphene	DPV	Acetaminophen	[59]
SnO_2-rGO	CV	Hydrogen peroxide	[60]
Gr/CuPc/PANI	CV	Ascorbic acid	[61]

EDDPT/GO	CV	Epinephrine, acetaminophen and dopamine	[62]
Graphene/$CuCo_2O_4$	DPV	Dopamine, melatonin and tryptophan	[63]
Nafion/TiO_2–graphene	DPV	Paracetamol	[64]
CoHCFNPs/graphene	I-T	Hydrogen peroxide	[65]
TiO_2@CuO-N-rGO	DPV	Flunitrazepam	[66]
NiO/graphene	DPV	Ascorbic acid	[67]
Ag/Au-graphene	CV	Carcinoembryonic antigen	[68]

3. GRAPHENE-CARBON MATERIAL-BASED SENSORS

Due to the different morphologies of carbon materials, the combination of 1D graphene and other carbon materials can achieve better performance than graphene. Carbon nanotubes, for example, can form networks [69–72]. The addition of graphene increases the permeability of molecules on the electrode surface. Moreover, adding carbon spheres between graphene can avoid layer-by-layer stacking. Therefore, composite materials assembled with different carbon materials can improve the performance of the electrode and improve the detection limit and sensitivity of drug detection [73–83].

Mohamed et al. [84] reported the use of GO, multiwalled carbon nanotubes (MWCNTs) and pyrogallol (PG) to prepare a high-sensitivity sensor for the detection of omeprazole. The results show that, compared with bare carbon paste electrodes, modified electrodes have a stronger current response, which can significantly improve the oxidation activity of omeprazole. Under optimal conditions, the electrochemical sensor can detect concentrations from 0.2 nM to 6 μM, and the detection limit is calculated to be 10.2 pM. The average recovery rates of this electrochemical sensor in practical preparations and human serum samples are 100.97% and 98.58%, respectively.

Mohamed et al. [85] reported a GO-MWCNT nanocomposite electrochemical sensor for the determination of tramadol hydrochloride. The electrochemical sensor assembled using GO-MWCNTs can linearly detect tramadolamine hydrochloride at concentrations from 2 nM to 1.1 mM. The detection limit and quantification limit were 0.5 nM and 0.499 nM, respectively. At the same time, this sensor can measure tramadol hydrochloride in the presence of ketorolac tromethamine and acetaminophen. This sensor has been successfully applied to the determination of tramadol hydrochloride in plasma. The recovery rate of tramadolamine hydrochloride in the sample is also very satisfactory, which proves that this sensor can be used for clinical analysis and quality control of drugs in pharmaceutical preparations.

Zhai et al. [86] synthesized water-dispersed sulfonated graphene sheets and oxygen-functionalized multiwalled carbon nanotube nanocomposites and then assembled a new type of electrochemical sensor to measure clenbuterol. There is a π-π stacking interaction between graphene sheets and oxygen-functionalized multiwalled carbon nanotubes. Due to the synergistic effect of

modified nanomaterials, the electrochemical sensor has shown very excellent performance. Using differential pulse voltammetry, the sensor can linearly detect clenbuterol hydrochloride at concentrations of 0.01-5.0 μM. Furthermore, this sensor successfully measured clenbuterol in liver samples.

Table 2 summarizes recently developed graphene-carbon composited based electrochemical sensors for drug determination.

Table 2. Recent developed graphene-carbon composited based electrochemical sensors for drug determination.

Materials	Method	Target	Reference
Graphene- oxygen-functionalized multi-walled carbon nanotube	CV	Clenbuterol	[86]
GO-MWCNTs	DPV	Tramadolamine hydrochloride	[85]
Vertically aligned carbon nanotubes and GO	CV	Rosuvastatin	[87]
RGO and carbon black	CV	Dopamine and paracetamol	[88]
MWCNT/RGO/Au	LSV	Norfloxacin	[89]

4. MOLECULARLY IMPRINTED AND POLYMER-BASED ELECTROCHEMICAL SENSORS

Molecularly imprinted technology (MIT) is a bionic recognition system used in polymer chemistry, material chemistry, biochemistry and other disciplines [90–92]. MIT has achieved rapid development in recent years because it can specifically recognize a certain target molecule in a system, and its recognition conditions are not harsh; thus, its application fields are relatively wide and include medicine, separation and purification, bionic catalysis, and electrochemical sensors [93–95]. A MIP is formed by template molecules and suitable monomers through a polymerization process, and then, the template molecules are washed off by certain methods to form multiple recognition sites corresponding to the spatial structure of template molecules so that the template molecules can be specifically recognized [96,97]. MIP-based electrochemical sensors combine molecularly imprinted polymers with electrochemical sensors to realize selective detection of target molecules. An MIP is used as the identification component, which is fixed on the surface of the signal converter by certain means. In the process of using MIPs to identify target molecules, a signal converter is used to generate electrical signals for detection.

