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Review

Electrocatalysts for enhanced sensing applications: a review of materials, performance metrics, and future prospects

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ABSTRACT

The integration of advanced electrocatalysts has revolutionized modern sensing technologies, enabling unique capabilities in environmental monitoring, healthcare diagnostics, and industrial process control. This review examines the current state and future directions of electrocatalyst materials and their applications in sensing platforms. It covers the remarkable progress in developing novel nanomaterial architectures, from metal-free catalysts to sophisticated hybrid systems, highlighting how strategic materials selection and design enhance key performance metrics including sensitivity, selectivity, and long-term stability. The review also covers a comprehensive discussion of metal-based, metal-free, and hybrid electrocatalysts with a focused analysis of their roles in enhancing sensor performance. Our discussion includes innovative approaches in materials engineering, particularly the emergence of two-dimensional materials like MXenes, and metal–organic frameworks, which have demonstrated exceptional potential for simultaneous multi-analyte detection. The review also provides insights into advanced voltammetric methods used in research for precise electrochemical sensing. Also, the review concludes by addressing challenges in scaling and sustainability, while exploring promising opportunities of combining artificial intelligence and smart sensor development.

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1 Introduction

Electrocatalysts play a significant role in facilitating electrochemical reactions that are important for a variety of energy and environmental technologies, including fuel cells, water electrolysis, and CO_2 reduction. Lately, their enhancement potential in sensing applications has caught up rather fast. Electrochemical sensors are experiencing high global demand due to their ease of use, affordability, and ability to provide real-time, on-site measurements. Market reports forecast substantial growth, driven by these sensors applicability in detecting and monitoring chemical and biological species across environmental, medical, food, and industrial fields. The integration of electrocatalysts into sensor platforms has demonstrated significant improvements in efficiency by lowering the overpotentials of electrochemical reactions, accelerating reaction kinetics, and expanding the electrochemically active surface area (ECSA) [1, 2]. The usage of electrocatalysts in sensing technologies is vital for developing high-performance sensors that might be sensitive, fast as well as dependable. Electrocatalysts facilitate electron transfer at the electrode–solution interface, which is fundamental in many sensing mechanisms. Through careful optimization of the composition, shape, and surface of electrocatalysts, researchers have notably advanced detection limits, response times, and selectivity for diverse analytes. Moreover, recent improvements in nanotechnology have delivered superior nanomaterials and hybrid catalysts as key additives in contemporary sensing systems, in addition to improving electrocatalytic performance through nanoscale effects and synergistic interactions. For instance, carbon-based nanomaterials, such as carbon quantum dots (CQDs), graphene quantum dots (GQDs), and hybrid materials like reduced graphene oxide (rGO), have emerged as highly effective electrocatalysts due to their large surface areas, high electrical conductivity, and excellent stability. These materials have been reported to facilitate efficient electron transfer, boost sensor sensitivity, and improve the overall performance of electrochemical sensors [3]. For example, one of the studies has demonstrated that combining reduced graphene oxide (rGO) with lanthanum zirconate ($La_2Zr_2O_7$) creates a composite material with enhanced catalytic properties. The rGO component introduces additional reaction sites and helps prevent electron–hole pair recombination, resulting in improved sensor performance [4].

Moreover, given the emphasis on green synthesis and energy-efficient processes, methods like room-temperature synthesis offer sustainable alternatives in the development of sensors. These approaches align with the broader goals of creating cost-effective, eco-friendly sensing technologies with improved functionality. In light of this, many researchers have focused on designing electrocatalysts for high-performance sensing applications using a variety of novel approaches, such as nanostructured materials, transition-metal addition, and other functional research techniques. This review focuses on emerging trends in electrocatalyst design for high-performance sensing applications. The main objective is to provide an overview of recent progress highlighting various strategies that have been used by researchers to enhance the sensing properties of sensors. We will explore the role of various electrical materials, such as transition metals, metal oxides, carbon-based nanomaterials, and hybrid systems to improve detection efficiency. In this review, we explore the fundamental concepts and critical performance metrics essential for selecting an electrocatalyst, examine the different types of electrocatalysts, and highlight the latest research developments. Finally, we discuss the opportunities and challenges associated with employing electrocatalysts in sensing applications.

