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Review article

Recent advances in the synthesis of ZnO-based electrochemical sensors

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ABSTRACT

Until now, various composites based on zinc oxide (ZnO) have been investigated in electrochemical sensors. The physical and electrochemical properties of ZnO and its structure can improve the selectivity, sensitivity, and adaptability of nanocomposites. Therefore, the focus on the fabrication of cheap ZnO-based electrodes with affordable and easy transportability has increased. In addition, the electrochemical behavior is affected by the structure and morphology of the ZnO-based composite in detecting pollutants such as volatile organic compounds, heavy metals, and toxins. Furthermore, ZnO-based nanostructures are efficient in the fabrication of electrochemical sensors in the food industry, pharmaceutical analysis, and medical diagnostics. In this review, various techniques in the synthesis of ZnO-based electrodes and their effect on the particle size, shape, and morphology of compounds have been collected. Since the performance of chemical sensors has a direct relationship with the structure of the composite used in its electrode, it is necessary to discuss the new production methods, new concepts, strategies, and challenges. Additionally, new gains highlight recent developments and sensing of various analytes in the monitoring systems. These sensors have demonstrated a strong growth acceleration which could lead to the development of recent technologies. At last, an optimistic outlook is provided on the future of ZnO-based sensors and their challenges.

KEYWORDS

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1. Introduction

Rapid population growth and consumption of fossil fuels have caused an increase in environmental pollution and the release of dangerous pollutants [1]. The toxicity of some pollutants is such that it endangers human health and the safety of the environment and has become an important issue of global concern [2]. In addition, industrialization and urbanization have accelerated the entry of dangerous volatile organic compounds, heavy metals, pharmaceutical derivatives, pesticides, and toxic gases into the atmosphere and flowing and underground waters [3]. Therefore, identifying and removing harmful combinations as well as environmental pollutants is of great importance. For this purpose, the development of versatile tools with favorably responsive, low-cost, and user-friendly systems for the quantification of pollutants at very low concentrations is essential [4].

Chemical sensors are sensory receptors that detect specific chemical stimuli such as changes in concentration or composition and the existence of a specific element in the environment [5]. Chemical sensors include a sensing layer that produces an electrical signal as a result of the interaction of the chemical species or analyte with this layer, which is finally amplified and processed. The main part of a chemical sensor is the sensing material that is in contact with a detector (transducer) and is responsible for identifying and linking with the target species in a complex sample. The detector converts the chemical signals produced as a result of the binding of the sensor element with the desired species into a measurable output signal. Chemical sensors are divided into several categories based on the type of converter or conversion of chemical change into a processable signal: optical sensors, mass sensors, thermal sensors that are sensitive to temperature changes in the environment, and electrochemical sensors that their operation is based on the interaction between chemistry and electricity [6]. Since electrochemical sensors have few detection limits and reporting signals can be diverse (current, voltage, etc.) with no generation of waste, electrochemical sensing is highly popular [7].

In general, electrochemical sensors operate through the redox reaction of the target species on the electrode surface and produce an electrical signal proportional to the concentration of the analyte species [8]. An electrochemical sensor consists of a sensor electrode (working electrode) and a counter electrode, which are separated by a thin layer of electrolyte [9]. The transducer element in electrochemical sensors is the same electrode that is responsible for detecting electrochemical reactions. After entering the sensor through a capillary inlet, the desired species passes through a water-repellent barrier and finally reaches the surface of the electrode [10]. An ideal sensor should have the following characteristics: i) the output signal should be proportional to the type and amount of the target species; ii) to act very specific to the target species; iii) to have high resolution and selectivity; iv) having high repeatability and accuracy; v) to have a high response speed (in milliseconds); vi) failure to respond to disturbing environmental factors such as temperature, ionic strength of the environment, vii) linearity etc. [11].

In electrochemical sensors, the reaction occurs on the electrode's surface, and due to chemical interaction, the electrode is oxidized or reduces the analyte. The electrodes of electrochemical sensors should be sensitive, selective, reversible, short-term responsible, and stable [12]. Sensory materials have a wide range including carbon-based materials, porous nanomaterials, metal oxides, and polymers [13]. Currently, sensors based on metal oxides have been developed on commercial sensors for various applications [14]. ZnO is one of the most popular semiconductors due to its non-toxicity, low cost, good stability, and environmentally friendly features. In addition, ZnO as an abundant metal oxide has shown great potential in electrode materials because of its specific surface area, small particle size, high electron mobility, tunable band structure, and beneficial oxidation capacity. Herein, we present a thorough review of ZnO-based sensors to guide the development of these composite synthesis approaches. These methods include elemental doping, coupling of metal and non-metal oxides, hybridization of metal sulfide, and all kinds of MXenes. In addition, different morphologies resulting from different synthesis techniques have been pointed out. At last, the challenges and outlooks of ZnO-based nanocomposites in developing electrochemical sensors are summarized.

2. Synthesis of ZnO-based composites

Synthesis methods of ZnO-based nanostructures are divided into physical and chemical categories. Typical techniques include sol-gel, hydrothermal, precipitation, solid state, microemulsion, thermal decomposition, and vapor solid-liquid.

2.1. Precipitation method

Precipitation synthesis is a low-cost convenient approach in the fabrication of nanoparticles [15]. In a case study, a ZnO/carbon/glassy carbon electrode has been prepared via the precipitation method by $Zn(CH_3COO)_2 \cdot 2H_2O$ and NH_3 during 1 h stirring to complete the reactions [16]. The FESEM image of the composite showed a spread of tightly packed hexagonal nanorods of ZnO/glassy carbon. Based on the FESEM pictures, the carbonaceous layers have dispersed on the rough surfaces of the zinc oxide. In comparison with bare ZnO nanorods, there was more noise in the XRD shape of the nanocomposite which is attributed to the amorphous carbon [17]. Cyclic voltammetry analysis exhibited the reversible oxidation-reduction behavior of the ferricyanide anions at pH>7. The high surface area of the nanoscale ZnO caused enhancement in the peak currents after the modification with glassy carbon. Glassy carbon bonded to carbon nanolayers led to revved electron transfer and higher ZnO/C ratio [18]. The electrochemical impedance spectroscopy (EIS) curve indicated that the ZnO/carbon/glassy carbon electrode has been successfully constructed through the precipitation method. In general, the incorporated carbon nanolayers in ZnO are reliable and accurate terbutaline sensors in the blood serum samples of athletes with proper sensitivity and selectivity. In another research, Dhinasekaran et al. [19] utilized the co-precipitation method for the synthesis of CuO/ZnO and Fe_2O_3/ZnO for urea detection via co-precipitation of iron chloride and copper chloride anhydrous with zinc acetate dihydrate. Adding iron and copper to zinc oxide changes its crystal structure and makes it more sensitive to urea. Morphological analysis of CuO/ZnO revealed spherical nanoparticles on platelet and agglomerated sheets. In the TEM image of Fe_2O_3/ZnO samples, sphere particles with agglomeration existed because of the substitution of iron cations in the zinc sites. By comparing the TEM images, it has been determined that the crystal structure of zinc oxide has been changed by adding copper and iron oxide by co-precipitation route. In both composites of CuO/ZnO [20] and Fe_2O_3/ZnO [21], the bandgap has been decreased compared with bare zinc oxide. In a recent study by Rahman et al. [22], CuO/ZnO/Nafion nanomaterials were prepared by applying the co-precipitation method in an alkaline solution. The FESEM image of the composite showed an irregularly flower-shaped structure without any additional impurity in the EDS analysis. This compound modifies the citric acid sensor by the wet chemical method. Previously, it was predicted that the modified CuO/ZnO/glassy carbon electrode is capable of the detection of a target analyte [23]. Recently, the co-precipitation method has been applied to the construction of Co_3O_4/ZnO nanoparticles for the detection of hydroquinone and resorcinol [24]. These two toxic and environmentally polluting solvents are used in cosmetics, dyes, rubber, and petrochemical industries and enter industrial effluents [25]. Their simultaneous detection is difficult due to their same properties and structural similarity. Hence, the Co_3O_4/ZnO composites have been employed to modify carbon paste electrodes to simultaneously sense hydroquinone and resorcinol electrochemically. Sodium carbonate, cobaltous chloride, sodium hydroxide, and zinc acetate were utilized as precursors. After the production of Co_3O_4 and ZnO by the co-precipitation method, both metal oxides have been ground in a mortar. Finally, Co_3O_4/ZnO nanoparticles and carbon paste electrodes have been mixed and kneaded on the paper. Nanoparticle structures of Co_3O_4/ZnO were seen clearly in the SEM images. The cyclic voltammetry of the modified electrode has exhibited an increase in the redox peak current compared to the bare carbon paste electrode [26], which is related to the large surface area of the novel synthesized electrode calculated by Randles-Sevick's equation [27].

