

Review

Progress in Layered Double Hydroxide-Based Materials for Gas and Electrochemical Sensing Applications

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Abstract: In the current scenario, it is considered that environmental pollution is one of the significant challenges for the global world. Various toxic and hazardous substances such as hydrazine, phenolic compounds, and pharmaceutical waste significantly contribute to environmental pollution. Exposure to such substances and compounds increases the chances of negative effects on human health as well as the environment. Therefore, it is considered that monitoring toxic gases and hazardous substances/compounds is of great significance. In the past few years, layered double hydroxide (LDH)-based materials have received significant interest for gas sensing and electrochemical sensing studies. The presence of layered structured, larger surface area, decent conductivity, and electrochemical properties makes them a suitable material for sensing applications. This motivates us to summarize the recent progress in the development of LDH material-based gas and electrochemical sensors for the detection of toxic and hazardous gases/compounds. It was observed in previous reports that LDH-based materials are promising candidates for gas sensing as well as electrochemical sensing applications. It was found that LDH and its composites may exhibit larger surface areas and high electrical conductivity when combined with other materials such as metal oxides, MXenes, polymers, and metal sulfides. Thus, researchers prepared hybrid composites of LDH-based materials for gas and electrochemical sensing applications. It is worth mentioning that many solvents which have negative impacts on the environment could not be detected by electrochemical methods, while some toxic compounds/substances could not be determined by gas sensing methods. This may create a gap between the determinations of different kinds of pollutants that exist in the environment. Thus, it is required to find a bi-functional material which can be used for kind of sensing technology. In addition, it may also overcome the limitations or gap between the two sensing techniques. LDH-based materials have demonstrated excellent performance in gas and electrochemical sensing technologies. Thus, it would be of great significance to employ the single LDH-based materials for gas as well as electrochemical sensing applications. In this review article, we have tried our best to compile the progress in the various LDH-based materials for gas sensing and electrochemical sensing applications towards the detection of hazardous compounds.

Keywords: gas sensors; layered double hydroxides (LDHs); hazardous compounds; sensors



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1. Introduction

In the past few years, it was observed that environmental pollution has been increasing significantly due to urbanization and industrialization [1]. The increase in industrial activities may lead to a substantial increase in the burning of fossil fuels, which releases harmful

pollutants such as carbon dioxide (CO₂), sulfur dioxide (SO₂), and particulate matters into the atmosphere, which may contribute to air pollution and climate change [2,3]. Industrial processes may also produce a large amount of waste water-containing chemicals such as hydrazine, hydrogen peroxide (H₂O₂), phenolic compounds, heavy metals, etc., which are often discharged into water bodies, and cause negative impacts on the environment, aquatic life, and human health [4–7]. In addition, exposure to toxic gases and hazardous compounds may cause several health issues such as chronic diseases and respiratory diseases [8]. Thus, it is clear that monitoring toxic gases and hazardous compounds would be of great significance in order to reduce their negative impacts on human health and the environment.

Sensor technology is one of the promising approaches to monitor toxic gases and compounds [9]. Gas sensing is an efficient technique for the monitoring of various toxic gases and has been widely used for the detection of various gases [10,11]. According to the reported studies, gas sensors have promising features for the detection of various organic volatile gases (VOCs) [12], whereas the electrochemical method is the most promising technique for the sensing of various toxic/hazardous compounds [13]. In previous reports, various materials such as reduced graphene oxide [14], carbon nitride [15], metal–organic-frameworks [16], MXenes [17], polymer [18], metal oxides [19], and layered double hydroxides (LDHs) [20] were used in sensing applications.

Recently, LDH-based materials have received enormous attention because of their excellent optoelectronic properties [20]. LDH materials, which are also known as metal alloy hydroxides or hydrotalcite, are inorganic materials with a two-dimensional structure (2D) [21]. Generally, LDH materials consists of a mixture of metal hydroxides with the presence of different anions (anionic layer) and oxidation states (cationic layer), which are present next to each other [22], and LDH materials possess excellent structural and chemical properties. The high specific surface area, acceptable stability, and layered structure of LDH materials makes them suitable for various optical and electrical applications. In previous reports, copper (Cu)-, nickel (Ni)-, aluminum (Al)-, cobalt (Co)-, magnesium (Mg)-, iron (Fe)-, and manganese (Mn)-based LDH materials were prepared by various synthetic procedures [23–30]. The LDH materials were also combined with various metal oxides and polymers to further improve their properties for various optoelectronic applications [29]. The LDH-based hybrid composites attracted the scientific community due to their unique functional, structural properties, and synergistic interactions. LDH-based composites also exhibited high electrical conductivity, larger surface areas, and various active sites for adsorption/catalytic reactions [31]. For gas sensing studies, LDH and its composites facilitated electron transfer and enabled the sensors to detect the toxic gases rapidly. Similarly for electrochemical sensing studies, LDH-based hybrid materials may enhance sensitivity, selectivity, signal response, and stability for the determination of heavy metals and environmental pollutants.

Herein, we report on the use of LDH-based materials for gas and electrochemical sensing of environmental pollutants. We believe that recent articles may be valuable to material scientists who are sincerely engaged in the development of cost-effective LDH-based materials for these sensing applications.

2. LDH-Based Materials in Gas Sensors

With the rapid industrial development and growing living standards, air pollution has become a serious global problem and poses serious risks to human and animal health, ecosystems, and the environment [32]. According to a World Health Organization (WHO) report, more than seven million people die worldwide every year due to air pollution [31]. Major contributors to air pollution are mobile vehicles, oil refineries, power plants, and

chemical industries. Therefore, monitoring and controlling combustion-related emissions are of prime importance to protect public health, assure food safety, support medical growth, enhance air quality, and improve environmental sustainability. A gas sensor is a chemical sensing device which detects gas molecules in the environment by converting chemical information into an electronic signal such as current, frequency, or voltage change and reduces risks to human health at an early stage [33–36]. Hence, there is an urgent need to develop advanced, high quality, efficient energy gas sensors for environmental monitoring and society protection for the real time detection and monitoring of toxic and flammable gases, as well as volatile organic compounds (VOCs) [37]. With the development of the internet and wireless technology, gas-sensing intelligent systems have been securely integrated with smart living, becoming a critical part of our daily life and modern industries [35]. Gas sensors are widely applied in biochemical, chemical, and food industries for applications in diagnostics, air quality monitoring, food testing, and the detection of toxic, flammable, and explosive gases [38]. For example, ethanol sensors are widely explored for various applications including alcohol detection on human breath for drunk driving, to assess the quality of wine, to monitor industrial leakages, and to ensure food and biomedical safety [39]. The advancement in science and technology has motivated the way for the development of gas sensors with high sensitivity, good selectivity, and long-term stability [40]. As a result, great efforts have been carried out to design novel sensing materials since the first chemresistive metal oxide-based gas sensor was introduced. So far, in addition to conventional semiconductor oxide, a variety of gas-sensing materials such as conductive polymer, organic compounds, and carbon nanotubes have been utilized for gas detection using techniques like catalytic, optical, electrochemical, and acoustic gas sensors [41,42]. However, the performance of the sensors is mainly dependent on some basic properties such as sensitivity, selectivity, detection limit, response, and recovery time. In addition, factors such as low power consumption, shape, size, and wireless functionality play a critical role in boosting gas-sensing performance and making sensors more appealing [43]. Figure 1 exhibits the classification of gas-sensing materials into two categories based on electrochemical and other principles, associated with fundamentals of the gas-sensing and gas-sensitive materials [44]. Nanomaterials have attracted significant attention due to their outstanding properties, such as large surface area, and superior mechanical, chemical, physical, as well as electrical, characteristics. The large surface area is critical for improving sensing performance by providing a greater number of active sites. Moreover, nanomaterials can be further functionalized. To enhance the sensitivity, selectivity, and stability of sensors, scientists have devoted substantial effort into modifying electrodes with various nanomaterials, such as multiwalled carbon nanotubes (MWCNTs), gold nanostars, metal–organic frameworks (MOFs), covalent organic frameworks (COFs), and layered double hydroxides (LDHs) [42].

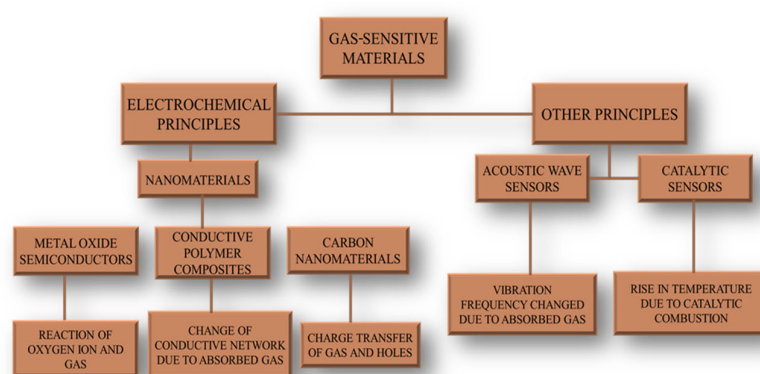


Figure 1. Classification of gas-sensing materials [9,10,34–36,44].

Among the various electrode modification nanomaterials, LDHs have attracted significant attention and have become a rapidly increasing area of research in recent years. LDHs are exciting materials characterized as two-dimensional (2D) anionic lamellar clay materials [40]. LDHs demonstrate various chemical and physical properties due to their unique molecular structures [45]. These include a high surface area, catalytic activity, chemical and thermal stability, anion exchange capability, tunable and flexible interlayer spacing, and cost-effectiveness [46]. LDHs, also referred to as hydrotalcites or metal hydroxide alloys, are inorganic materials made up of layers of brucite-like sheets stacked together [46]. These 2D layered structures are aligned parallel and are held together by electrostatic interactions such as hydrogen bonds or Vander Walls forces, along the vertical axis as shown in Figure 2 [47]. LDHs are made up of a combination of metal hydroxides with different oxidation states (forming cationic layer) and anions (forming anionic layers) ordered next to each other (Figure 2) [47]. The natural inorganic hydrotalcite was first discovered in Sweden around 1842, and approximately a century later, Feitknecht successfully synthesized it in the laboratory in 1942 [48,49]. It was then registered as the first patent under the name of Brocker in 1970 [47]. However, research on using LDHs as gas sensors began back in 2006. The general formula of an LDH is $[M_{(1-x)}^{2+} M_x^{3+} (OH)_2]^{x+} (A^{n-})_{x/n} \cdot mH_2O$, where M^{2+} represents a divalent cation (Mg^{2+} , Zn^{2+} , Ni^{2+} , Co^{2+} , Ca^{2+} ...), M^{3+} is a trivalent cation (Al^{3+} , Ga^{3+} , Cr^{3+} , Fe^{3+} ...), A^{n-} denotes the interlayer anion of valance n (CO_3^{2-} , SO_4^{2-} , polyoxometalates, porphyrins, etc.), and X is the molar ration of $M^{3+}/(M^{2+} + M^{3+})$, typically ranging from 0.2 and 0.4 due to structural stability factors (Figure 2) [47,50–52].

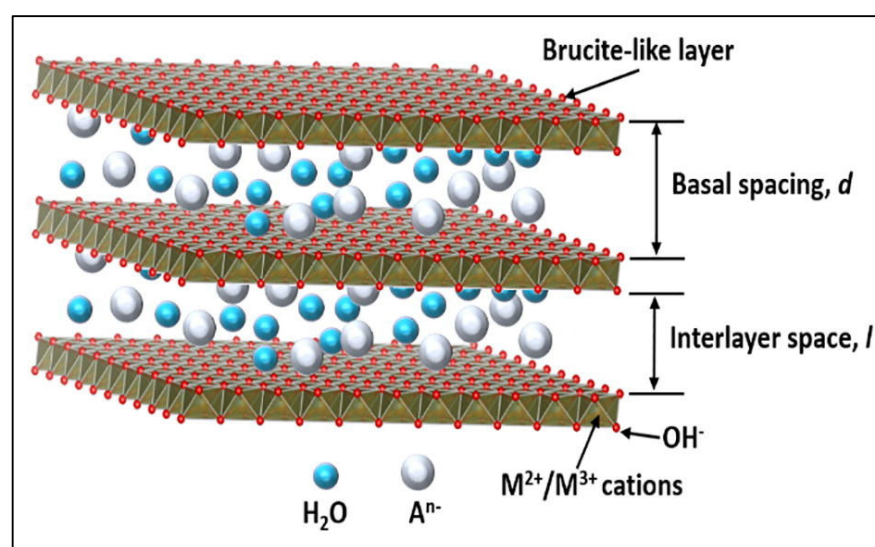


Figure 2. Schematic illustration of typical LDH structure and its chemical components [52].