2 Fundamentals of electrocatalysis

Electrocatalysis refers to the acceleration of an electrochemical reaction at the electrode–electrolyte interface with the help of a catalyst. The catalyst facilitates charge transfer between the electrode and the adsorbed species by reducing the activation energy and improving the efficiency of the reaction. This is especially true in detection applications. Electrocatalysts increase the sensitivity and specificity of electrochemical sensors through fast and accurate detection of the analyte. The fundamental reaction typically involves the adsorption of the reactant onto the electrode surface, charge transfer, chemical transformation of the adsorbed species, and breakdown. The efficiency of these steps is influenced by the properties of the catalyst used, such as total surface area, active sites, and electronic structure [5, 6]. The efficiency of electrode processes is affected by various factors, including the electrode composition, reactants, products, and intermediates present at the electrode–electrolyte

interface. The primary goal is to boost reaction kinetics, thereby increasing the electrical current output. This enhancement is typically achieved through two main approaches: first, by facilitating the formation of advantageous chemical bonds between the reacting species and the electrode surface, and second, by altering the reaction mechanisms to lower the activation energy barrier. By manipulating these factors, researchers in electrocatalysis strive to design more effective and energy-efficient electrochemical systems for various applications [7]. A plot between reaction progress and potential energy is shown in Fig. 1 illustrating how the catalyst reduces the potential energy required for a particular reaction.

An effective electrocatalyst should possess four important characteristics, such as remarkable catalytic activity for swift reaction kinetics, extensive surface area to maximize reaction sites, excellent electrical conductivity for efficient electron transfer, and high durability to maintain performance over time. [9]

In electrocatalysis, some key factors need consideration. They are electron transfer kinetics, catalyst–electrode interaction, and electrocatalyst design.

First, the electron transfer rate is crucial for determining the performance of the electrocatalyst. Faster kinetics generally lead to higher sensor sensitivity and faster response times. Recent research on transition metals and metal-free catalysts often focuses on how modifications in electronic properties enhance electron transfer rates. Therefore, it is necessary to choose an electrocatalyst in the view of improving the electron transfer rate. For instance, in a study by Massah et al. using H_2N-MIL-101(Cr)-CPE modified electrodes, it was found that the charge transfer resistance dropped significantly from 7.8 kΩ [for bare Carbon Paste Electrode (CPE)] to 0.13 kΩ (catalyst-modified), indicating a substantial improvement in electron transfer kinetics due to catalyst modification. This demonstrates how enhancing electron transfer rates can drastically improve electrocatalyst efficiency [10].

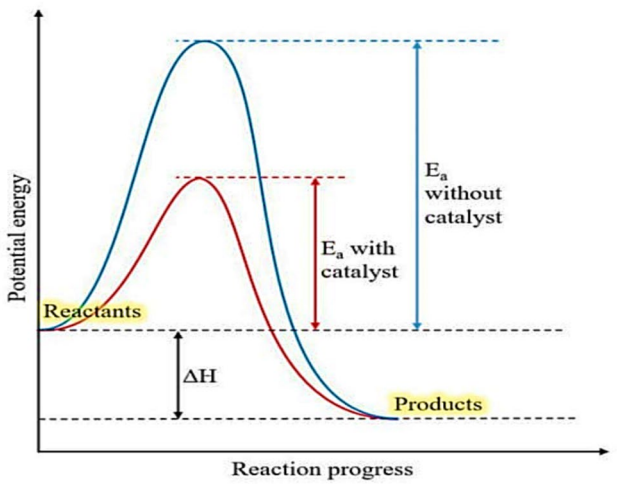

Fig. 1 Catalyst effect on lowering activation energy in reaction progress (adapted from [8] with permission from MDPI, under CC by 4.0 license.)

Second, the strength of the interaction between the catalyst and the electrode material also plays a major role in determining the stability as well as the performance of the electrocatalyst. Strong interactions ensure efficient electron flow but must also allow for easy desorption of products to avoid catalyst poisoning. In the same study by Massah et al. mentioned above [10], the presence of amine groups on the Metal–Organic Framework (MOF) significantly improved electrostatic attraction, leading to a higher anodic peak current (12.40 μA) in the H_2N-MIL-101(Cr)-modified electrode compared to the bare electrode (9.19 μA).