2.2. Hydro/solvothermal method

The hydro/solvothermal method is one of the famous and widely used techniques in the synthesis of nanomaterials [28]. Ganesamurthi et al. [29] have applied the hydrothermal process to the formation of NiO/ZnO composite by nickel acetate, zinc acetate, glycine, and sodium sulfate in distilled water. The reactions were completed in a high-pressure autoclave at 180 °C after 2 h. After filtration and washing, the output stream from the reactor was annealed at 500 °C. FESEM picture of the obtained powder displayed the microstructure of the NiO/ZnO sample. The electrochemical impedance spectroscopy (EIS) analysis could help to understand the interaction of the electrolyte and electrode in KCl/$[Fe(CN)_6]^{3-,4-}$ solution. According to the comparison of the charge transfer resistance of the pristine and modified electrodes, it was suggested that NiO/ZnO could be a favorable binary metal oxide for electrochemical Rutin sensing. It should be noted that previously, hydrothermally assembled NiO/ZnO microspheres have been employed for the efficient detection of SO_2 [30]. Hydrothermal synthesis is based on the solubility of precursors in the aqua environment [31]. For example, chloroauric acid ($HAuCl_4.3H_2O$) and zinc acetate have been solved in deionized water to construct a uniform solution and conducted in the hydrothermal reaction medium [32]. The colloidal product (Au/ZnO) has been dried and then used as a glassy carbon modifier for biomolecule sensing of gallic acid or trihydroxybenzoic acid with the formula $C_6H_2(OH)_3CO_2H$ as a phenolic compound. Gold-decorated zinc oxide has shown high electro-catalytic properties via sensitivity and detection limit. In the composite fabrication designed by Yang et al. [33], a one-pot hydrothermal technique is adopted to prepare ZnO-Au/reduced graphene oxide (rGO) nanocomposite through in situ growth of Au nanoparticles to increase the weak conductivity of zinc oxide (Fig. 1). By controlling the conditions including temperature, pressure, and pH of the environment, the morphology of the desired product can be achieved [34, 35]. Donut-shaped ZnO has been coated by Au and graphene nanoparticles which created effective conductive bridges [36]. In another work, binder-free ZnO nanoparticles on carbon cloth have been prepared through a facile hydrothermal method to apply in the hydroxychloroquine sensors [37]. Hydroxychloroquine, a usual drug, has been suggested for the treatment of chronic inflammatory diseases such as COVID-19 [38]. According to Madhu and colleagues' literature [39], Kokulnathan et al. [37] have transferred zinc nitrate hexahydrate and hexamethylenetetramine mixture into a sealed reactor

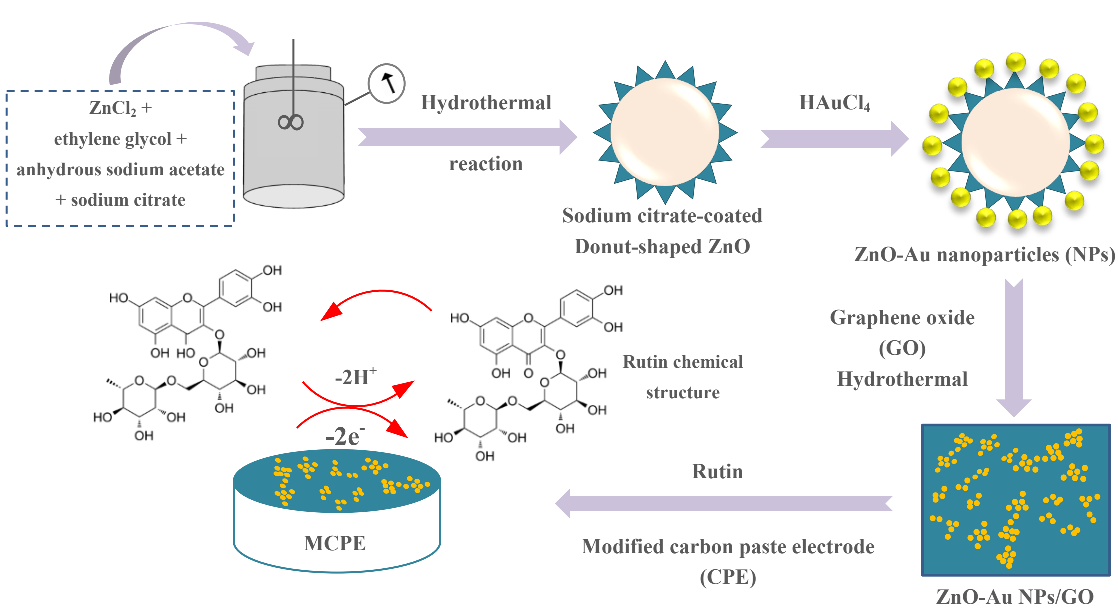

Fig. 1. Schematic route for the preparation of ZnO-Au/reduced graphene oxide composite and the mechanism of rutin sensing.