Electrochemical sensors based on graphene and molecularly imprinted technology combine the advantages of both, which greatly improves the sensitivity and selectivity of the sensor. Graphene quantum dots (QDs) are graphene nanosheets with a size less than 100 nm. Graphene quantum dots not

only have the advantages of the fast electron transfer rate of graphene but also solve the problem of dispersion in water caused by the strong π - π stacking effect and van der Waals force. In addition, graphene QDs have good biocompatibility. Therefore, graphene quantum dots have been widely used in molecularly imprinted electrochemical sensing. Rao et al. [98] prepared a novel molecularly imprinted sensor for the detection of bisphenol S (BPS). This sensor is made of hollow nickel nanospheres wrapped with graphene quantum dots. The material increases the active site area on the electrode and improves the electrode conductivity. The modified glassy carbon electrode was inserted into a solution containing the functional monomer pyrrole and template molecule BPS. The sensor with a molecularly imprinted polymer film was prepared by cyclic voltammetry. The preparation process is shown in Figure 1. Various experiments have proven that the sensor not only has high sensitivity, selectivity, reproducibility and stability but also has a wide detection range and can detect BPS in different plastic samples.

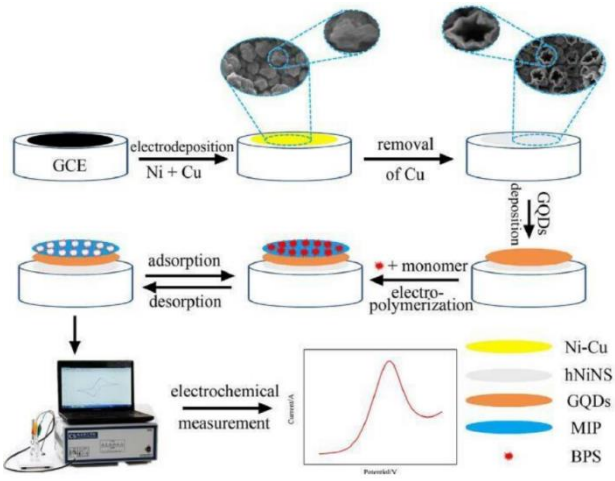

Figure 1. Preparation of BPS MIP electrochemical sensor modified by hollow nickel nanospheres coated with graphene quantum dots. Reprinted with permission from reference [98].

Li et al. [99] successfully prepared a magnetic graphene oxide molecularly imprinted electrochemical sensor functionalized with gold nanoparticles and used it to detect dibutyl phthalate. MgO was prepared by in situ chemical coprecipitation of Fe^{3+} and Fe^{2+} in an alkaline GO solution. Gold nanoparticles were prepared by reduction of trisodium citrate. The recombination between gold nanoparticles and MgO is realized by S-Au bonds. Magnetic GO functionalized with gold nanoparticles was copolymerized with the functional monomers methacrylic acid and dibutyl phthalate, the crosslinking agent glycol dimethacrylate and the initiator azodiisobutyronitrile to obtain a molecularly imprinted polymer. An MIP-based electrochemical sensor for the sensitive and selective detection of dibutyl phthalate was obtained by coating the polymer droplet on the gold electrode. The schematic diagram is shown in Figure 2.