The final and foremost important one is the design. It is necessary to improve the size, morphology, and composition of electrocatalysts to enhance their performance significantly. For example, nano-sized catalysts offer a higher surface-to-volume ratio, increasing the number of active sites and boosting catalytic performance. Researchers are exploring the use of hybrid materials that combine metallic and non-metallic components to enhance electron transfer and surface interactions for sensor applications. For example, a recent study developed an electrochemical apta sensor that paired gold nanoparticles with polydopamine and an iron-based metal–organic framework (Fe-MOF). This sensor demonstrated high sensitivity, a wide detection range, low detection limits, and strong repeatability when used to measure carcinoembryonic antigen levels in serum samples. The exceptional performance is attributed to the abundant carboxyl groups and unsaturated iron sites within the porous Fe-MOF structure, which provide ample-binding sites for the CEA-specific aptamer probes. Additionally, the redox properties of the polydopamine and Fe-MOF components help facilitate rapid electron transfer, further amplifying the sensor's signal output [11]. To summarize, the key elements of electrocatalysis highlight the role of electron transfer dynamics and the interactions between catalysts and electrodes in boosting the

responsiveness and accuracy of sensors. The development of electrocatalysts, especially through the fine-tuning of surface area and structure, is essential for enhancing sensitivity and reaction rates, both of which are necessary for effective electrochemical sensing. By making specific changes to catalysts to optimize electron transfer rates, it is possible to make considerable improvements in sensor sensitivity and overall functionality.

2.1 Key performance metrics

When developing or selecting electrocatalysts for sensing applications, it is crucial to evaluate their performance based on a set of essential metrics (Fig. 2). These key performance indicators help researchers and engineers assess how effectively a catalyst can enhance detection in electrochemical sensing devices. The metrics, shown in the figure as sensitivity, selectivity, response time, and stability were selected based on their direct influence on the analytical reliability, functional efficiency, and long-term usability of the sensor. In addition, these criteria were also prioritized based on a comprehensive review of existing literature to align with widely accepted standards in the field. By carefully considering these metrics, one can improve the overall efficiency and suitability of an electrocatalyst for a given sensing application.

2.1.1 *Sensitivity*

Sensitivity is a crucial parameter that determines a sensor's ability to detect small concentrations of an analyte, often represented by the slope of the calibration curve [12]. As shown in Fig. 3, the slope of the calibration curve serves as a crucial indicator of an electrocatalyst's sensitivity. This metric quantifies the relationship between changes in analyte concentration and the corresponding signal response (current vs. concentration). A steeper slope in the calibration curve suggests that the sensor can detect smaller variations in analyte levels, thus indicating higher sensitivity in measurements. Highly sensitive electrocatalysts generate a strong response even to low analyte concentrations, making them suitable for detecting trace levels of gases, ions, or biomolecules [13]. In a study by Alouni et al., the effect of decorating graphene with zinc oxide (ZnO) nanoparticles (NPs) was investigated for NO_2 detection. Two graphene sensors with different ZnO loadings, 5 wt.% and 20 wt.%, were prepared, and their responses to NO_2 at room temperature were compared. The results showed that the graphene loaded with 5 wt.% ZnO NPs (G95/5) exhibited higher sensitivity in detecting low concentrations of NO_2 compared to the one with 20 wt.% ZnO NPs (G80/20). Furthermore, measurements under dry and humid conditions revealed that the G95/5 sensor's sensitivity increased almost eightfold when the background changed from dry to 70% relative humidity,

Fig. 2 Key performance metrics for an electrocatalyst

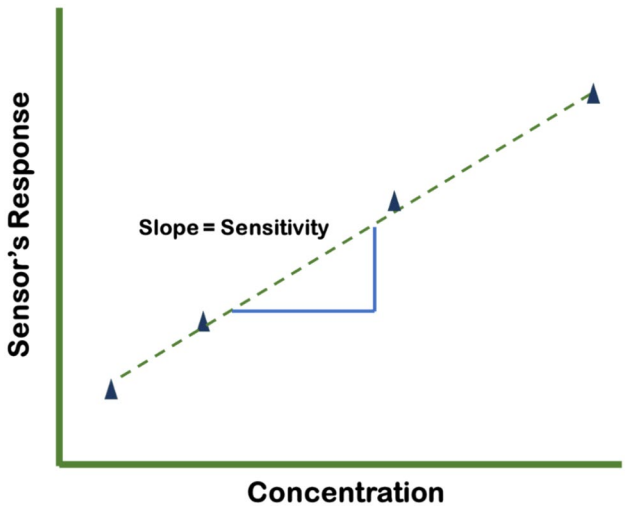

Fig. 3 Calibration graph showing the slope as an indicator of electrocatalyst sensitivity