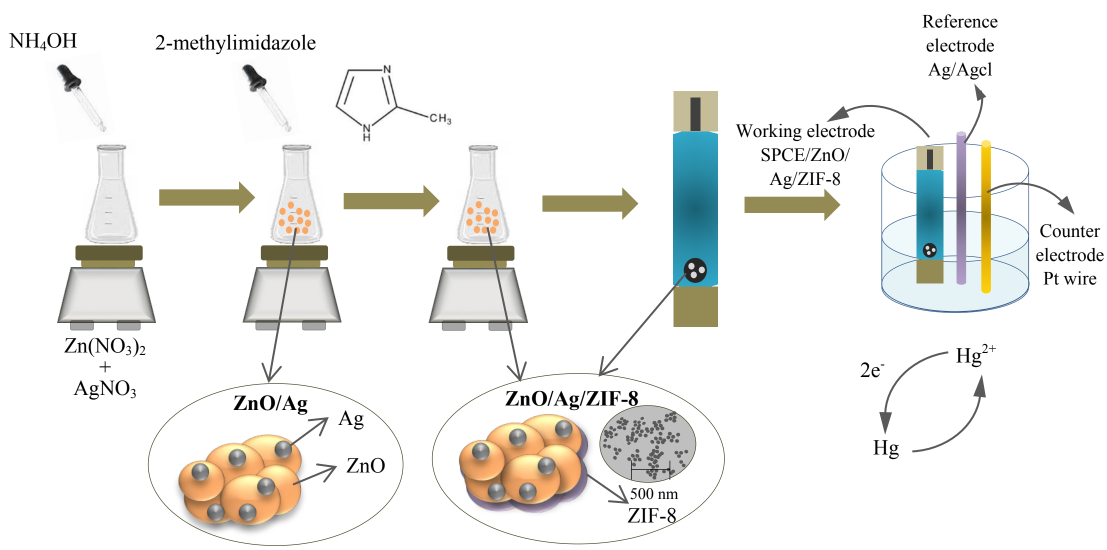

Fig. 2. Schematic illustration of the preparation of Ag/ZnO/ZIF-8 composite for Hg^{+2} detecting.

followed by immersing pretreated carbon cloth in the growth solution. The morphological feature of the final product named ZnO/CC has been analyzed by SEM image. The nanograins of zinc oxide have anchored on the carbon cloth and its surface was covered by the carbon ions of sp^2 hybridization, causing easy absorption of zinc ions for crystallization of zinc oxide [40]. The as-prepared composite has displayed a decreased charge-transfer resistance, increased electrochemical surface area, and excellent sensitivity. The acidity of the electrolyte is an important factor in the ZnO/CC action for the hydroxychloroquine sensing.

2.3. Sol-gel method

The sol-gel method is considered a wet chemical process that is used in the preparation of nanomaterials, particularly metal oxides [41]. In a case study, Arabbani et al. have synthesized Ag/ZnO/ZIF-8 through a simple sol-gel technique to detect mercury [42]. They have taken various routes to obtain the best fabrication method. In 1st approach, 2-methylimidazole in CH_3OH has been mixed with Ag/ZnO at 20 °C. In another process, $Zn(NO_3)_2$ and $AgNO_3$ have been dissolved in 2-methylimidazole/CH_3OH solution (Fig. 2). The crystal arrangement of the final product may vary during different manufacturing processes [43]. The first composite has been seen as a nanoplate-like form in the SEM image, while the 2nd composite looked smaller than the other sample, which indicates the nucleation stage during the preparation process has decreased with time for the first sample [44]. The second composite exhibited excellent activity, selectivity, and capability towards the detection of mercury ions. The sol-gel method for making Cu/ZnO composite is also used for non-enzymatic amperometric glucose measurement [45]. The simple sol-gel fabrication method permitted achieving a large surface area. The procedure route has been followed according to Omri et al. [46] reported work with zinc acetate in methanol and copper chloride. The final output has been centrifuged, washed, and annealed under supercritical conditions to prepare for glassy carbon electrode/Cu-ZnO/chitosan fabrication. Scherer's equation was used to estimate the crystal size [47], which was less than 30 nm in scale. In addition, it has been found that defects and internal stress can broaden the XRD peak, so the actual value of crystallite size is higher than the average size calculated with Scherer's formula [48, 49]. Nanomaterials-based zinc oxide has some advantages such as high sensitivity and selectivity, lower time, and accurate detection [50]. In another research, the combination of sol-gel synthesis for nucleation and hydrothermal preparation for growing zinc oxide nanorods on a polyethylene terephthalate substrate coated with tin oxide has been successfully applied to fabricate a wurtzite hexagonal structure in nanoscale (entire process under 100 °C) [51]. The whole procedure was below 100 °C, which is a low-temperature approach and can conduct the morphology of zinc oxide nanoparticles towards the best structure.

2.4. Combustion route

The combustion technique suggests an easygoing strategy for acquiring nanomaterials at high temperatures [52]. Energy necessity for this procedure is restrained to the beginning stage only, because the products are obtained by employing the thermal reactions [53]. For example, citric acid has been applied as both fuel and complexing agent for the synthesis of clay-doped ZnO composite, as shown in Fig. 3 [54]. Clay minerals are appropriate adsorbents due to their high capability to absorb metal particles especially heavy metals [55]. The FESEM image of the sample indicates the homogenous spheres of ZnO infused in clay. The performance of sensors improved with clay/ZnO nanocomposite has been enhanced in experiments in 0.1 M KOH for uric acid, resorcinol, and ascorbic acid, indicating the prospect of the clay/ZnO system usage in real-world sensing usages. In another study by Rudresha and coworkers [56], the flash combustion method was employed to fabricate doped CuO-ZnO nanoparticles. Copper (II) nitrate and zinc nitrate ground in citric acid were placed in the porcelain crucible as raw materials. The crucible containing the material was ignited in a furnace under stable conditions and then cooled to ambient temperature overnight. It should be noted that the size of fine powder crystallites increases with enhancing amount of zinc oxide. The cyclic voltammogram of electrodes displayed the

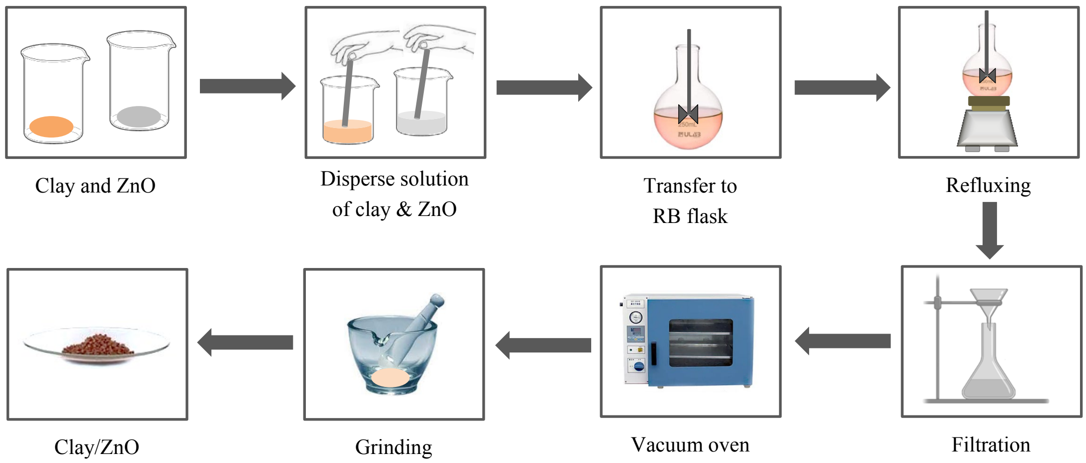

Fig. 3. Flowchart for the preparation of clay/ZnO by combustion-reflux route.

lowest limit of detection of arsenic metal ($\times 10^{-3}$ mol/l). The reported linear fit supports diffusion-controlled activity [57]. Previously, the solution combustion method has been applied in the synthesis of zinc oxide for the electrochemical detection of dopamine [58]. In addition, multiwalled carbon nanotubes (MWCNTs)/Ti-doped ZnO composites have been synthesized via the solution combustion method followed by hydrothermal impregnation [59]. The gas sensing performance of self-assembled porous ZnO/In_2O_3 heterostructures through the solution combustion method has been examined towards Cl_2 and other hazardous gases detection [60]. The schematic structure of the gas sensor and sensitivity of ZnO/In_2O_3 heterostructure upon exposure to 50 ppm of different gases are shown in Fig. 4. Here, sensitivity is defined as the ratio of sensor resistance in test gas to air. As can be seen in this figure, the ZnO/In_2O_3 sensor has a much higher selectivity against chlorine than the others. Different sensitivity under similar situations can be related to different gas types having various intrinsic energy levels to absorb, repel, and react in the active sites of the surface of the sensor material [61]. The ZnO nanoparticles can act as hosts of Pb^{+2} ions without changing the hexagonal wurtzite structure utilizing biomass via the green synthesis method [62]. For this purpose, the eco-friendly solution combustion path achieves a suitable morphology and excellent crystallinity which is confirmed by different spectral analyses. The production of water and carbon dioxide gas molecules during combustion reactions leads to the formation of pores and voids in the composite [63]. According to the evidence, the size and morphology of ZnO are controlled by lead ions [64].