Gas sensors composed of organic–inorganic hybrids offer several advantages, such as molecular-scale thickness control, low cost, and high reproducibility. However, their design is challenging due to difference in vapor pressure and decomposition temperatures between the organic and inorganic components using standard physical techniques. Taking LDH nanosheets into account, they can serve as 2D building blocks for producing ultrathin films (UTFs) with superior functionalities. Therefore, (calcein-ZnS)₃₀ UTF was fabricated using two-step layer by layer (LBL) assembly process using calcein and exfoliation Zn₂Al layered LDH nanosheets, followed by an in situ gas/solid reaction with H₂S [50]. The synthesized (calcein-ZnS)₃₀ UTF composite, made up of organic molecules and inorganic semiconductors, facilitates the development of an innovative ethanol sensor with high sensitivity, fast response, as well as quick recovery time, operating at a relatively low working temperature (90 °C). Xiao et al. [53] reported the synthesis of a {pyrenetetrasulfonate(PyTS)/ZnS}_n UTF

sensor using an LBL electronic deposition technique. This process consists of an alternating assembly of 1,3,6,8-PyTS and exfoliated Zn₂Al LDH nanosheets, followed by an effective in situ gas/solid sulfurization reaction of H₂S. The UV/Vis spectroscopy was used to observe the assembly process, which indicates the regular stepwise growth of (PyTS/LDH)_n UTFs with each deposition cycle. The performance of both synthesized (PyTS/LDH)_n UTFs and sulfurized (PyTS/LDH)_n UTFs was investigated in the presence of various gases such as ammonia, hydrogen, carbon monoxide, methane, acetylene, and ethanol. While both sensors exhibited sensitivity to all tested gases, the sulfurized (PyTS/LDH)_n UTFs showed a much better response to ethanol, even at a relatively low operating temperature. They reported that the enhanced performance of the sensor towards ethanol could be attributed to the synergistic interactions between the inorganic ZnS and organic pyrene components. Morandi et al. reported the synthesis of Pt/Zn/Al layered double hydroxides as gas-sensing materials [54]. They employed two simple synthesis routes, classical co-precipitation and the sol-gel method, for the synthesis of a Pt/Zn/Al LDH gas sensor. The Pt/Zn/Al LDH sensor synthesized with the help of the sol-gel method was found to be pure, and the results are consistent with previously reported X-ray diffraction (XRD) results (Figure 3a). However, the same Pt/Zn/Al LDH sensor synthesized via the co-precipitation method exhibited trace amounts of the ZnO phase in its XRD pattern (Figure 3b). The sensor's performance was then investigated using different VOCs such as CO (100 ppm), CH₄ (500 ppm), and benzene (C₆H₆, 10 ppm) with and without the addition of Pt. These measurements were performed at 450 °C using a conductance measurement method. As shown in Figure 3c, the addition of Pt improves the electrical response, with the highest sensitivity found for CH₄ in dry air. However, it is difficult to distinguish the response of different gases in dry air. Therefore, the sensing performance of all three VOCs was also investigated in the presence of wet air. As illustrated in Figure 3d, carbon monoxide exhibits the highest response under these conditions.

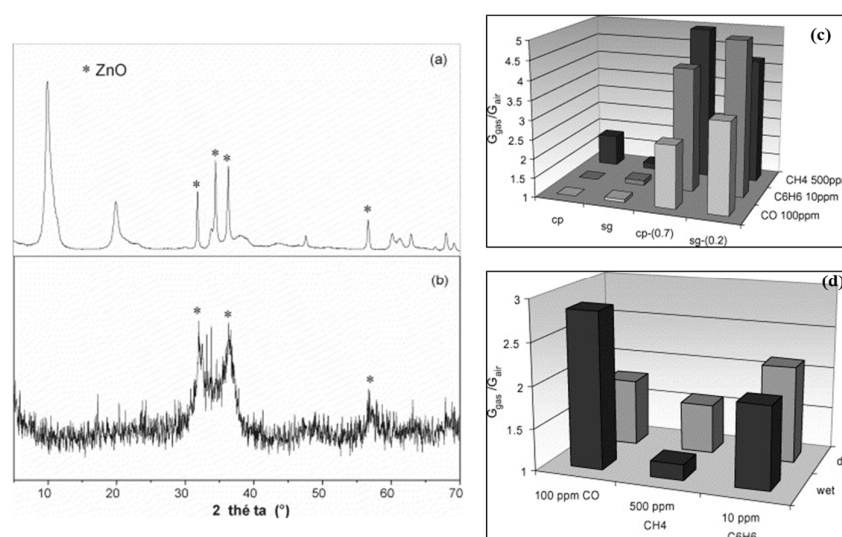


Figure 3. (a) XRD pattern of co-precipitated LDH (a) with 0.7% of Pt and (b) cp-2 mixed oxide. (c) Responses to CO, C₆H₆, and CH₄ of two Pt-containing samples (cp-(0.7), sg-(0.2)) and two correspondent samples without Pt (cp, sg) at 450 °C in dry air. (d) Responses to CO, CH₄, and C₆H₆ in dry and wet air at 400 °C of cp-(2) thick films [54].

In another study, Guan et al. reported the synthesis of a ZnO/ZnAl₂O₄ sensor via the calcination method [55]. In a typical synthesis approach, all starting reagents were dissolved in deionized water using the co-precipitation method. The resulting slurry was then transferred to a Teflon-lined autoclave and aged at 100 °C for 12 h. Finally, the products

were centrifuged and washed several times with water and ethanol followed by calcination at different temperatures in air. The sensing performances of the sensors calcined at various temperatures were investigated against ethanol. They reported that the sensor calcined at 1000 °C exhibited a better response to ethanol compared to other samples calcined at 600 and 800 °C. Additionally, the sample calcined at 1000 °C presents fine repeatability and good selectivity for ethanol. They reported that the improved performance of the sample calcined at 1000 °C could be attributed to better crystallinity of ZnAl_2O_4 than that calcined at 800 or 600 °C, respectively. Liu et al. [55] reported the preparation of a $\text{ZnO}/\text{ZnFe}_2\text{O}_4$ composite sensor with a hexagonal nanostructure via a calcination method. For this firstly, $\text{Zn}_2\text{Fe-LDH}$ with sodium dodecyl sulfate (SDS) was synthesized using a combination of co-precipitation and hydrothermal methods at 100 °C for 10 h. The resulting powder was filtered and washed several times with water and dried at 60 °C for 12 h in electric oven. Finally, the $\text{ZnO}/\text{ZnFe}_2\text{O}_4$ composite sensor was calcined at various temperatures to obtain the desired hexagonal structure as shown in Figure 4a. They focused on the typical problem tackled by metal oxide sensors, which usually need high operating temperatures around 200 °C. They found that by applying light irradiation, the operating temperature could be significantly lowered (Figure 4a). They observed that under light illumination, the gas-sensing performance of the $\text{ZnO}/\text{ZnFe}_2\text{O}_4$ composite, calcined at 600 °C, was significantly enhanced for detecting triethylamine (TEA) at a reduced operating temperature of 80 °C. The gas-sensing performance of the $\text{ZnO}/\text{ZnFe}_2\text{O}_4$ sensor towards TEA was investigated through independent resistance change measurements conducted at an operating temperature of 80 °C. In a typical gas-sensing experiment, liquid TEA was first injected followed by gas evaporation. The chamber was then irradiated with a light wavelength of $\lambda > 320 \text{ nm}$ and a power of $150 \text{ mW}/\text{cm}^2$. It can be seen from Figure 4b that the $\text{ZnO}/\text{ZnFe}_2\text{O}_4$ sensor exhibited a gradual increase in R_0/R_t upon light irradiation for varying TEA concentrations ranging from 5 to 1000 ppm. Initially, the resistance (R_t) for TEA decreased means R_0/R_t continues to increase upon introducing TEA gas into the chamber. This might be the chemical reaction of TEA with surface absorbed oxide ions (O_2^-) resulting in electron transfer back to the sensor, leading to a decrease in resistance (R_t). However, the sensor's response (R_0/R_t) decreases to the initial value after switching off the light and being exposed to air (Figure 4b). As shown in Figure 4c, a comparative evaluation of the $\text{ZnO}/\text{ZnFe}_2\text{O}_4$ sensor calcined at 600 °C was investigated to assess its performance in the presence and absence of light illumination, while maintaining all other experimental conditions the same. The results indicate that a noticeable difference in response across all TEA concentrations clearly show that light illumination significantly enhances the gas-sensing performance of the sensor. Hong et al. reported the synthesis of hierarchical flower-like Ni-Al-LDH and Ni-Fe-Al-LDH intercalation sensors using a facile one-step hydrothermal approach for NO_x sensing at room temperature (RT) [56]. In another study, Sun et al. reported the preparation of three-dimensional hierarchical flower-like a Mg-Al-LDH sensor using a simple hydrothermal method for sensing NO_x at RT [57]. They reported that the hierarchical porous nanostructure provides natural channels for efficient and fast carrier transportation, resulting in fast response and recovery times to 100 ppm NO_x at RT. Qu et al. [58] designed a porous double-shelled nanocage $\text{ZnO}/\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}$ sensor using a unique metal-organic framework route. They reported that the designed $\text{ZnO}/\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}$ sensor demonstrates high sensitivity and selectivity towards xylene compared to pristine ZnO nanocages. The outstanding gas-sensing performance of the $\text{ZnO}/\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}$ sensor is ascribed to the large surface area, high porosity, and synergistic effect of ZnO and $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}$.

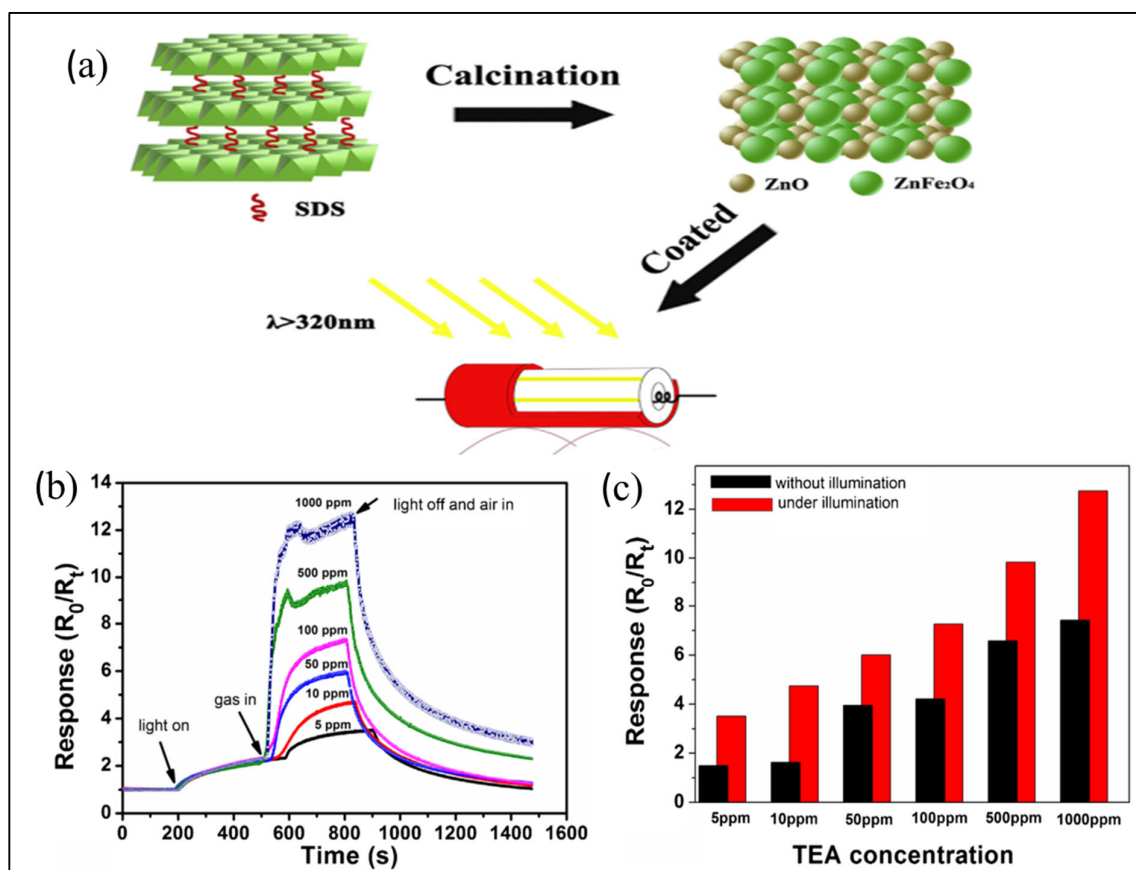


Figure 4. (a) Pictorial presentation for synthesis of gas sensor based on ZnO/ZnFe₂O₄ composite. (b) Sensing response towards various TEA concentrations as function of time under light illumination at working temperature of 80 °C. (c) Comparative sensing response for TEA in presence and absence of light illumination at 80 °C [55].