highlighting its sensitive responsiveness to ambient moisture [14]. Lei et al. reported the development of an electrochemical sensing membrane by incorporating a Cu/ZIF-8/Ag composite into the porous structure of a titanium substrate to boost detection sensitivity. Electrochemical impedance spectroscopy (EIS) demonstrated a significant decrease in electron transfer resistance under dynamic flow conditions compared to a static environment. Additionally, limiting current analysis revealed improvements in glucose mass transfer efficiency and a reduction in boundary-layer thickness, which ultimately improved the sensitivity [15]. In another study, Sri Balaji et al. proposed a method to enhance the sensitivity of a rotating ring-disk electrode by engineering a heterostructure composed of niobium oxide, nitride, and carbide embedded in nitrogen-doped carbon (referred to as Nb_2O_5/NbN/NbC@NC). The presence of this heterointerface contributes active sites for improved electrochemical interaction, while the nitrogen-doped carbon facilitates efficient charge transfer during detection. This approach demonstrated effective sensitivity in various media, including phosphate buffer, vegetables, grains, and environmental samples [16].

2.1.2 Selectivity

Selectivity in catalysts refers to their ability to distinguish the target analyte from other substances in a sample. This property is crucial for practical sensing applications, as interference from non-target species can compromise sensor accuracy. In selective sensing, catalysts that facilitate specific electrochemical reactions, such as the selective oxidation of glucose or other target molecules, enable more precise detection of analytes by promoting particular electrochemical processes [17, 18]. For example, in a study by Hussein et al., they showcased the high selectivity of a ZnO/Co_3O_4/reduced graphene oxide (rGO) nanocomposite as a non-enzymatic glucose sensor. This sensor demonstrated impressive performance metrics, including a sensitivity of 1551.38 μA mM^{-1} cm^{-2}, a low detection limit of 0.043 μM, and a wide linear range from 0.015 to 10 mM when operated at a potential of 0.55 V. Notably, selectivity tests revealed that the ZnO/Co_3O_4/rGO nanocomposite responded strongly to glucose while showing minimal interference from other substances like uric acid, ascorbic acid, fructose, maltose, and sucrose. This demonstrates its capability to selectively detect glucose even in complex biological samples [19]. For applications of this nature, it is essential that an electrocatalyst possesses selectivity for the specific analyte being detected.

In general, both sensitivity and selectivity are important. Sensitivity is fundamental for identifying small amounts of analytes, enabling sensors to react to even slight variations in analyte levels. Besides, high selectivity allows sensors to reliably differentiate target analytes from other materials, ensuring consistent functionality in complex environments where interference from non-target substances might otherwise impact the outcomes.

2.1.3 Response time and detection limit

The response time and detection limit are crucial indicators of a sensor's ability to quickly and accurately detect an analyte. In electrochemical sensing, these performance metrics directly impact a sensor's practical utility. Recent advancements in nanomaterial design have led to significant improvements in these areas [20]. For instance, Jafari et al. prepared a hybrid electrocatalyst combining a CeO_2/CuO/NiO heterostructure with nitrogen-doped reduced graphene oxide (N-rGO). This complex material, synthesized through a multi-step process including co-precipitation, heat treatment at 750 °C, and hydrothermal techniques, demonstrated exceptional glucose sensing capabilities in alkaline conditions. The N-rGO component enhanced electrical conductivity and electron transfer, contributing to efficient glucose oxidation. Importantly, this sensor achieved an impressively low detection limit of 0.053 μM, enabling the measurement of extremely low glucose concentrations, and an exceptionally fast response time of approximately 2.9 s [21]. Moreover, in particular, hydrogen (H_2) sensors are important for hydrogen energy development and safety monitoring, but fast and accurate detection of low concentrations remains a challenge. Several scientists have recently improved and still improvising the response time and detection limit in H_2 sensors [22–24]. For example, in a study by Zhang et al., Pd-doped rGO/ZnO-SnO2 nanocomposites were developed, achieving impressive hydrogen sensing capabilities. The sensor exhibited exceptionally fast kinetics, with a response time of only 4 s when exposed to 100 ppm of H_2. Additionally, it showed a recovery time of 8 s under the same conditions.

Perhaps, most impressively, the sensor achieved an extremely low detection limit of 50 ppb when operated at 380 °C. These values highlight the importance of response time and detection limit in ensuring efficient and reliable hydrogen sensing [25].