2.5. Spray pyrolysis technique

Spray pyrolysis is another common, inexpensive, and facile synthesis method for the preparation of metal oxides especially ZnO [65] and their composites [66]. Dhamodharan and his team [67] used this method to fabricate ZnO on ITO substrate according to their previous study [68]. FESEM image indicated the vertical growth of zinc oxide nanorods from the substrates and TEM analysis demonstrated the hexagonal structure in suitable agreement with the rod-like morphology. In another research, the deposition of aluminum on ZnO nanolayers has been investigated by chemical spray pyrolysis method with $Zn(CH_3COO)_2.2H_2O$ and $AlCl_3.6H_2O$ in ethanol [69]. The

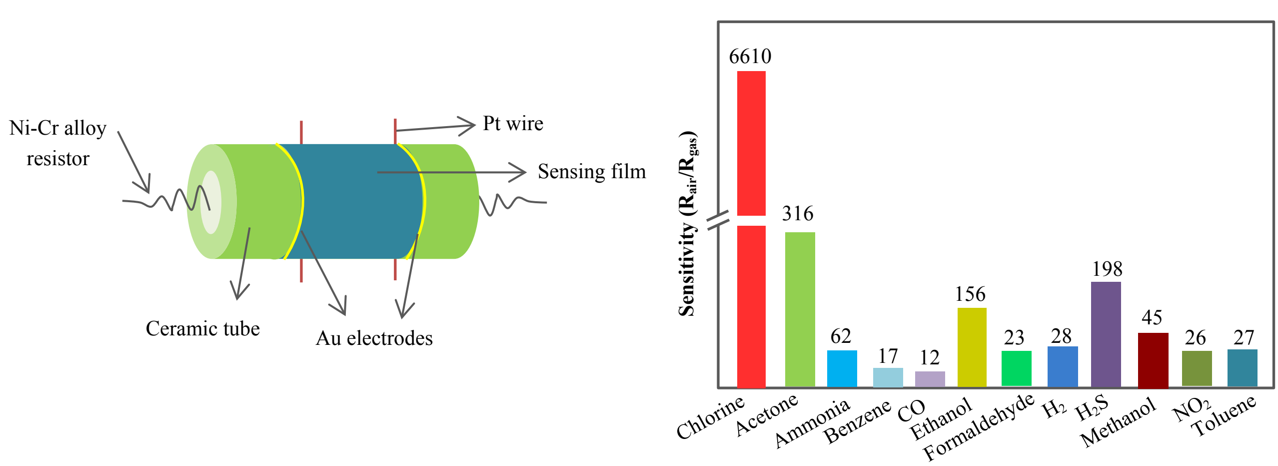

Fig. 4. Schematic structure of the gas sensor and sensitivity of ZnO/In_2O_3 heterostructure against to 50 ppm of various gases (370 °C).

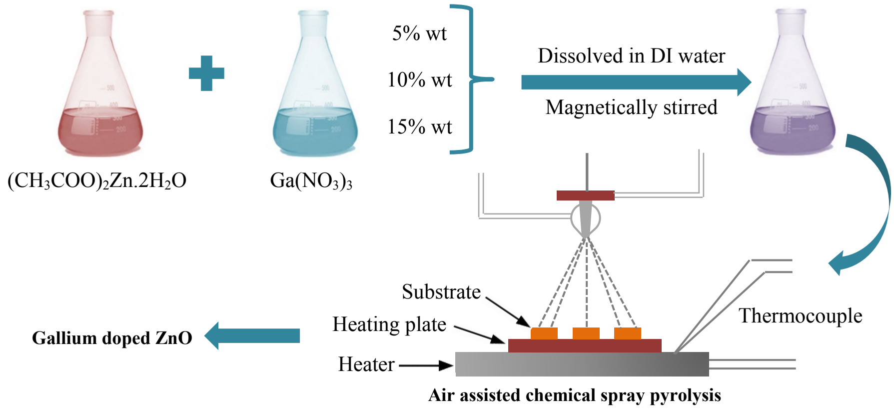

Fig. 5. Schematic preparation of Ga-doped ZnO nanocomposite by spray pyrolysis technique.

hexagonal wurtzite phases have been reported in the XRD results in nanoscale crystalline powders. Additionally, it has been found that Al doping has a positive effect on CO gas detection with ZnO thin films [70]. The improved sensor response of this nanocomposite to carbon monoxide is related to surface roughness, crystalline size, and defect chemistry, which are provided with chemical spray pyrolysis synthesis. In another study, $ZnCl_2$ as a precursor suspension was used for the fabrication of a ZnO-based glucose sensor via a specified spray pyrolysis method at 300 °C [71]. ZnO/glass and Si/ZnO cathode performance have been investigated in phosphate buffer solution. Raman spectroscopy and XRD analysis indicated a hexagonal wurtzite structure of as-prepared samples. After the solution reaches the substrates, zinc oxide nanoparticles grow on the substrates simultaneously with chemical reactions. In addition, it has been claimed that a low-cost glucose sensor can be made using pyrolysis spray. Karthik and coworkers [72] have tried to dope Ni on ZnO via ultrasonic spray pyrolysis deposition by nickel acetylacetonate in a mixture of acetic acid, methanol, and deionized water. Choosing the right temperature for the substrates ensures the accurate construction of the layers. The high amount of nickel has a negative result on gas sensing response because the extra nickel particles located in inter-grain zones create clusters and inhibit these spaces. Furthermore, the distribution of thermal energy is an important parameter in the morphology [73], crystallinity [74], and growth rate [75] of the nanoparticles. A new nanocomposite, GO/ZnO (graphene oxide/ZnO) is an interesting nanomaterial with widespread benefit in electrochemical sensing [76]. In a recent experiment, the spray pyrolysis technique and compressed air have been utilized for the preparation of GO/ZnO nanolayers. The crust-like structure of the GO/ZnO crystals has been exhibited as expected according to the FESEM picture. Previously, it has been claimed that the antibacterial activity of ZnO depends on the solubility of ZnO, which has been verified [77]. Therefore, the said composite cannot inhibit the growth of E. coli at very low ZnO concentrations. The Eu (metal)-doped ZnO films on the glass substrate have been reported via a spray pyrolysis approach [78]. It has been found that the crystallite size and lattice parameters are affected by the europium amount. Metal doping by replacing Zn^{2+} and O^{2-} in the crystal network of zinc oxide can adjust the natural band gap and its conduction behavior [79]. Recently, the successful doping of gallium on zinc oxide has been done by Ani and colleagues [80] to measure small amounts of carbon monoxide gas. The preparation procedure is shown in Fig. 5. Ga cations as dopants supply electrons and zinc cations result in less lattice distortion [81]. Similar changes in growth orientation with Ga-doping have been observed by other researchers [82, 83]. In Ga-doped ZnO, the surface defect, especially the oxygen vacancy, is the main reason for the accurate measurement of carbon monoxide, which is also analyzed by the XPS technique.