Zhang et al. developed a highly sensitive, durable, and active sensor for sensing VOCs using a combination of p-n heterojunction [59]. The sensor was prepared using a facile thermal conversion of hierarchical CoTi LDHs precursors at 300–400 °C [59]. The prepared optimized mesoporous hierarchical Co₃O₄–TiO₂ nanocomposite demonstrated excellent sensing performance for toluene and xylene at an operating temperature of 115 °C. In another study, Kang et al. designed a novel Ni–Al–LDH sensor using a hydrothermal method for detecting different gases including ozone, H₂, NO₂, and ethanol [60]. Their investigations indicate that the synthesized Ni–Al–LDH sensor exhibited exceptional and selective detection of ozone, even in the presence of other interfering gases such as H₂, NO₂, and C₂H₅OH. Moreover, the developed sensor exhibited an outstanding response and recovery times, along with excellent selectivity and stability towards ozone. A phenomenological sensing performance of the chlorine intercalated LDH sensor was investigated by Polese et al. [48] for five different VOCs (CO, CO₂, NO, NO₂, and CH₄). Their study highlighted that LDHs are a class of nanomaterials that exhibit outstanding properties such as large surface area, high porosity, and excellent chemical interaction capabilities with a wide range of analytes. Additionally, these materials are easy to synthesize and tailor for sensing performance. In another study, Qu et al. [61] used a MOF-based synthesis approach to develop a Co₃O₄/NiCo₂O₄ double-shelled nanocage for acetone sensing. To design a MOF-derived Co₃O₄/NiCo₂O₄, zeolite imidazolate framework-67 (ZIF-67) was first synthesized. Afterwards, the synthesized ZIF-67 was then dispersed into an ethanol solution containing Ni(NO₃)₂·6H₂O during continuous stirring. Finally, the mixture was annealed at 350 °C for 2 h, as shown in Figure 5a. To confirm the morphology, crystallinity,

and elemental composition of the synthesized $\text{Co}_3\text{O}_4/\text{NiCo}_2\text{O}_4$ double-shelled nanocages sensor, SEM, TEM, XRD, and elemental analyses were carried out. It could be seen from the XRD analysis (Figure 5b,e) that the synthesized sensor is pure and crystalline in nature, which is consistent with the reported literature. The scanning electron microscopic (SEM) image of ZIF-67 and Co_3O_4 are displayed in Figure 5c and 5d, respectively. Surface analysis exhibits the formation of rhombo-dodecahedron-shaped structures with an average size of 500 nm. The SEM results for ZIF-67/Ni-Co LDH and $\text{Co}_3\text{O}_4/\text{NiCo}_2\text{O}_4$ are depicted in Figure 5f and 5h, respectively. On the other hand, transmission electron microscopic (TEM) images of the ZIF-67/Ni-Co LDH and $\text{Co}_3\text{O}_4/\text{NiCo}_2\text{O}_4$ are presented in Figure 5g and 5i, respectively. The SEM results exhibited the formation of a rougher surface texture of $\text{Co}_3\text{O}_4/\text{NiCo}_2\text{O}_4$ compared to the ZIF-67/Ni-Co LDH.

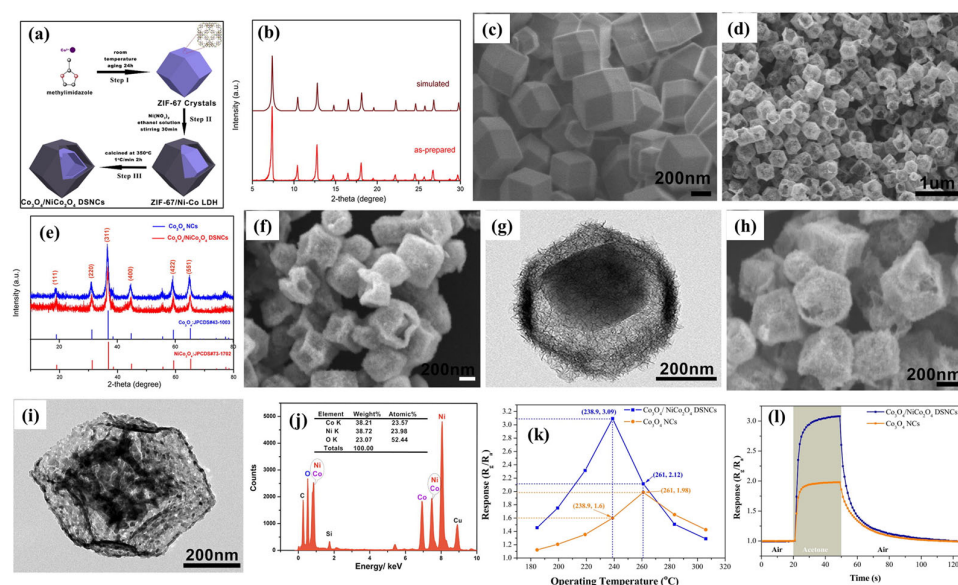


Figure 5. (a) Schematic illustration for synthesis of $\text{Co}_3\text{O}_4/\text{NiCo}_2\text{O}_4$ double-shelled nanocages. (b) XRD pattern of synthesized ZIF-67 and simulated sample. SEM image of (c) ZIF-67 and (d) Co_3O_4 . XRD patterns of (e) Co_3O_4 and $\text{Co}_3\text{O}_4/\text{NiCo}_2\text{O}_4$. SEM (f,g) TEM images of ZIF-67/Ni-Co LDH. SEM (h,i) TEM images of $\text{Co}_3\text{O}_4/\text{NiCo}_2\text{O}_4$. EDX spectrum (j) of $\text{Co}_3\text{O}_4/\text{NiCo}_2\text{O}_4$. (k) Sensing performance of Co_3O_4 and $\text{Co}_3\text{O}_4/\text{NiCo}_2\text{O}_4$ sensor for 100 ppm acetone as function of operating temperature, and (l) response of Co_3O_4 and $\text{Co}_3\text{O}_4/\text{NiCo}_2\text{O}_4$ sensor for 100 ppm acetone at 238.9 °C. Reproduced with permission [61].

TEM analysis further confirmed the formation of a double-shelled nanocage structure. In order to confirm the elemental composition of the $\text{Co}_3\text{O}_4/\text{NiCo}_2\text{O}_4$ double-shelled nanocage sensor, Energy Dispersive X-ray Spectroscopic (EDX) analysis was carried out, which indicated the presence of Co, Ni, and O elements, as shown in Figure 5j. The sensing performance of the synthesized sensors was examined for acetone at different temperatures to study the impact of operating temperature, a key parameter influencing the performance of semiconducting sensors. Therefore, the sensing performance of the $\text{Co}_3\text{O}_4/\text{NiCo}_2\text{O}_4$ sensor was tested for 10 ppm acetone as a function of temperature to obtain the optimum temperature, as shown in Figure 5k. The optimum temperature for $\text{Co}_3\text{O}_4/\text{NiCo}_2\text{O}_4$ was found to be 238.9 °C. Therefore, the rest of the sensing experiments were performed at the optimum temperature, which indicated that the $\text{Co}_3\text{O}_4/\text{NiCo}_2\text{O}_4$ sensor revealed fast response and recovery times for 100 ppm acetone (Figure 5l).

Li et al. [62] demonstrated the development of core-shell of polystyrene spheres combined with cobalt-based LDHs for the detection of ethanol. In another study, Qu et al. [63] fabricated the porous hollow $\text{Co}_3\text{O}_4/\text{ZnCo}_2\text{O}_4$ nanostructure composite using

a self-sacrificing template method for acetone sensing. Zhang et al. [64] established an ultra-sensitive and selective NO_x gas sensor with an ultra-low detection limit by prepared expanded graphite/NiAl LDH nanowires using a hydrothermal method. It was reported in the literature that nanocomposites made up of combinations of organic and inorganic materials have attracted significant interest in the field of sensors due to the synergistic effects between respective components. In this contest, LDHs have emerged as promising host-matrices, providing a confined and stable environment for uniform composite formation. In this contest, Qin et al. proposed that LDH-based sensing devices could be a possible solution due to their exclusive structural properties [65]. To explore this possibility, a 3D nanocomposite of polyaniline (PANI) and ZnTi-LDHs was developed using a hydrothermal method, where PANI was uniformly joined into the ZnTi-LDH matrices. The optimized ZnTi-LDH sensor exhibited outstanding sensing performance, confirmed by its high response and notable long-term stability at RT. In order to detect the nitrogen dioxide (NO_2) gas, which plays a key role for the formation of photochemical smog, PM2.5, acid rain, and ozone formation in the atmosphere, an LDH-based sensor was developed by Liu et al. [66]. They used a simple one-step hydrothermal method for the preparation of a 3D flower-like CoAl-LDH nanocomposite sensor. To improve gas sensitivity of a 3D flower-like CoAl-LDH sensor, fluoride ion was used as a functional template agent. The functionalized optimized sensor demonstrated excellent sensing performance towards NO_2 detection, as confirmed by ultrafast response and recovery times. Zhang et al. [67] reported the synthesis of 3D flower-like NiZnAL ternary sensor with multi-metal oxide and ultra-thin porous nanosheets for the detection of NO_x . In another study, a 3D flower-like Zn and Al sodium dodecyl sulfate-LDH sensor intercalated by anions was synthesized using a hydrothermal method. In this system, urea was used a precipitant and sodium dodecyl sulfate as a functional templating agent for the detection of NO_2 [68]. Lang et al. [69] highlighted that an ideal gas sensor must exhibit fast response and recovery times, along with excellent repeatability and long-term stability, to contribute effectively to environmental protection. To fulfill this purpose, they developed the 3D flower-like layered Ni-Co-LDH composite sensor using a simple hydrothermal method. In a typical procedure, $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in ratios of 3:1, 1:1, and 1:3 were dissolved in distilled water along with hexamethylenetetramine (HMT) under continuous stirring for 4 h. The resulting suspension then transferred to Teflon-lined autoclave and was heated at 95°C for 12 h. the obtained powder was washed several times with water and ethanol and dried at 60°C in oven. The samples were denoted as Ni3Co1, Ni1Co1, and Ni1Co3 (Figure 6a).

In order to confirm the flower-like structure of the sensors, SEM analysis was conducted. The authors found that synthesized materials are composed of uniform 2D sheets making a 3D flower-like structure. Moreover, the nanosheets were interconnected and intertwined similar to petals and almost perpendicular to the outer surface, providing a diameter of 3–4 μm . This may be attributed to the fact that during hydrothermal synthesis, the nanosheets gradually deposited on each other, producing a shape similar to a flower-like structure. The performance of the synthesized 3D flower-like NiCo-LDH sensor was investigated to detect NO_2 gas at RT. Figure 6b exhibits the sensing response of the optimized Ni1Co1 sensor against different concentrations (0.01–100 ppm) of NO_2 at RT. As shown in Figure 6b, as the concentration of NO_2 increases, the reaction of the Ni1Co1 sensor also increases. The lowest concentration detected by Ni1Co1 and Ni3Co1 was found to be 0.1 ppm, whereas the lowest concentration detected by Ni1Co3 was found to be 0.05 ppm. Figure 6c exhibits the response and recovery times of the Ni3Co1, Ni1Co1, and Ni1Co3 sensors towards NO_2 sensing. Among all the sensors, Ni1Co1 indicates the highest sensing response and recovery times for NO_2 sensing, as shown in Figure 6c. The calibration curve for Ni1Co1 was obtained from the logarithm of sensor response ($\log S$)

and the logarithm of gas concentration ($\log \text{NO}_2$). It can be seen from Figure 6d that the calibration curve shows a linear relationship. They also measure the stability and selectivity, which are very important parameters for sensors to be used at an industrial level. The stability of the Ni1Co1 sensor was performed by repeatability using the sensor for 14 consecutive cycles, as shown in Figure 6e. The selectivity of the sensor was investigated in the presence of other interfering gases such as CH_4 , CO , NH_3 , H_2S , H_2 , and NO_2 at the same experimental conditions at RT. As shown in Figure 6f, the optimized Ni1Co1 sensor is more selective towards NO_2 gas. Moreover, the long-term stability of the Ni1Co1 sensor was subjected to 100 ppm NO_2 every 5 days at RT. As shown in Figure 6g, after 60 days of stability measurement, the sensor response is excellent. The performance of the gas sensors based on LDH materials are shown in Table 1.