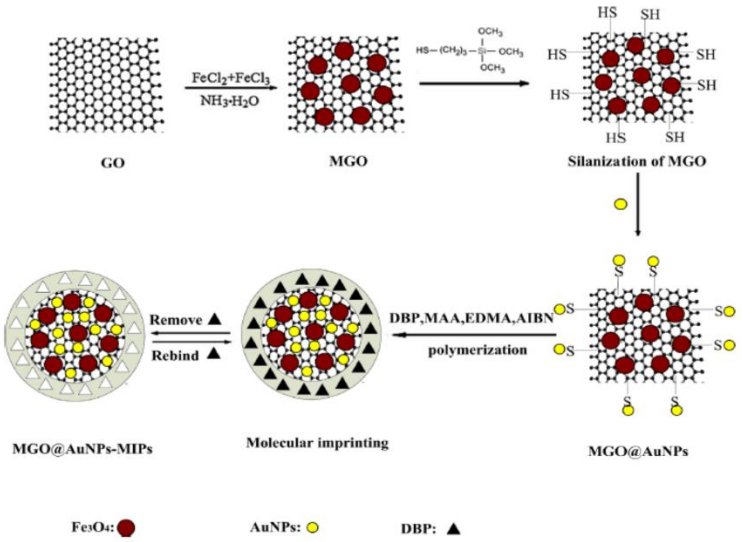

Figure 2. Preparation of magnetic graphene oxide MIP electrochemical sensor functionalized with gold nanoparticles. Reprinted with permission from reference [99].

Mostafavi et al. [100] synthesized a diclofenac MIP-based electrochemical sensor using polyaniline, reduced graphene oxide (rGO) and triphenylamine as crosslinking agents. They used CV to study the electrochemical behaviour of diclofenac and optimized the parameters that affect the determination of diclofenac. The cyclic voltammogram shows an obvious oxidation current peak at 0.5 V, which can be used for detection. Under the optimal conditions, the MIP-based electrochemical sensor is proposed to detect diclofenac linearly in the range of 5–80 mg/L. The corresponding detection limit was calculated to be 1.1 mg/L. The relative standard deviation of diclofenac analysis using this method was 2.43%.

Table 3 summarizes recently developed graphene MIP-based electrochemical sensors for drug determination.

Table 3. Recent developed graphene MIP electrochemical sensors for drug determination.

Materials	Method	Target	Reference
N-acryloyl-4-aminobenzamide-GQDs	DPV	Ifosfamide	[101]
poly(para-aminobenzoic acid) on 3D Pd nanoparticles-porous graphene-carbon nanotubes	DPV	Quercetin	[102]
Ionic liquid–graphene	CV	Methyl parathion	[103]
Chitosan–graphene	CV	Bisphenol A	[104]

Graphene- 4-amino-3-hydroxy-1-naphthalenesulfonic acid	CV	Melatonin	[105]
Polypyrrole–Graphene–Au NPs	CV	Levofloxacin	[106]
GQDs-metronidazole	CV	Metronidazole	[107]
Au NWs/graphene oxide	DPV	Cefixime	[108]

5. CONCLUSIONS AND OUTLOOKS

Sensor platforms based on graphene materials have been widely used in various analytical fields and have shown momentum in their rapid development. They have become a research hotspot in sensing analysis, electrocatalysis and functional materials. The intrinsic properties of graphene materials with different properties and the functional design and construction of composite materials can enhance the sensitivity of target analytes from the aspects of linear range, sensitivity and detection limit and improve the practicability of the electroanalytical sensing platform from the perspective of stability and selectivity.

However, from the existing research results, there are still some limitations of the graphene sensing interface. For example, precise control between the structure, composition and size of graphene materials and their electrocatalytic properties has not been fully realized. Although the electroreduction of graphene on the electrode surface can be controlled at a macroscale, the microstacking mode, the stability of interface bonding and the artificial control and design of microstructures are affected by many factors, so the goal of full control cannot be achieved. Due to the limitation of reduction conditions, it is still difficult to prepare composites with multidimensional complex structures, such as rich networks and porous multichannel structures. Due to the limitation of preparation conditions, it is difficult to realize the structural diversification of multicomponent composites, such as the doping of heteroatoms.

In view of the above shortcomings, graphene composite electrochemical sensing platforms still need to be explored and studied from theoretical and experimental perspectives:

(1) In order to improve the conductivity, biocompatibility and electrochemical performance of graphene, the surface of graphene was doped and modified based on the existing chemical modification methods of graphene surface and interface in order to optimize the electronic structure of the material interface.

(2) In order to solve the problems such as curl, agglomeration, interlayer stacking and difficult dispersion of graphene materials, inorganic nano particles, polymers, proteins, biological small molecules and even cells are explored. Then, we will build a novel electrochemical biosensor platform and develop high-precision, high sensitivity and label-free electrochemical biosensors.

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