2.6. Ultrasonication method

To reduce the time in the synthesis of nanoparticles, the emerging method of ultrasound has been invented to improve the properties of materials during chemical reactions with ultrasound waves [84]. The acoustic cavitation generated through ultrasonication directly affects the reaction rates, crystallinity, morphology, and scale of particles [85]. In a new study, Li and coworkers have prepared a Cu^{+2} sensor based on a UiO-66-NH_2/ZnO nanocomposite via an ultrasonic-mixing technique followed by vacuum drying [86]. One of the common metals in water is copper, which causes human poisoning by biological accumulation; hence the content of copper ions in water should be measured. From the SEM and TEM images of the synthesized sample, it can be seen that the said nanocomposite is uniformly dispersed with a nanorod structure. It is worth noting that UiO-66-NH_2 has a large total pore volume and BET surface area [87]. In another work by Balram et al. [88], functionalized multi-walled carbon nanotubes (fMWCNTs)/ZnO composite have been investigated via a sonochemical synthesis method. Both laser-assisted and ultrasonication procedures have been utilized for the preparation of 3D flower-like ZnO nanoparticles and fMWCNTs/ZnO composite according to Fig. 6. Previous evidence has shown that the electrochemical reduction process of 4-nitrophenol in the vicinity of the pre-prepared composite is controlled by diffusion [89]. In another study, it has been reported that GO-Fe/ZnO nanohybrid could modify screen-printed carbon electrodes [90]. For this purpose, the sonochemical approach has been employed to hybridize 3D honeycomb-like ZnO with iron nanoparticles/loaded GO. Graphene oxide is prepared by Hammer's improved method with graphite functionalization [91]. Based on the SEM image of the as-prepared sample, zinc oxide retains a honeycomb form after the sonochemical process.

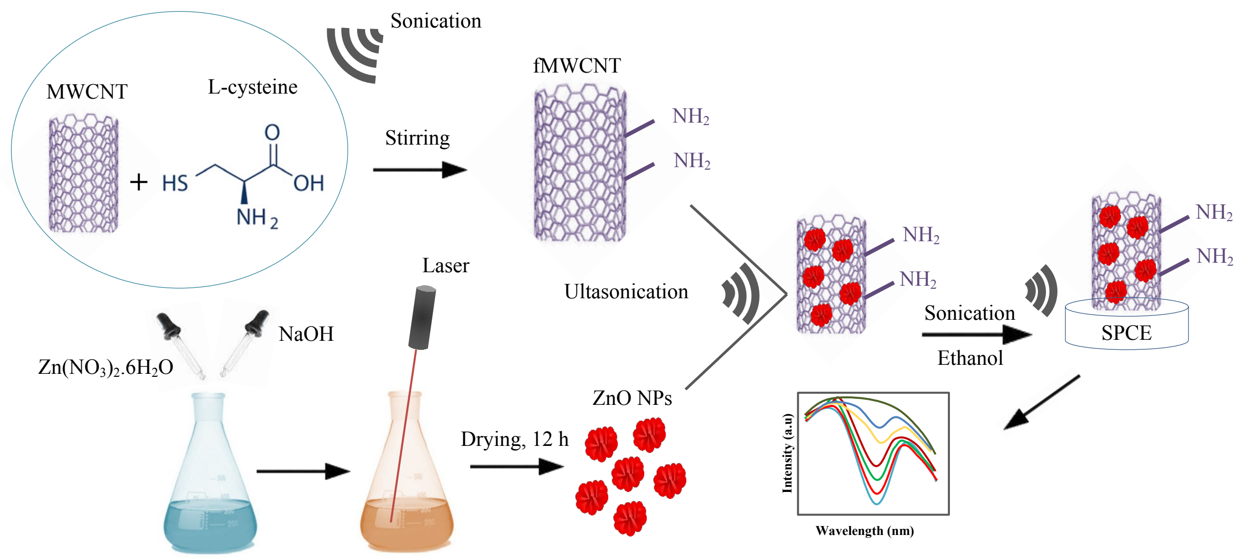

Fig. 6. Schematic representation of the sonochemical synthesis of functionalized multi-walled carbon nanotubes (fMWCNTs)/ZnO composite for 4-nitrophenol detection.

MXenes or transition metal carbides, nitrides, or carbonitrides are an emerging new class of 2D atomically thin layer composites with many applications in materials science, engineering, chemistry, and physics [92–95]. Titanium carbide, $Ti_3C_2T_x$ as one of the most widely explored MXenes, has been investigated for electrochemical sensors [96, 97]. In a new test, Ti_3C_2 has been combined with graphitized multi-walled carbon nanotubes (G-MWCNTs) and zinc oxide nanoparticles for the detection of dopamine to prevent neurological diseases in the human body [98]. Here, the purpose is not to describe how to prepare MXenes, which of course has been mentioned in many papers, but briefly, MXene can be obtained by etching the aluminum layer from the Max phase [92]. As shown in Fig. 7, ZnO/G-MWCNTs/$Ti_3C_2T_x$ composites have been prepared by sonication for 1.5 h in total. The Al etching of $Ti_3C_2T_x$ may cause the difference in layer spacing, hence the hierarchical pore structure is observed in the ZnO/G-MWCNTs/$Ti_3C_2T_x$ nanocomposites [99]. The specific surface area of the mentioned composite is about 45 m^2/g and higher than pure $Ti_3C_2T_x$, and ZnO nanospheres, which indicates that G-MWCNTs and ZnO could increase the performance of $Ti_3C_2T_x$. In addition, it is inferred from the SEM image that large amounts of G-MWCNTs and ZnO are well attached to $Ti_3C_2T_x$. Nitrogen adsorption isotherm also confirms the presence of mesopores in all compounds, which are in agreement with type IV isotherms [100]. For the enzymatic electrochemical glucose sensing in sweat samples, Myndrul et al. [101] have designed a ZnO/MXene skin-attachable stretchable sensor prepared via sonicating method. Based on the MILD route, Ti_3AlC_2 has been etched in the LiF/HCl solution to construct $Ti_3C_2T_x$ MXene [102], and the simple catalyst-free oxidative-metal-vapor-transport process has prepared ZnO powder [103]. To fix the uneven distribution of $Ti_3C_2T_x$ on the ZnO surface, APTES treatment (an additional chemical treatment) is required to form a coherent layer of $Ti_3C_2T_x$ nanoparticles on the ZnO. Therefore, uniform coverage of $Ti_3C_2T_x$ nanoflakes on the entire ZnO surface (individual "legs") can be seen in the TEM images. Liu and Li have proved that titanium–oxygen has the most stable bond in the $Ti_3C_2T_x$ functional group [104]. Therefore, the Ti–OH bond forms most of the terminated groups after the formation of $Ti_3C_2T_x$/ZnO/powder, which is approved by XPS spectra. Previously, Wang and colleagues [105] reported that the interaction between $Ti_3C_2T_x$ and ZnO facilitates electron transfer and there is a direct relationship between the rate capacity and the specific surface area, and the number of 3D channels for fast diffusion of charge carriers.