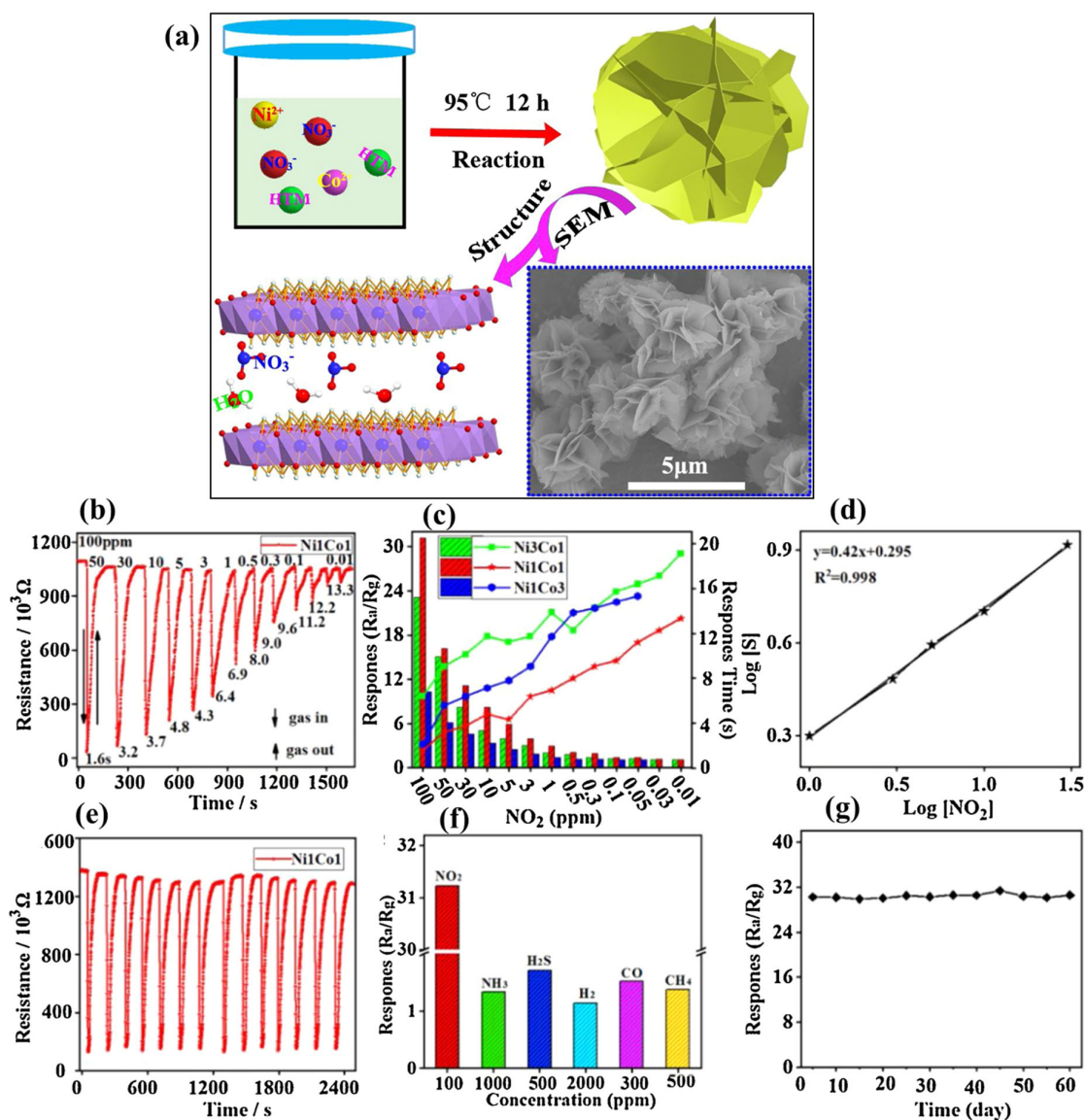


Figure 6. (a) Schematic illustration of preparation of 3D flower-like NiCo-LDH gas sensor. (b) Response and recovery times of optimized Ni1Co1 sensor towards varying concentrations of NO_2 gas, (c) gas response and recovery times of NiCo-LDH sensor at different molar concentration for NO_2 at RT, (d) NO_2 calibration curve of Ni1Co1 composite, (e) dynamic response of Ni1Co1 sensor 100 ppm NO_2 for 14 cycles, (f) selectivity of Ni1Co1 sensor in presence of different interfering gases, and (g) stability of Ni1Co1 sensor for 100 ppm NO_2 for 60 days. Reproduced with permission [69].

Table 1. Sensing activity and comparison of gas sensors based on LDH [70–85].

Sensors	Analytes	Detection Range (ppm)	Response Time (min)	Recovery Time (min)	Response (S)	References
Ni-Cr-AL-LDH	Acetone, ethanol	500–5000	6, 1	3, 1	$R_g - R_a / R_a \times 100$	[70]
NiO/NiGa ₂ O ₄ -LDH	Toluene	0.1–5	4.7	6.3	R_g / R_a	[71]
ZnAl–Cl, ZnFe–Cl, ZnAl–NO ₃ , and MgAl–NO ₃ -LDH	Acetone, ethanol, ammonia, chlorine	Fixed concentrations of 10% of saturated vapors	3.1, 3.1, 3.9, 2.3	2.3, 4.05, 3.7, 3.2	$R_g - R_a / R_a \times 100$	[72]
CuCr-, ZnCr-, and ZnTi-LDH	Methanol, ethanol, acetone	1–40	---	0.61, 0.71, 1.48	$R_g - R_a / R_a$	[73]
ZnTi-LDHs/rGO	NO ₂	0.05–20	0.033	0.86	$R_g - R_a / R_a \times 100$	[74]
CoNi- LDH on GO (GO@LDH)	2,4-Dimethyl benzaldehyde	10–300 µg/L	----	-----	-----	[75]
zinc–chromium-LDH	SO ₂	0.1–100	0.33	5.12	$R_g - R_a / R_a \times 100$	[76]
zinc–chromium-LDH	SO ₂	0.1–100	0.33	2.8	$R_g - R_a / R_a \times 100$	[77]
NiCo-LDH	Methylene Blue, Methylene orange	0.005–10	4.5, 2.2	---	$R_g - R_a / R_a$	[78]
Zn–Al-LDH	SO ₂	-	-	-	R_{gas} / R_{air}	[79]
Cu/Fe -LDH	Imidacloprid	-	-	-	$R_g - R_a / R_a$	[80]
(CeO ₂)x/Ni-Al LDH	Methanol, ethanol, and acetone	25–300	1.01, 0.666, 0.42	3.45, 0.91, 2.13	R_{air} / R_{gas}	[81]
CeO ₂ /Ni–Al-LDH	Ethanol	25–300	0.666	1.27	R_{air} / R_{gas}	[82]
Ni-Al LDH	Methanol, ethanol, and acetone	20- 300	0.2, 0.83, 0.33	2.25, 1.95, 1.9	R_{air} / R_{gas}	[83]
(CeO ₂)x/Ni-Al-LDH	Methanol, ethanol, and acetone	25–300	1.01, 0.666, 0.41	3.45, 0.92, 2.13	R_{air} / R_{gas}	[84]
ZnCo-LDHs	Ethanol	50–250	---	----	R_g / R_a	[85]

3. LDH-Based Electrochemical Sensors

Electrochemical sensors are promising voltammetric sensing technology for the detection of biomolecules, hazardous compounds, drugs, etc. In this report, we have summarized the progress in LDH-based materials for the sensing of toxic and hazardous substances. It is known that endocrine-disrupting substances or compounds such as bisphenol A (BPA; 2,2-bis(4-hydroxyphenyl)propane) are one of the environmental pollutants that may have negative impacts on aquatic life and environment. BPA is widely used in various products and can be easily leached to water, food, or soil, and it can further migrate to aquatic organisms and human bodies [86]. BPA may be responsible for various diseases such as sexual dysfunction, neurotoxicity problems, cardiovascular, lower sperm quality, and various cancers such as breast cancer. Thus, determination and monitoring of BPA is necessary; Zhan et al. [86] reported the preparation of 1-aminopropyl-3-methylimidazolium tetrafluoroborate-modified zinc-aluminum (Zn-Al) LDH (ILs-LDH) by co-precipitation method. The authors found that ILs-LDH has a disk-like surface morphology by SEM-based analysis. The glassy carbon electrode, i.e., GCE, was coated with ILs-LDH towards the sensing of BPA. The authors used cyclic voltammetry (CV), differential pulse voltammetry (DPV), and electrochemical impedance spectroscopy (EIS) techniques for the sensing of BPA. The ILs-LDH-coated GCE exhibits improved electrochemical performance for the sensing of BPA and an acceptable linear range of 0.02 to 3 µM with a limit of detection (LOD) of 4.6 nM and excellent selectivity, stability, and reproducibility. The ILs-LDH-coated GCE shows that the proposed BPA sensor can be employed for real sample studies

with a recovery range of 94.9% to 102%. Hydrogen peroxide (H_2O_2) has been known as a ubiquitous compound in the processes and as an intermediate product for biological reactions and aerobic organisms. H_2O_2 is also used in various industrial applications such as textile, medical, packaging, and waste water treatment. Despite various applications, H_2O_2 has negative influences on human and aquatic life. Thus, the sensing of H_2O_2 is of great significance. Heli et al. [87] synthesized cobalt-aluminum (CoAl) LDH nanoshells integrated multiwalled carbon nanotube (MWCNT) composites via a reflux assisted method. The synthesized CoAl-LDH/MWCNT composite was characterized by XRD, which revealed the presence of a rhombohedral structure of LDH. The formation of the composite was also confirmed by XRD and suggested that LDH material was successfully deposited on MWCNTs. The SEM and TEM analyses indicated the formation of nanoshell-integrated MWCNTs. The carbon paste electrode (CPE) was modified with a CoAl-LDH/MWCNTs composite (denoted as MCPE) and adopted as a non-enzymatic H_2O_2 sensor. Chronoamperometry (CA) and CV analyses show excellent electrochemical behavior of the MCPE and stability with good selectivity in the presence of various interfering substances. Heavy metals are major threats for human beings and the environment due to their highly toxic nature. Mercury (Hg) is one of the heavy metals and possesses hazardous properties and it may be responsible for various health problems such as kidney failure, lung diseases, and nervous system disorders. Thus, a novel Hg sensor was developed using mercaptocarboxylic acid (thioglycolic acid; TGA) intercalated magnesium (Mg)-Al LDH as the electrode modifier. An anion exchange method was adopted for the preparation of Mg-Al-TGA LDH, which was further coated on GCE and explored as a Hg sensor. The authors achieved an LOD of 0.8 nM by employing square wave anodic stripping voltammetry (SWASV) as a sensing technology and Mg-Al-TGA LDH/GCE as the working electrode [88]. In another report [89], co-precipitation and hydrothermal routes methods were used for the fabrication of copper oxide (CuO)@manganese (Mn)Al LDH. The fabrication mechanism for the formation of CuO@MnAl is depicted in Figure 7a. The GCE was modified with the prepared CuO@MnAl and this electrode (CuO@MnAl/GCE) was used for H_2O_2 sensing applications by employing amperometry analysis. Figure 7b shows the amperometric responses of CuO@MnAl/GCE for various concentrations of H_2O_2 . The potential for the sensing of H_2O_2 was fixed as -0.85 V. It was observed that current responses increase with increasing concentrations of H_2O_2 and a calibration plot between the current values and concentrations of H_2O_2 is depicted in Figure 7c. The current response was linearly increased, and authors achieved a decent LOD of $0.126 \mu\text{M}$ and a linear range of $6 \mu\text{M}$ to 22 mM was obtained. Selectivity is one of the challenging tasks; therefore, the authors studied the selectivity of CuO@MnAl/GCE for H_2O_2 via amperometry analysis. It is clear from Figure 7d that CuO@MnAl/GCE has excellent selectivity for H_2O_2 in the presence of various interfering substances. The long-term stability and reproducibility of CuO@MnAl/GCE were also checked by measuring the current response with 5 mM H_2O_2 , as shown in Figure 7e. The authors observed that CuO@MnAl/GCE has acceptable stability for 30 days and decent reproducibility (inset of Figure 7e).