2.7. Microwave-assisted approach

The microwave-assisted synthesis method has been used since the last century in industrial and academic laboratories to prepare a wide range of organic substances, which has solved safety and reproducibility concerns with the advancement of equipment [106]. In a recent study, Chakraborty et al. [107] prepared conical ZnO-Ag_2O in an aqueous medium utilizing a microwave synthesis technique at 80% (640 W power output) with silver nitrate, sodium hydroxide, and zinc acetate dehydrate. The peaks of the XRD pattern correspond to the cubic phase of Ag_2O and the hexagonal wurtzite phase of ZnO [108]. In addition, EDS spectra showed the elemental composition of ZnO-Ag_2O nanocomposite to be very pure and without any impurity or pollution. The particle size distribution diagram had a peak at 90 nm for the nanocomposite containing 1% and 5% Ag_2O in 40 to 140 nm. The application of the prefabricated ZnO-Ag_2O semiconductor here is mainly the simultaneous detection and removal of 4-Nitrophenol in the aquatic environment, which is an importance cantaminant according to USEPA. In addition to silver oxide, other metal oxides are used in combination with ZnO to fabricate electrodes for electrochemical sensors. In this regard, ZnO-CeO_2 nanoparticles have been reported as a highly sensitive, reproducible, and reliable electrode for nitroaniline sensing [109]. In another work, Nawaz et al. [110] successfully utilized a facile microwave-assisted strategy to prepare rGO/ZnO-based sensor for the detection of 2,4,6-Trichlorophenol as a hazardous phenolic compound. The XRD pattern indicates the successful synthesis of graphene oxide according to the modified Hummer method [111]. The

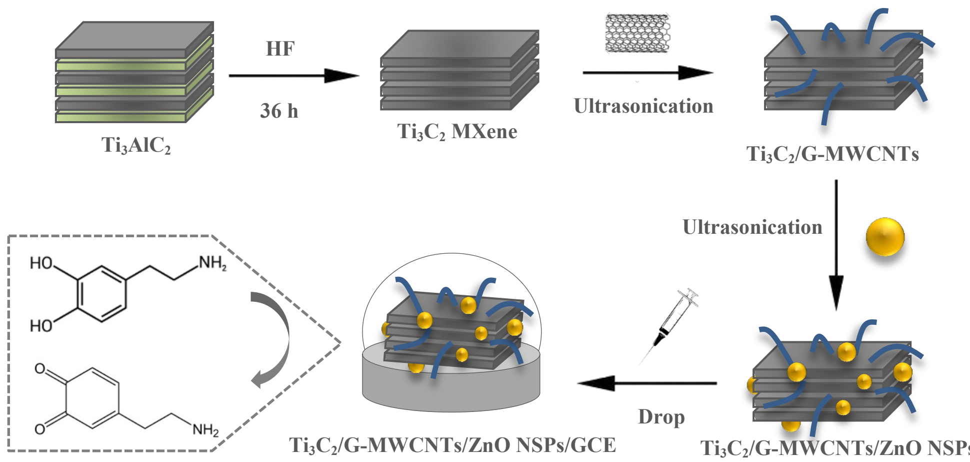

Fig. 7. Schematic explanation of the synthesis of ZnO/G-MWCNTs/$Ti_3C_2T_x$ composites for sensing dopamine.

microwave-assisted synthesis of engineered ZnO/rGO nanoparticles can produce a composite with high phase purity and ultra-fine sheet height, which has excellent sensitivity in monitoring the phenolic ring. It is worth mentioning that rGO/ZnO composite prepared by microwave has a high capability in non-enzymatic electrochemical glucose sensing [112]. Furthermore, It has been reported that microwave-assisted biogenic electrochemical route as a green strategy using mangosteen peel (biogenic reducing agent and stabilizers) can be applicable to produce Ag/ZnO nanoparticles [113]. Interestingly, the temperature of microwave radiation has a significant effect on the size and crystallinity, which are important factors on the performance of electrochemical detecting [114, 115]. This eco-friendly and cost-effective substrate proposes a platform for carbaryl sensing in agricultural products. Another strategy to increase the sensitivity is the use of quantum dots on ZnO, which was mentioned in the work of Yan et al. [116]. They developed ZnO/MWCNTs composite with carbon dots using the rapid microwave-assisted approach for the simultaneous determination of catechol and hydroquinone.

2.8. Self-assembly method

Self-assembled compounds are the result of specific and local interactions of individual or linked components that spontaneously form a molecular or macroscopic organized structure or pattern [117]. For example, Au/Nafion/$Ti_3C_2T_x$/ZnO composites have been prepared via a self-assembly process to fabricate a dopamine electrochemical sensor [118]. Exfoliation of aluminum from Ti_3AlT_x has been described in previous literature by LiF-HCl etching [119]. XRD peaks [120] and SEM images etc. confirmed the formation of $Ti_3C_2T_x$ monolayers and the synthesis of $Ti_3C_2T_x$-based nanocomposite. XPS spectra of ZnO/$Ti_3C_2T_x$ verify relatively high strength in Ti $2P_{3/2}$ of TiO_2 which indicates the co-complexation of $Ti_3C_2T_X$ in ZnO nanosheets and proves that the desired composite has been successfully produced [121]. In another work, a biomolecule assembly constructed chicken ovomucoid (CHOM) bioconjugate and ZnO quantum dots (QDs) have been studied for the detection of allergy to (CHOM) [122]. The high percentage of allergy to trypsin inhibitor in children and adults makes it important to detect it in food products. Self-assembled ZnO QD bioconjugate can label chicken ovomucoid and other kind of lectins through electrostatic interaction and bring a new generation of sensors for allergens assay. In another research, a humidity sensor has been established by a self-assembled hierarchical MWCNTs/ZnO composite, as shown in Fig. 8 [123]. The results have shown that with the absorption of water molecules at high humidity, the composite layers swell and their distance increases, which leads to an enhancement in the resistance of the sensor [124, 125]. In addition, MWCNTs/ZnO composites can modify carbon paste electrodes for the determination of avanafil with good recovery in commercial dosage [126]. In the detection of ethylenediamine, an electrochemical sensor has been suggested based on hierarchically nanosize star-shaped ZnO [127]. Self-assembled nano- and microparticles of ZnO have been prepared via a simple hydrothermal synthetic approach. The XRD spectrum has indicated the as-synthesized sample is exclusively composed of zinc oxide with a hexagonal wurtzite structure of ZnO particles. It is believed that the growth mechanism followed the natural growth of ZnO crystals [128]. However, in this study, the in-situ synthesis of hexagonal ZnO was carried out with the help of the conducting agent cetyltrimethylammonium bromide. The sensor made of prism-shaped zinc oxide particles has taken an important step in the electrochemical measurement of amines. In a new work, Farhan et al. [129] have modified carbon paste electrode with ZnO nanoparticles for the determination of difloxacin HCl. The traditional carbon paste electrode included powder graphite, an ion exchanger, and a plasticizer. It should be noted that the mobility of the ionophore molecules and the dielectric constant of the paste phase are influenced by the type of plasticizer [130, 131].

3. Recent application of the ZnO-based electrochemical sensors

To date, several ZnO-based electrodes have been utilized in the electrochemical sensors. As provided in Table 1, many synthesis techniques are employed to detect gases, drugs, and biomolecules. This table is compiled based on the latest achievements of researchers.

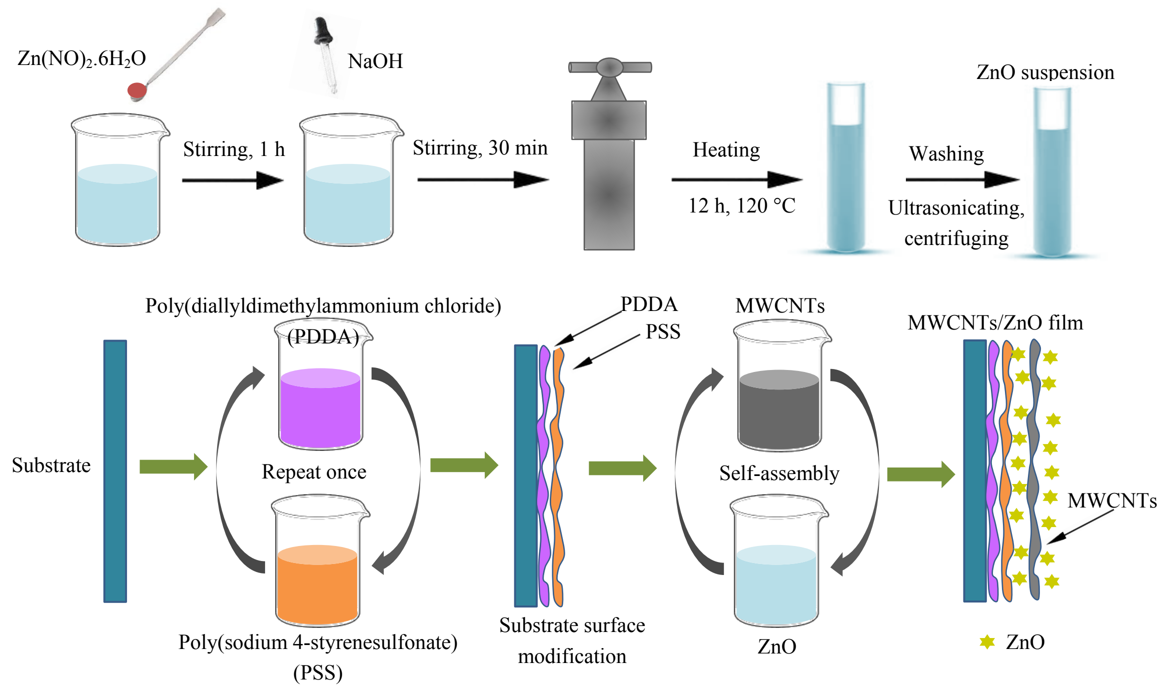

Fig. 8. Synthesis of ZnO suspention and MWCNTs/ZnO nanocomposite through electrostatic layer-by-layer self-assembly technique.

Table 1. Comparison of the electrochemical performances of recent ZnO based sensors.

Material	Synthesis method	Detection	Sensitivity/response	Detection limit	Ref.
ZnO/carbon/glassy carbon	Co-precipitation	Terbutaline	0.30411 µA/M	25 nM	[16]
CuO/ZnO/graphite	Co-precipitation	Urea	0.48 $mA.\mu g^{-1}.cm^{-2}$	2.5 µg/ml	[19]
Fe_2O_3/ZnO/graphite	Co-precipitation	Urea	2.07 $mA.\mu g^{-1}.cm^{-2}$	2.5 µg/ml	[19]
Nafion/ZnO–CuO	Co-precipitation	Citric acid in fruit juices	0.4358 $\mu A.\mu M^{-1}.cm^{-2}$	21.78 ± 1.09 µM	[22]
ZnO/Co_3O_4	Co-precipitation	Hydroquinone & resorcinol	10 µM, 10 µM	3.226 µM, 2.92 µM	[24]
NiO/ZnO/glassy carbon	Hydrothermal-anealing	Rutin	11.0 nM	2.52 $\mu A.\mu M^{-1}.cm^{-2}$	[29]
ZnO-Au	Hydrothermal	Gallic acid	0.0536 $\mu A.\mu M^{-1}$	0.0118 µM	[32]
ZnO-Au/rGO	Hydrothermal	Rutin	0.06 $\mu mol.l^{-1}$	1.0 $nmol.l^{-1}$	[33]
ZnO/carbon cloth	Hydrothermal	Hydroxychloroquine	0.279 $\mu A.\mu M^{-1}.cm^{-2}$	0.09 µM	[37]
Ag/ZnO/ZIF-8	Sol-gel	Hg^{2+}	56.06 $\mu A.\mu M^{-1}.cm^{-2}$	40 nM	[42]
Cu/ZnO/chitosan	Sol-gel-annealing	Amperometric glucose	36.641 $\mu A.mM^{-1}.cm^{-2}$	57 µM	[45]
ZnO doped CuO	Flash combustion	Arsenic		10^{-3} $mol.l^{-1}$	[56]
MWCNT/Ti-doped ZnO	Combustion-hydrothermal	Glutamate & ascorbic acid			[59]
In_2O_3/ZnO	Solution combustion	Cl_2 gas	6610		[60]
Graphite-Pb-ZnO	Solution combustion	Paracetamol & dextrose	1 to 5 mM		[62]
Ni-doped ZnO/sodalime glass	Ultrasonic spray pyrolysis deposition	Propane & CO	4×10^4		[72]
Undped ZnO	Dip coating	Propane & CO	6		[72]
GO/ZnO	Spray pyrolysis	Nitrophenols, antibiotics, biomolecules			[76]
Ga-doped ZnO	Spray pyrolysis	CO	0.08 ppm		[80]
UiO-66-NH_2/ZnO	Ultrasonic mixing	Cu (II)	6.46 $\mu A.\mu M^{-1}$	0.01435 µM	[86]

Table 1. Continued.