Li et al. [90] proposed the fabrication of ternary LDH electrode material for heavy metal ion sensing applications. An iron(Fe)/magnesium(Mg)/nickel(Ni) ternary LDH was synthesized via a co-precipitation method. The fabricated ternary Fe/Mg/Ni LDH was deposited on GCE and the SWASV technique was utilized for the determination of lead (Pb(II)). A remarkably good LOD of $0.032 \mu\text{M}$ was obtained for Pb(II) sensing. Excellent selectivity and stability was also obtained for Fe/Mg/Ni LDH-modified GCE towards Pb(II) sensing. Polycyclic aromatic hydrocarbons, i.e., PAHs, are globally known as environmental contaminants and receive significant concern because of their higher toxicity and bioaccumulative properties [91]. Cadmium (Cd)/Al LDH was obtained using a green

electrochemical synthetic method. The XRD results revealed that prepared Cd/Al LDH has a poor crystalline nature while SEM analysis suggested the presence of a regular and small sheet structure. Cd/Al LDH further coated GCE and its sensing activity for anthracene was studied by employing EIS, CV, and DPV methods. The constructed Cd/Al/GCE electrode shows an LOD of 0.5 fM with decent selectivity. The NiFe LDH was fabricated on a nickel foam electrode using a hydrothermal method [92]. Their phase purity, formation, and crystalline nature were evaluated by XRD while SEM analysis was adopted for the study of morphological characteristics. It can be seen that NiFe LDH has decent crystalline properties with good phase purity and XRD results confirmed the growth of NiFe LDH on the Ni foam electrode (Figure 8a). The SEM-based results clearly show that NiFe LDH has hierarchical spheres (Figure 8b). The fabricated NiFe LDH-based nickel foam electrode was employed for H₂O₂ sensing. The NiFe LDH-based nickel foam electrode shows that an LOD of 0.5 µM was achieved using an amperometric method. This proposed electrode also shows good selectivity in the presence of potassium chloride, sodium chloride, dopamine, glucose, and uric acid (Figure 8c).

In a previous study, MgFe LDH was also reported by employing a one-step hydrothermal method [93]. MgFe LDH was immobilized on graphene sheets and the resulting composite (MgFe LDH/graphene/GCE) demonstrated higher electrochemical activity, which may be ascribed to the presence of synergism in the fabricated MgFe LDH/graphene composite. The authors investigated simultaneously the determination of Cd (II) and Pb (II). The LOD of 5.9 nM and 2.7 nM were reported for the detection of Cd (II) and Pb (II), respectively. The MgFe LDH/graphene composite-modified GCE also displayed good recovery for real sample studies in lake water. Zhan et al. [94] fabricated a novel BPA sensor by utilizing exfoliated Ni₂P/Al LDH as an electrode modifier. Ni₂P/Al was prepared by using l-asparagine as a pre-intercalator and synthesized Ni₂P/Al LDH was exfoliated, as demonstrated in Figure 9. The exfoliated Ni₂P/Al LDH-modified GCE was explored as a BPA sensor and the DPV technique was utilized as voltametric approach towards the determination of BPA. The mechanism of BPA sensing is illustrated in the DPV graph displayed in Figure 9. This proposed sensor shows a linear range of 0.02 to 1.51 µM, an LOD of 6.8 nM, and good selectivity, reproducibility, and stability.

Nitrite is widely used in various industries including the food industry, agriculture (as a fertilizer), and corrosion science (as an inhibitor). Unfortunately, nitrite has toxic properties and can interact with amines to transform to carcinogenic N-nitrosamines. Additionally, it can have negative effects if it is present in drinking water with higher concentrations. Mg/Al LDH was prepared on carbon paper (CP) using a hydrothermal method. Surface morphological investigations revealed that Mg/Al LDH has a flower-like structure and grew on CP. This electrode demonstrated decent electrochemical performance for the sensing of nitrite with decent selectivity and stability [95]. Metronidazole (MNZ; 2-(2-methyl-5-nitroimidazole-1-yl) ethanol) is the derivative of nitroimidazole which is used for the treatment of protozoal diseases such as giardiasis and trichomoniasis [96]. MNZ may cause some health-related issues such as genotypic, mutagenic, genotoxic, and carcinogenic side effects. Thus, Vilian et al. [96] fabricated a novel MNZ sensor by using NiCo LDH as the electrode material and the authors prepared NiCo LDH on carbon nanofibers (CNFs) via a hydrothermal method (Figure 10a). SEM analysis revealed that CNF–NiCo-LDH with a dense hierarchical nanowire structure was successfully anchored on the surface of CNFs (Figure 10b). The synthesized CNF–NiCo-LDH-based electrode was used as a MNZ sensor by using a DPV-based sensing method. The authors observed that the current value increases with increasing concentrations of MNZ (Figure 10c) and a calibration plot between the current value and concentration of MNZ (Figure 10d) indicated that current values linearly increase and a decent linear range of 3 to 57 nM was obtained with an LOD

of 0.13 nM. The authors also stated in this article that the proposed CNF–NiCo–LDH–GCE for the sensing of MNZ has excellent anti-interfering properties and can be used for the selective detection studies of MNZ in pharmaceutical industries.

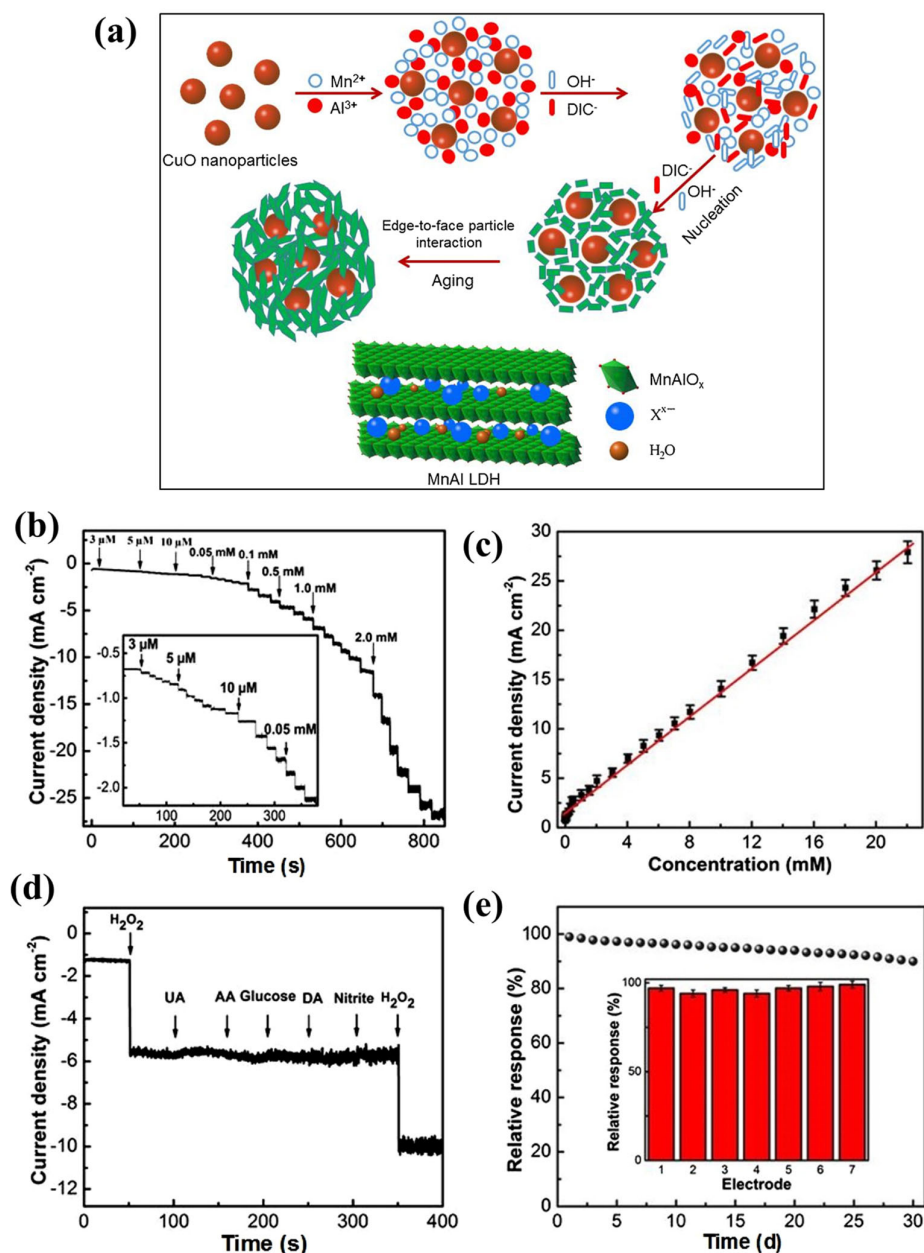


Figure 7. (a) Schematic picture for synthesis mechanism of CuO@MnAl NSs. (b) Amperometric response of CuO@MnAl/GCE with different concentrations of H₂O₂ at −0.85 V and (c) calibration curve between current versus concentrations. (d) Selectivity test and (e) variation in current responses for 5 mM H₂O₂ with time (s). Inset shows current values for seven electrodes for 5 mM H₂O₂. Reproduced with permission [89].

Wang et al. [97] fabricated zeolitic imidazole framework (ZIF-67)/LDH nanosheets (NSs) for the sensing of α -naphthol and β -naphthol. Synthetic protocols for the preparation of ZIF-67/LDH composites can be seen in Figure 11a. XRD results show the presence of good phase purity and a decent crystalline nature of the obtained samples (Figure 11b). ZIF-67/LDHNS (Co/Al LDH nanosheets) was coated on GCE and electrochemical studies revealed the excellent electrochemical behavior of the ZIF-67/LDHNS-modified GCE. DPV results demonstrated decent LOD of 62 and 94 nM for the sensing of α -naphthol and

β -naphthol, respectively. The decent linear range of 0.3 to 150 μ M with high stability, reproducibility, and selectivity were also highlighted by the authors. It was observed that LDHNS@ZIF-67/GCE may be used as an efficient sensor for the simultaneous determination of α -naphthol and β -naphthol (Figure 11c) with good selectivity.

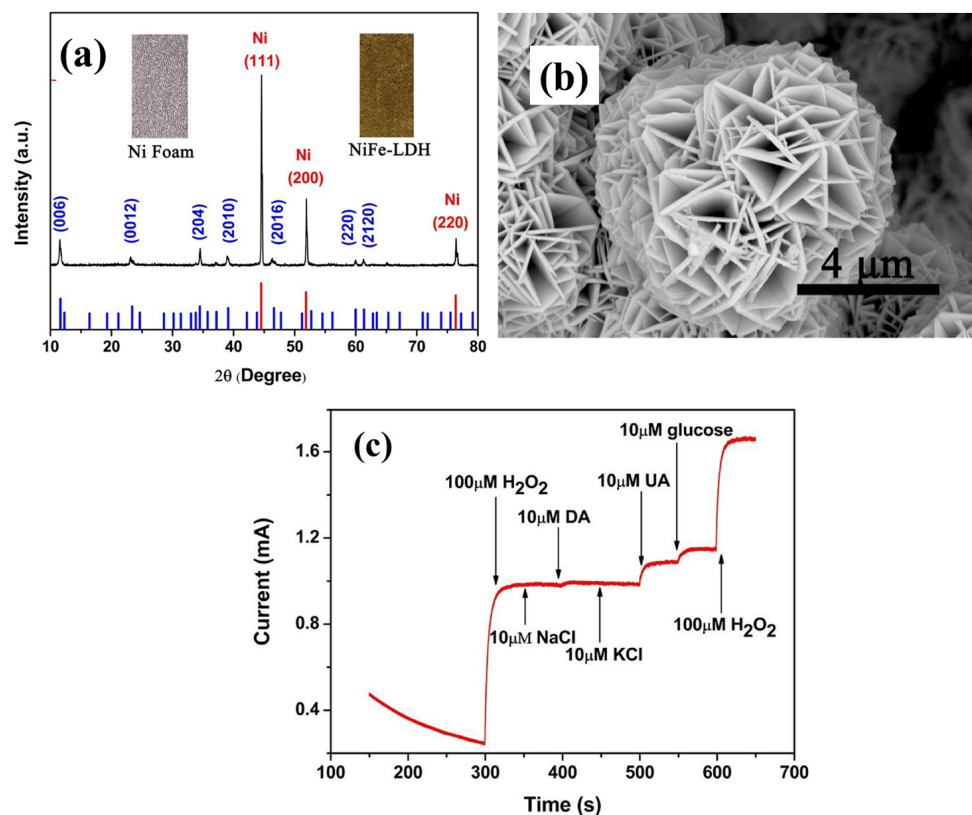


Figure 8. (a) XRD and (b) SEM results for Cd/Al LDH. (c) Selectivity test of Cd/Al LDH/GCE for H_2O_2 . Reproduced with permission [92].

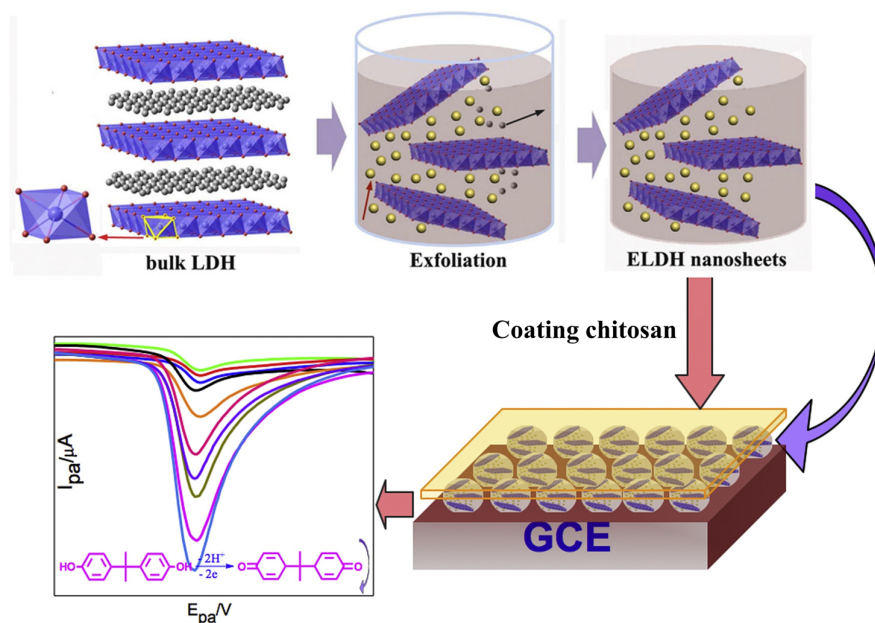


Figure 9. Schematic illustration of fabrication of BPA sensor. Reproduced with permission [94].

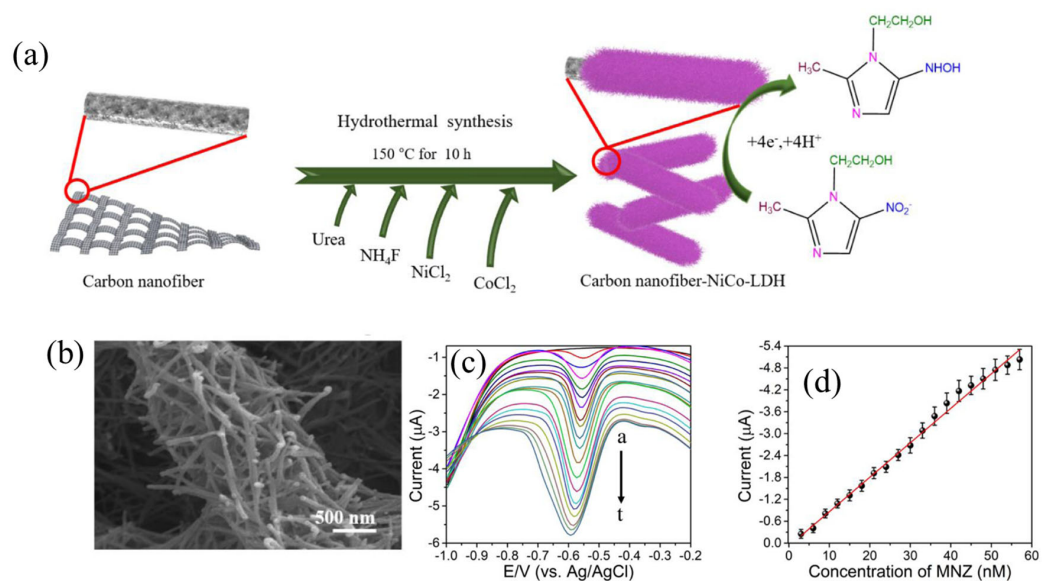


Figure 10. (a) Schematic graph shows formation of CNF-NiCo LDH and SEM image (b) of CNF-NiCo LDH. (c) DPVs of CNF-NiCo LDH/GCE for MNZ sensing under various concentrations and (d) calibration curve of current versus concentrations of MNZ. Reproduced with permission [96].

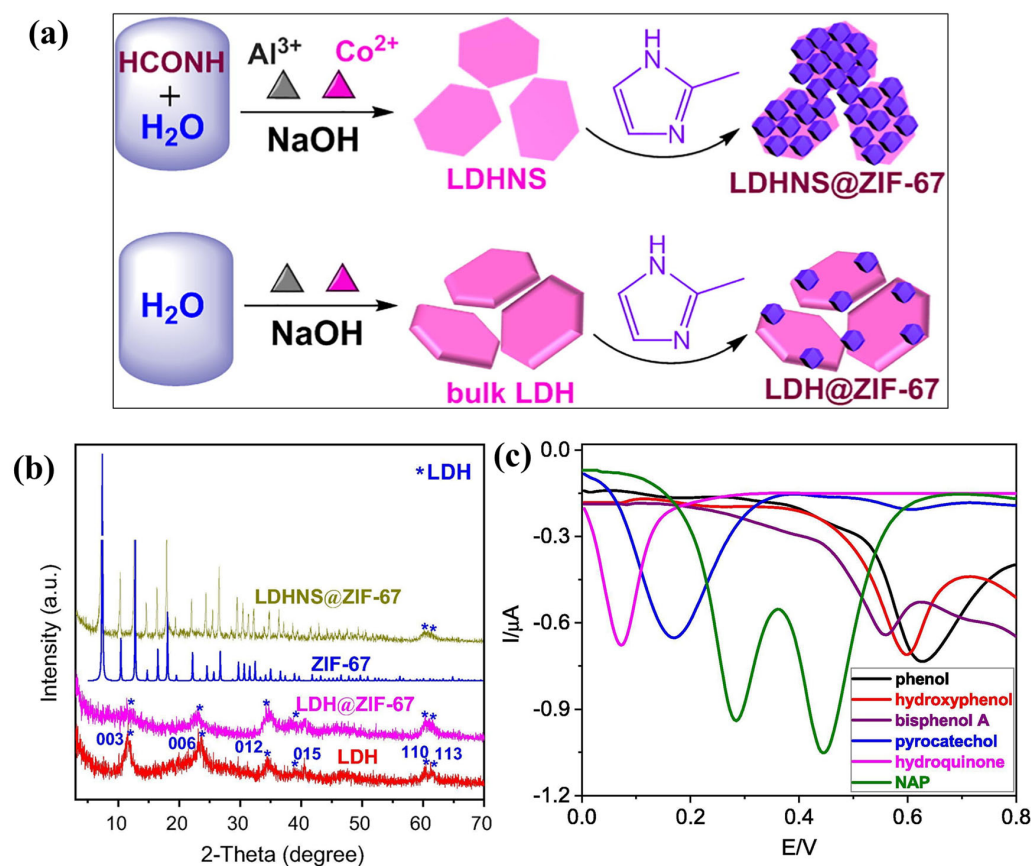


Figure 11. (a) Schematic illustration of formation of LDHNS@ZIF-67. (b) XRD patterns of LDH, ZIF-67, LDH@ZIF-67, and LDHNS@ZIF-67. (c) Selectivity test using DPV method. Reproduced with permission [97].

In a different report [98], a unique reaction–diffusion framework (RDF) was adopted for the formation of CoAl-LDHNS (CoAl-LDHNS@ZIF-67). The proposed CoAl-LDHNS@ZIF-67/GCE shows LOD of 54 and 82 nM for the sensing of α -naphthol and β -naphthol, respectively, with linear range of 0.3 to 150 μM . It was believed that the presence of hierarchically

structured functional LDH materials may exhibit better electrochemical activity and can be explored for sensing applications. Joseph et al. [99] reported the formation of a novel FeMn LDH entrapped tungsten carbide (WC) composite for the determination of diphenylamine (DPA) via a hydrothermal method. An FeMn LDH/WC-based electrode showed a wide linear range of 0.01 to 183.34 μM with an LOD of 1.1 nM and good selectivity via the DPV method. In other work [100], a NiCo LDH/WC composite (WC@NiCo-LDH) was also formed by employing the simple and efficient hydrothermal method. The WC@NiCo-LDH-modified electrode was able to maintain excellent electrochemical activity for the sensing of norfloxacin (NRF) using CV, DPV, and amperometric methods. The authors reported LOD of 0.005 μM and 0.002 μM using DPV and amperometry methods, respectively. The linear range of 0.02–83.4 μM and 0.002–346 μM were reported for the DPV- and amperometry-based methods, respectively. This improved performance was attributed to the high surface area, electrical conductivity, and synergism between the WC and LDH materials. Karupiah et al. [101] reported the preparation of two-dimensional (2D) hydrogen ammonium ZnMo LDH (AZnMo LDH) via a co-precipitation method and integrated it with 1D vapor grown carbon fiber (VGCF) using an ultrasonication method. AZnMo-LDHs@VGCF was explored as the sensing material for the determination of dimetridazole (DMZ) and an LOD of 0.021 μM , sensitivity of $1 \mu\text{A} \mu\text{M}^{-1} \text{cm}^{-2}$, and linear range of 0.25 to 520.75 μM were obtained using the DPV method.

Oxygen vacancy-based Co-Al LDH (OV-LDH) was integrated with hydroxylated MWCNTs (H-MWCNTs) by a simple self-assembly approach, as demonstrated in Figure 12 [102]. It is believed that O-vacancies may enhance the electrochemical performance of Co-A LDH and the presence of H-functional groups in H-MWCNTs can enable the composite to form H-bonds with O-vacancies and improve the stability. The resulting OV-LDHs/H-MWCNT composite-based electrode showed excellent LOD of 0.074 and 0.076 μM for hydroquinone, i.e., HQ, and catechol, i.e., CC, respectively. The proposed sensor also has excellent recovery in industrial waste water and suggested its potential for practical applications.

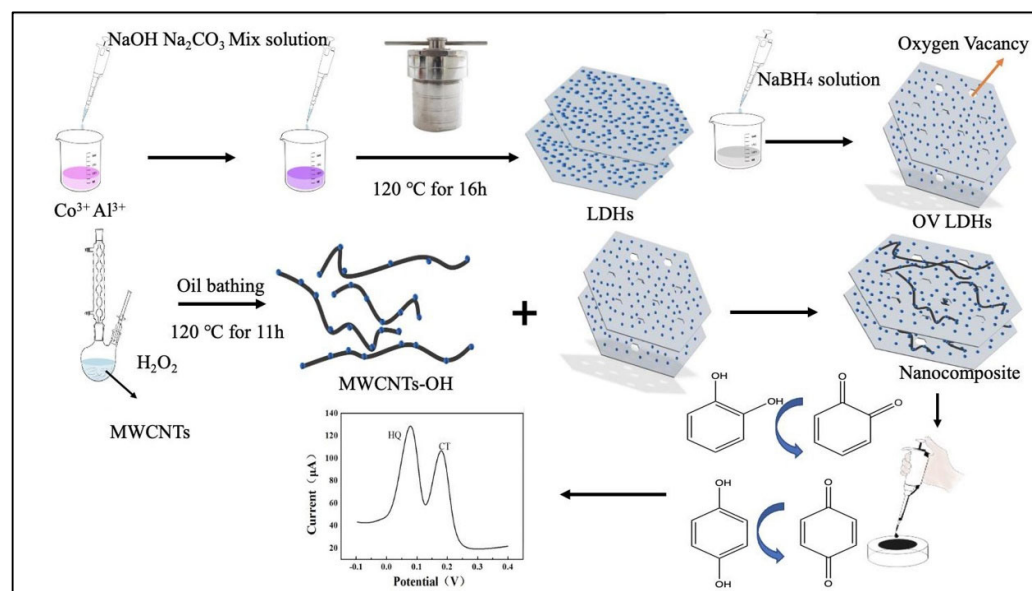


Figure 12. Schematic picture for formation of OV LDH/H-MWCNTs and their application for CC and HQ sensing. Reproduced with permission [102].