Material	Synthesis method	Detection	Sensitivity/response	Detection limit	Ref.
fMWCNTs/ZnO	Ultrasound	4-nitrophenol	11.44 $\mu A.\mu M^{-1}.cm^{-2}$	0.013 μM	[88]
GO-Fe/ZnO	Sonochemical	Antipsychotic drug chlorpromazine	7.56 $\mu A.\mu M^{-1}.cm^{-2}$	0.02 μM	[90]
G-MWCNTs/Ti_3C_2/ZnO	Ultrasound	Dopamine	0.01–30 μM	3.2 nM	[98]
$Ti_3C_2T_x$/ZnO	Ultrasonicaton-mixing	Glucose	0.05–0.7 mM	17 μM	[101]
Ag_2O-ZnO	Microwave-assisted assembly	4-nitrophenol	1.6 $\mu A.\mu M^{-1}.cm^{-2}$	23 nM	[107]
ZnO/rGO	Microwave	2,4,6-trichlorophenol	0.01 μM	0.0067 μM	[110]
Bio-Ag/ZnO	Electrochemically microwave-assisted biogenic	Carbaryl pesticide	0.303 $\mu A.\mu M^{-1}.cm^{-2}$	~0.27 μM	[113]
Ni^{3+} doped ZnO	Solution combustion	Ascorbic acid			[132]
$2ZrO_2$–7ZnO	Solution combustion	Evan's blue dye			[133]
In/ZnO	Spray pyrolysis	CO	1 ppm		[134]
ZnO/$Ti_3C_2T_x$ p-n heterostructure	Heating-sonication	NO_2	54% of 10 ppm		[135]
ZnO/$Ti_3C_2T_x$	Hydrothermal	Acetone			[136]
N-doped Ti_3C_2/ZnO	Thermal treatment	Chloramphenicol	0.1 ng/ml	0.019 ng/ml	[137]
ZnO/$Ti_3C_2T_x$/Nafion/Au	Self-assembly	Dopamine	96 nA/μM	0.076 μM	[118]
ZnO/$Ti_3C_2T_x$	Solvothermal	NO_2 gas			[138]
MXene/La^{3+}-doped ZnO/hemoglobin	Hydrothermal	H_2O_2	0.2 μM	8.0×10^{-8} M	[139]
$Ti_3C_2T_x$ MXene/GO/CuO/ZnO	Hydrothermal	NH_3	96% at 200 ppm	4.1 ppm	[140]
MOFs-derived In_2O_3/ZnO/$Ti_3C_2T_X$	Electrostatic self-assembly	Ethanol	2.15 ppm		[141]
ZnO/MWCNTs	Ultra-sonication	Epinephrine	0.4 μM to 2.4 μM	0.016 μM	[142]
ZnO/poly(diallyldimethylammonium chloride)	Sparking-drop casting	Glyphosate	0 μM–5 mM		[143]
ZnO quantum dots decorated carbon nanotubes (CNTs)	Thermal vapor deposition-metal sparking	Methanol	15.8% of 500 ppm		[144]
Nd_2O_3/ZnO	Wet-chemical	2,4-dinitrophenol	28.481 $nA.nM^{-1}.cm^{-2}$	0.33 pM	[145]
ZnO–$YMoO_4$	Precipitation-thermal polymerisation	Uric acid	0.9839 nM	14.8 nM	[146]
MoS_2-ZnO	Hydrothermal	Glucose	639.12 $\mu A.\mu M^{-1}.cm^{-2}$	~0.025 μM	[147]
$ZnMnO_3$/ZnO	Co-precipitation-thermal calcination	Chlorpromazine	0.760 $\mu A.\mu M^{-1}$	0.019 μM	[148]
RuO_2 doped ZnO	Wet-chemical	L-glutamic acid	5.42 $\mu A.\mu M^{-1}.cm^{-2}$	96.0 ± 5.0 pM	[149]
Cu doped ZnO/TX-100-surfactant MCPE	Co-precipitation	Paracetamol & adrenaline	10 μM 10 μM	4.6 μM 3.9 μM	[150]
Cu doped ZnO	Hydrothermal	Dopamine	2630 nA/μM	55 nM	[151]
ZnO-rGO	Self-assembly	Dopamine	458 nA/μM	0.167 μM	[152]
MnO_2/ZnO	Sonication-air drying	Ciprofloxacin in honey	0.5 μM	0.21 μM	[153]
Nickel phthalocyanine/ZnO/CNTs	Ultrasound	Dopamine	0.16 μA/μM	7.0 nM	[154]
ZnO- crude black pepper	Microwave irradiation	Uric acid	40.485 $\mu A.mM^{-1}.cm^{-2}$	1.65 μM	[155]
ZnO doped CeO_2	Microwave	Nitroaniline	550.42 $\mu A.mM^{-1}.cm^{-2}$	0.25 mM	[109]
Pd–$Au_{gr\text{-}alloy}$@ZnO core-shell	2 step hydrothermal	H_2 gas	174		[156]
ZnO-doped graphitized carbon	Hydrothermal	Hydroxychloroquine	10^{-3} M	1.33×10^{-7} M	[157]
ZnO/$ZnFe_2O_4$	Simple mixing	Furazolidone	0.78 $\mu A.\mu M^{-1}.cm^{-2}$	0.65 μM	[158]
rGO@ZnO	Hydrothermal	Ascorbic acid	0.1 mmol		[159]

4. Conclusions and outlooks

It is necessary to maintain balance in the environment and monitor environmental pollution. On the other hand, identifying bacteria and viruses and measuring harmful compounds for the human body is a way to deal with all kinds of diseases. Therefore, the focus on the development of electrochemical sensors with high detection capability and good selectivity has increased among researchers. Among the nanocomposites used in manufacturing electrochemical sensors, ZnO is highly popular due to its nontoxicity, biocompatibility, low price, and outstanding features. In this review, the new techniques for the preparation of ZnO-based nanomaterials have been outlined with a dissection of the structure and morphology. There are simple, facile, and economical methods for the synthesis of hybrid nanocomposites of ZnO, which produce nanostructures with high quality and specific surfaces.

Various synthesis techniques are explored for the preparation of ZnO nanostructures, such as the precipitation method, hydro/solvothermal method, sol-gel route, solution combustion approach, ultrasonication procedure, spray pyrolysis way, microwave-assisted approach, self-assembly thechnique, simple mixing, and combined wet-chemical method. The performance of electrochemical sensors depends on the type of composite used in its electrode. Electrochemical sensors with nanostructured electrodes have high accuracy, selectivity, and sensitivity, which is of special interest in the field of nanotechnology. Besides, multidimensional sensors have shown better performance than one-dimensional nanostructures. Although it is easy to fabricate 0D zinc oxide, its low mobility is considered a weakness that can be solved by adding another dimension. Currently, controlling the dimensions and scale of produced particles is one of the challenges of researchers.

The following points should be considered in future research so that a better future for the development of composites based on zinc oxide is ahead:

- Choosing a useful method to better control the synthesis to maintain the particle size, crystallinity, morphology, and increase the specific surface area. The crystal arrangement of the final product depends on the type and conditions of the synthesis process.
- Detailed examination of synthesis conditions (temperature, acidity, agent, concentration) in various methods of preparation of electrodes based on zinc oxide and their effect on growth rate, surface roughness, crystalline size, distance of nanosheets, and defect chemistry.
- Probably, by combining two or more synthesis methods, the actualization potential and obtaining a suitable nanocomposite can be provided for constructing electrodes for commercial electrochemical sensors.
- Introduction of hybrid nanocomposite suitable for various applications in the measurement of gases, biomolecules, heavy metal ions, etc.
- Utilizing doped zinc oxide nanocomposites with interesting nanostructures as a competitor for semiconductor hybridization.
- Development of new hybrid nanomaterials with high photoelectric performance, high temperature resistance, acceptable selectivity, and excellent response time in facing different environmental conditions.
- Understanding the interference behavior and possible interactions between the compounds used in the preparation of nanomaterials.
- Production of cost-effective and environmentally friendly nanocomposites without any impurity or pollution.
- Developing a durable, stable, and high-performance electrochemical sensor that is fabricated through a green synthesis strategy.

CRediT authorship contribution statement

Asieh Akhoondi: Writing – original draft, Resources, Supervision.
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Muhammad Nawaz Sharif: Writing – review & editing.
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Hadi Davardoost: Writing – review & editing.
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Data availability

As this is a review article, no new data were generated. All information is publicly available or cited appropriately within the article.

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