The NiCo LDH/functionalized halloysite nanotubes (NiCo-LDH/F-HNTs) were proposed as efficient electrode modifiers by Kokulnathan et al. [103] and showed an LOD of 0.002 μM , linear range of 0.01 to 33.4 μM , and sensitivity of $13.0 \mu\text{A} \mu\text{M}^{-1} \text{cm}^{-2}$ towards

the determination of parathion (PT). It was stated that the presence of synergism, higher conductivity, various active sites, rapid electron transport, and high surface area are the reasons for the enhanced sensing activity of the NiCo-LDH/F-HNT-based electrode for the sensing of PT. Lv et al. [104] also reported the fabrication of CoAl LDH decorated hematite (CoAl-LDH/ α -Fe₂O₃) for the sensing of H₂O₂, which demonstrated an LOD of 0.04 μ M and linear range of 1 to 2000 μ M including a sensitivity of 132.0 μ A/ $\text{mM}\cdot\text{cm}^2$. A Co-based LDH integrated with gold nanoparticles (Au NPs) was fabricated as an electrode modifier for the sensing of H₂O₂ [105]. The AuNPs/Co-LDH-based electrode demonstrated an LOD of 0.19 μ M and a sensitivity of 406.61 μ A $\text{mM}^{-1}\text{cm}^{-2}$ for the determination of H₂O₂ using an amperometry method. Diethofencarb (DFC) is used to fight fungal attacks in the agriculture industries to improve crop production [106]. However, the negative influences of DFC motivated the electrochemists to fabricate a sensor for monitoring DFC. A Zn-chromium (Cr) LDH/vanadium carbide (VC) was prepared by using a hydrothermal method, as shown in Figure 13. It was observed that ZnCr LDH has a nanoflower-shaped structure, which is attached on the VC surface. ZnCr-LDH/VC was coated on a screen-printed carbon electrode, i.e., SPCE, for electrochemical sensing applications. ZnCr-LDH/VC/SPCE exhibited an LOD of 2 nM for the monitoring of DFC via the DPV method. This sensor was also efficient in detecting the DFC in a real sample analysis in water and tomato samples.

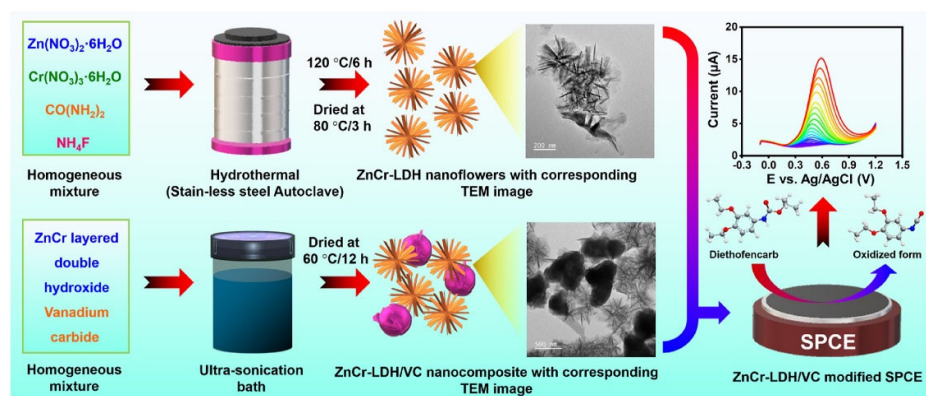


Figure 13. Schematic picture illustrating ZnCr LDH/VC composite for sensing of DFC. Reproduced with permission [106].

Carmoisine is an azo dye which has negative environmental impacts and its monitoring is of great significance [107]. Ni-Co LDH was obtained under facile conditions and was coated on a screen-printed graphite electrode (SPGE) for the sensing of carmoisine in the presence of tartrazine via the DPV method. The LOD of 0.09 μ M was achieved for the detection of carmoisine with a linear range of 0.3 to 125 μ M. Kokulnathan et al. [108] reported the construction of carbendazim (CDM) in the residues of the environment. For the sensing of CDM, the authors prepared a NiCo LDH and coated it on GCE and EIS, CV, and DPV techniques were used to determine CDM. The authors achieved an LOD of 0.001 μ M and a linear range of 0.006 to 14.1 μ M, with a sensitivity of 3.38 μ A $\mu\text{M}^{-1}\text{cm}^{-2}$. Singh et al. [109] reported the fabrication oxidized graphitic carbon nitride (O-g-CN)/copper (Cu)-Al LDH composite for the construction of a diclofenac sodium (DS) sensor. It was found that Cu-Al LDH was homogeneously deposited on the O-g-CN surface. The obtained O-g-CN/Cu-Al LDH was deposited on the surface of GCE, which demonstrated an acceptable LOD of 0.38 μ M with a linear range of 0.5 to 60 μ M via the DPV method. It is understood that phenolic compounds, for example, benzenediol (BD), have poor biodegradability and toxicity and pose threats to the environment and human life [110]. In this regard, AZnMo LDH was prepared with carbon black (CB) and proposed as a working electrode for the sensing of BD. AZnMo LDH/CB showed low electrical resistance and higher electrochemical reactivity

with improved electron transport. The presence of synergistic interactions between AZnMo LDH and CB enhanced the sensing mechanism of HQ, CC, and resorcinol (RC) via DPV. The authors reported LOD of 0.0054 μM , 0.0018 μM , and 0.075 μM towards HQ, CC, and RC, respectively. The proposed electrode was also validated towards real sample investigations in water, soil, and environmental samples. In 2023, a simple and cost-effective strategy was applied for the formation of ternary MnFeZn LDH and deposited on the GCE surface towards the sensing of flutamide (FLA) [111]. The formation of MnFeZn LDH is illustrated in Figure 14. A linear range of 0.019 to 2735.79 μM and LOD of 12.9 nM were obtained by using the DPV method. The proposed sensor also showed stability of 50 cycles with real sample sensing in river water. Thus, this type of sensor can be applied for practical applications.

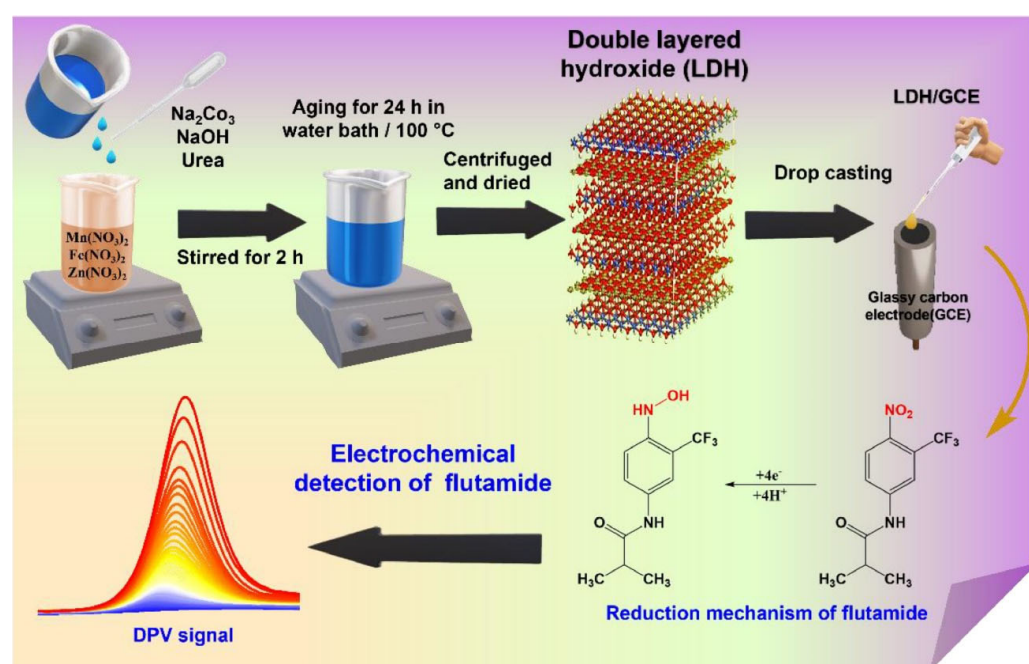


Figure 14. Schematic graph shows formation of MnFeZn LDH for FLA sensing. Reproduced with permission [111].

He et al. [112] reported the sensing of glucose and H_2O_2 using the CoZn-LDH@CuO-modified electrode via an amperometry method. An interesting LOD of 0.17 μM and high sensitivity of 4585 $\mu\text{A mM}^{-1}\text{ cm}$ were obtained via CoZn-LDH@CuO NSA/CF. Tajik et al. [113] fabricated a Ni-Co LDH/MWCNT composite and modified it with CPE to develop the 4-aminophenol (4-AP) sensor. The authors adopted CV, DPV, and chronoamperometry techniques for the electrochemical characterization of the Ni-Co-LDH/MWCNTs/CPE towards the sensing of 4-AP. The electrochemical studies exhibited a wide linear range of 0.02 to 700 μM , a sensitivity of 0.076 $\mu\text{A}/\mu\text{M}$, and an LOD of 0.01 μM . Ni-Co-LDH/MWCNTs/CPE also worked well for selectivity studies and real sample investigations in water samples. In 2024, a hydrazine (N_2H_4) sensor was developed by Zhou et al. [114] by employing an LDH-modified electrode. The authors obtained NiCo-LDH elf-assembled hollow nanocages (HNCs) by employing an in situ etching approach via a Cu_2O nanocube template (Figure 15a). Furthermore, the authors found that this unique structure is beneficial for electrochemical sensing applications and constructed a NiCo-LDH HNCs/CP electrode for the determination of N_2H_4 . The pH was optimized by recording CVs of NiCo-LDH HNCs/CP in different pH conditions for the sensing of N_2H_2 (Figure 15b). The higher activity of NiCo-LDH HNCs/CP was observed for N_2H_4 under a pH of 14. However, oxygen evolution at pH 14 may interfere with the sensing of N_2H_4 ;

therefore, the authors used pH 13 for further electrochemical studies. Furthermore, the authors observed that chronoamperometry is highly sensitive compared to the CV and further studies were carried out using chronoamperometry. The potential for the determination of N_2H_4 was also optimized and 0.6 V was found to be suitable and efficient (Figure 15c). The amperometric response of the NiCo-LDH HNCs/CP electrode shows that current response increases with the addition of N_2H_4 via chronoamperometry at 0.6 V (Figure 15d). This rapid increase in the current response was found to be linear by calibrating current responses versus the concentration of N_2H_4 , as shown in Figure 15e. An interesting LOD of $0.135 \mu\text{M}$ and a linear range of $3 \times 10^{-3} \text{ mM}$ to 2 mM and $3 \mu\text{M}$ to 6 mM were observed with good selectivity and stability.

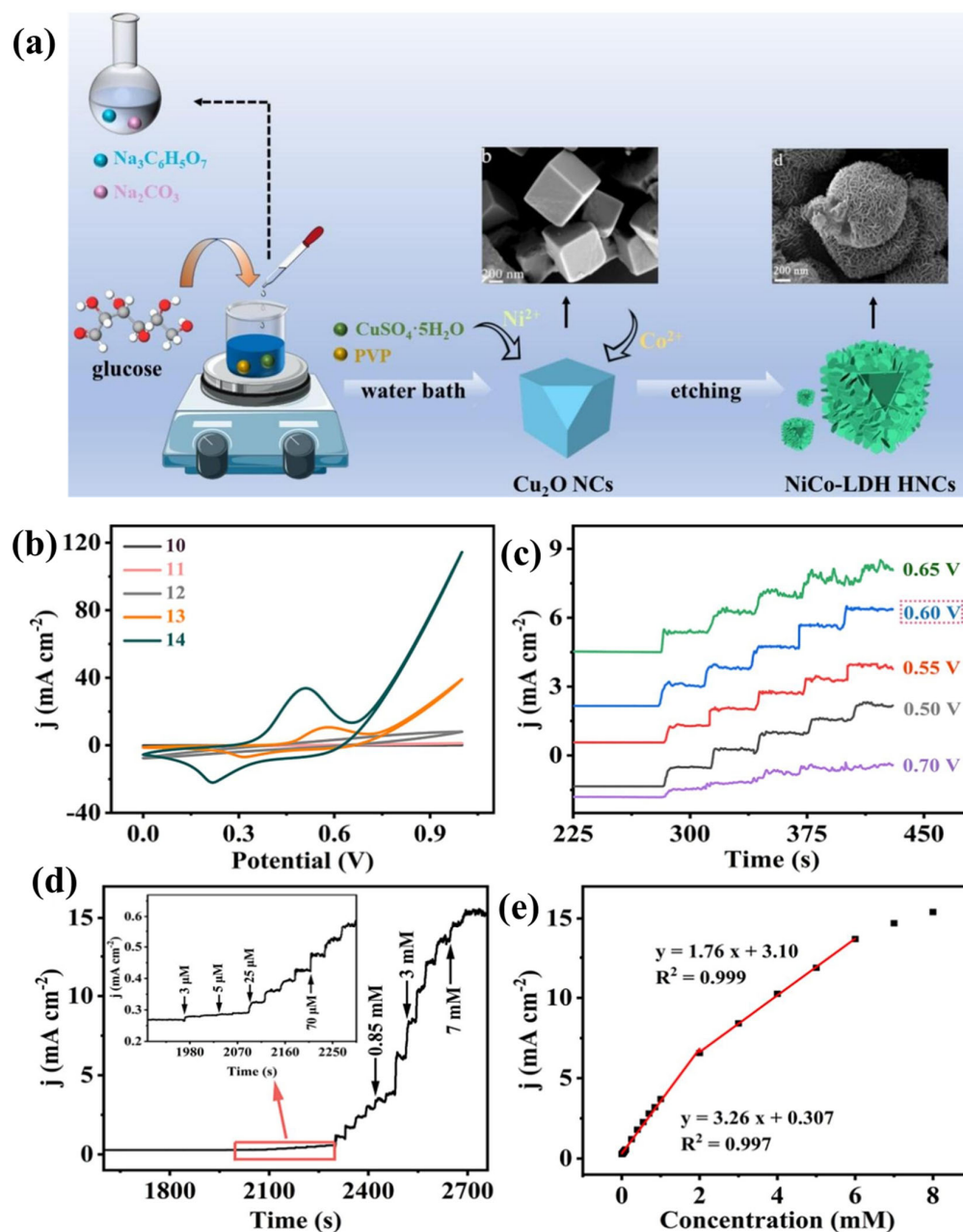


Figure 15. (a) Schematic illustration of formation of NiCo-LDH HNCs. (b) CV graphs of NiCo-LDH HNCs/CP for 1 mM N_2H_4 (under different pH conditions). (c) Amperometric responses of NiCo-LDH HNCs/CP with successive spike of 1 mM N_2H_4 (at different potentials). (d) Amperometric graph of NiCo-LDH HNCs/CP for N_2H_4 at 0.6 V. Inset enlarged graph. (e) Calibration curve between current versus concentration of N_2H_4 . Reproduced with permission [114].

Khan et al. [115] obtained a decent LOD of 0.004 μM and a linear range of 0.05 to 50 μM towards the detection of pentachlorophenol (PCP) by fabricating a Ni-Al LDH-modified GCE. EIS investigations revealed that Ni-Al LDH-modified GCE has good electrical conductivity and electrochemical ability for the detection of PCP. Ashry et al. [116] obtained an LOD of 0.3 μM for the sensing of paroxetine by using a β -Cyclodextrin/Zn-Fe LDH/g-CN composite-based electrode whereas Ragumoorthy et al. [117] developed a Mesalazine (MLZ) sensor by utilizing Co-Al LDH/GCE, which exhibits an LOD of 0.029 μM and a linear range of 0.049 to 665.1 μM . In a recent report by Stanley et al. [118], a propyl gallate (PG) sensor was developed by fabricating a NiFeCo-LDH and graphene aerogel (NiFeCo-LDH/GA)-based electrode and an LOD of 0.87 nM with excellent sensitivity of 41.22 $\mu\text{A } \mu\text{M}^{-1} \text{cm}^{-2}$ was achieved. The authors stated that the presence of high surface area and active sites enhanced the sensing of the proposed electrode for the determination of PG. In another report [119], NiFeCu-LDH/GA/SPCE was also explored as a PG sensor, which demonstrated an LOD of 0.004 μM and a linear range of 0.02 to 279.1 μM with excellent recovery in real samples. In another study [120], a Ni/Co LDH hollow cake (HC) was prepared by using a Co-based ZIF precursor via a benign ion etching process. The Ni/Co LDH HC-based electrode showed an LOD of 0.22 μM and a sensitivity of 7050 $\mu\text{A mM}^{-1} \text{cm}^{-2}$ for the monitoring of H_2O_2 . Guo et al. [121] reported the synthesis of NiMoO₄ nanorods@NiCo-LDH nanosheets for the sensing of H_2O_2 . The NiMoO₄ NRs@NiCo-LDH NSs/CC was explored as an electrode and an LOD of 112 nM with a wide linear range of 1 μmol to 9.0 mmol was observed concerning the monitoring of H_2O_2 using amperometry. The performance of the various reported sensors are displayed in Table 2.

Table 2. Sensing performance of reported sensors using LDH-based electrodes.

Electrode Material	Analyte	Detection Limit (μM)	Linear Range (μM)	Sensitivity ($\mu\text{A}/\mu\text{M}\cdot\text{cm}^2$)	Sensing Method	References
ILs-LDH modified GCE	BPA	0.0046	0.01–3.0	-	DPV	[86]
CoAl-LDH/MWCNTs	H_2O_2	5 $\mu\text{mol dm}^{-3}$	100–4000 $\mu\text{mol dm}^{-3}$	118 $\text{mA dm}^3 \text{mol}^{-1} \text{cm}^{-2}$	CA	[87]
Mg–Al-TGA LDH/GCE	Hg	0.8 nM	2–800 nM	-	SWASV	[88]
CuO@MnAl/GCE	H_2O_2	0.126	6 μM to 22 mM	-	Amperometry	[89]
Fe/Mg/Ni LDH/GCE	Pb	0.032	0.03–1	68.1 $\mu\text{A } \mu\text{M}^{-1}$	SWASV	[90]
Cd/Al/GCE	Anthracene	0.5 fM	0.1–100.0 pM	-	DPV	[91]
NiFe LDH/nickel foam	H_2O_2	0.5	5×10^{-4} –0.84 mM	-	Amperometry	[92]
MgFe LDH/GCE	Cd (II)	5.9 nM	0.1–1.0	-	SWASV	[93]
MgFe LDH/GCE	Pb (II)	2.7 nM	0.1–1.0	-	SWASV	[93]
Exfoliated Ni ₂ P/Al LDH/GCE	BPA	6.8 nM	0.02 to 1.51 μM	-	DPV	[94]
MgAl LDH/CP	Nitrite	0.03	14.8–222 μM	-	Amperometry	[95]
CNF–NiCo-LDH-GCE	MNZ	0.13 nM	3 to 57 nM	1.294 $\mu\text{A nM}^{-1} \text{cm}^{-2}$	DPV	[96]
WC@NiCo-LDH	NRF	0.005	0.02–83.4	6.53 $\mu\text{A } \mu\text{M}^{-1} \text{cm}^2$	DPV	[97]
WC@NiCo-LDH	NRF	0.002	0.002–346	40.81 $\mu\text{A } \mu\text{M}^{-1} \text{cm}^2$	Amperometry	[98]
WC@FeMn-LDH	DPA	0.0011	0.01–183.34	-	DPV	[99]
WC@NiCo-LDH	NRF	0.005	0.02–83.4	-	DPV	[100]
AZnMo-LDHs@VGCF	DMZ	0.021	0.25–570	1	DPV	[101]
OV-LDHs/H-MWCNTs/GCE	CT	0.074	0.5–150	-	DPV	[102]

Table 2. Cont.

Electrode Material	Analyte	Detection Limit (μM)	Linear Range (μM)	Sensitivity ($\mu\text{A}/\mu\text{M}\cdot\text{cm}^2$)	Sensing Method	References
OV-LDHs/H-MWCNTs/GCE	HQ	0.076	0.5–150	-	DPV	[102]
NiCo-LDH/F-HNTs	PT	0.003	0.012–24.5	-	DPV	[103]
CoAl-LDH/ $\alpha\text{-Fe}_2\text{O}_3$	H_2O_2	0.04	0.001–2	-	PEC	[104]
AuNPs/Co-LDH	H_2O_2	0.19	4 μM –16 mM	406.61 $\mu\text{A mM}^{-1}\text{cm}^{-2}$	Amperometry	[105]
ZnCr-LDH/VC	DFC	0.002	0.01–228	-	DPV	[106]
Ni–Co LDH NSs/SPGE	Carmoisine	0.09	0.3–125	-	DPV	[107]
NiCo-LDH	CDM	0.001	0.006–14.1	3.38	DPV	[108]
o-g-C ₃ N ₄ /CuAl-LDH/GCE	DS	0.64	0.5–60	-	DPV	[109]
AZnMo-LDHs/CB	HQ	0.0054	0.05–971	-	DPV	[110]
AZnMo-LDHs/CB	CC	0.0018	0.1–1036	-	DPV	[110]
AZnMo-LDHs/CB	RC	0.075	0.5–1408.5	-	DPV	[110]
MnFeZn-LDH	FLA	0.012	0.0199–2735.7	5.04	DPV	[111]
CoZn-LDH@CuO NSA/CF	H_2O_2	0.17	0.8 μM –3.5 mM	4585 $\mu\text{A mM}^{-1}\text{cm}^{-1}$	Amperometry	[112]
Ni-Co-LDH/MWCNTs/CPE	4-AP	0.01	0.02–700	0.076 $\mu\text{A}/\mu\text{M}$	DPV	[113]
NiCo-LDH	Hydrazine	0.135	3–2 $\times 10^3$	3260 $\mu\text{A mM}^{-1}\text{cm}^{-2}$	Amperometry	[114]
Ni-Al-LDH	PCP	0.004	0.05–50	-	DPV	[115]
CoAl-LDH/GCE	MLZ	0.029	0.049–665.1	-	DPV	[117]
NiFeCo-LDH/GA	PG	0.00087	0.001–297.43	-	DPV	[118]
NiFeCu-LDH/GA	PG	0.004	0.02–279.1	-	DPV	[119]
NiCo-LDH HC	H_2O_2	0.22	-	7050 $\mu\text{A mM}^{-1}\text{cm}^{-2}$	Amperometry	[120]
NiMoO ₄ NRs@NiCo-LDH NSs/CC	H_2O_2	0.112	1–9000	-	Amperometry	[121]

4. Conclusions and Future Perspectives

This article reviewed the progress in the development of 2D LDH and its composite-based materials for gas sensing and electrochemical sensing applications. The progress in LDH materials based on Mn, Cu, Fe, Mg, Co, Ni, Gd, and Al metals were compiled for sensing studies. The reported literature exhibits that LDH-based materials have promising features such as decent surface area and conductivity and physiochemical properties for the detection of various gases and hazardous compounds. The presence of synergy effects in the fabricated LDH-based composites provide active sites for catalytic reactions/adsorption of toxic gases and facilitate charge transport, which resulted in improved sensing performance. We believed that tuning the composition of the LDH-based materials may improve the selectivity and sensitivity of the gas sensors. In addition, LDH-based materials may offer various advantages such as the development of gas sensors at low temperatures, which makes them promising candidates for practical applications at a large scale. On the other side, LDH-based materials coated on different electrodes such as CPE, GCE, or SPCE showed excellent electrochemical activity for various electrochemical reactions. The LDH-based materials are found to be promising electrode modifiers for the construction of electrochemical sensors for the detection of environment pollutants. Unfortunately, despite the excellent sensing performance of the 2D LDH-based materials, some challenges still exist, which include structural instability and low electronic conductivity. The limited conductivity of LDHs may also restrict their applications as electrode modifiers in electrochemical sensors and sensing material for gas sensors. It can be noted that MXenes are

highly conductive material and are widely used as conductive support for the preparation of hybrid composites for various optoelectronic applications. The presence of synergistic interactions in the MXene-based composites may further improve their physicochemical properties. To further enhance the performance of LDH-based materials, it is crucial to combine LDHs with appropriate conductive materials to boost their overall conductivity. Thus, we also believe that future investigations may focus on the design and fabrication of simple and benign synthetic methods, with a combination of LDH with MXenes, conductive materials, or other layered metal sulfides to further improve the electrical conductivity and charge transport properties of the LDH materials. The combination of MXene and LDH materials may be a great idea to develop a highly efficient hybrid material for gas sensing and electrochemical applications. LDH/MXene-based hybrid composites may be adopted as sensing materials which can be further used for commercial purposes such as monitoring hazardous compounds or toxic gases for environment-related fields.

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