2.1.4 Stability and durability

Stability and durability are critical factors in the long-term performance of electrocatalysts, especially for sensors operating in harsh conditions. These properties ensure that catalysts maintain their activity over extended periods without degradation, resisting corrosion, fouling, and poisoning [26, 27]. A recent study by Nazir et al. exemplifies this importance, showcasing a novel Sb_2S_3–NiS_2 heterostructure electrode for detecting carbendazim, mercury (Hg^{2+}), and arsenic (As^{3+}). This nanocomposite demonstrated superior electrocatalytic activity due to its high conductivity, charge capacitance, synergistic effects, abundant active sites, and metal trap centers. Crucially, it exhibited excellent stability, maintaining performance over weeks of use [28]. This aligns with broader research trends emphasizing the significance of stability and durability in electrocatalyst design. While sensitivity and response time are important, the ability of a catalyst to function reliably over extended periods without frequent replacement or recalibration is equally vital for practical sensor applications. Consequently, researchers continue to prioritize the development of materials that combine high catalytic activity with long-term stability under diverse operational conditions.

The following reasons show why response time, detection limit, and stability are important.

- A quick response time allows sensors to detect and relay changes in analyte concentration almost instantaneously, which is critical in applications requiring real-time monitoring.
- A low detection limit expands the sensor's capability to detect trace amounts of analytes, enhancing its suitability for applications that demand high precision.
- Stability over time is essential for long-term sensor use, especially in harsh environments. A stable electrocatalyst resists degradation, maintaining performance across varied operational conditions and extended periods.

2.1.5 Cost and scalability

The high cost of electrocatalyst materials, particularly precious metals, has driven research into more affordable alternatives for commercial sensing applications. This includes exploring metal-free and hybrid electrocatalysts that offer similar performance at lower costs. For industrial applications, scalability is crucial, prompting the development of cost-effective synthesis methods suitable for mass production. Various researchers have focused on reducing costs while making products commercially viable [29]. For example, Lima et al. made a significant contribution by developing a simple, low-cost method for fabricating disposable electrochemical devices using graphite and nail polish. Their sensor demonstrated impressive performance, including high sensitivity and a low detection limit, and was successfully applied to analyze a compound in sports drinks, yielding results comparable to established methods [30]. This work exemplifies the ongoing efforts to create electrocatalysts that are both efficient and economically viable for widespread commercial use, bridging the gap between laboratory innovations and practical sensing technologies. Also, considering both the cost and environmental benefits, green synthesis techniques also come as a viable choice. Several research works highlight the green synthesis techniques like Microwave-assisted synthesis, Biosynthesis, and Coprecipitation methods with natural chelating agents. For example, Lin et al. developed a simple, rapid, and environmentally friendly method for synthesizing Au–Ag alloy nanoparticles using Melaleuca quinquenervia leaf extract (MQLE) as both a reducing and capping agent, combined with microwave-assisted green synthesis techniques [31]. Similarly, Anjana et al. reported the synthesis of stable silver nanoparticles (AgNPs) using Cyanthillium cinereum through a rapid, microwave-assisted green approach, where the plant extract served dual roles as a reducing and capping agent. These nanoparticles were designed for the purpose of detecting neurotransmitters, such as dopamine [32]. Such innovations in low-cost, sustainable as well as scalable techniques represent significant milestones in advancing commercial sensing technologies.

3 Types of electrocatalysts

Electrocatalysts are the materials that speed up the electrochemical reactions that occur at the surface of electrodes or on solid/liquid interfaces. These materials are typically categorized into three main groups based on their composition and metal content: metallic, non-metallic, and composite electrocatalysts. Each category offers unique properties and advantages for various electrochemical applications, allowing researchers and engineers to select the most appropriate type for specific needs.

3.1 Metal-based electrocatalysts

Over the past several decades, researchers in surface science have conducted extensive investigations into the relationship between surface structure and catalytic performance. These studies have primarily utilized single-crystal metal planes as model catalysts [33]. A key finding from this research is that surfaces with more open structures tend to demonstrate significantly enhanced catalytic activity and stability. In the context of face-centered cubic (fcc) metals, which include elements like platinum, palladium, rhodium, iridium, and gold, high-index planes are of particular interest [34]. These planes are characterized by Miller indices where at least one index exceeds unity, resulting in an open surface structure. Such surfaces feature a high concentration of atomic steps and kink sites. The atoms at these steps and kinks have fewer neighboring atoms, resulting in lower coordination numbers. This low coordination leads to increased reactivity, with groups of typically five to six such atoms forming active sites capable of efficiently breaking chemical bonds. Consequently, high-index planes generally exhibit superior catalytic properties compared to their low-index counterparts. Low-index planes, such as {111} and {100}, are composed of closely packed atoms with surface atomic coordination numbers of 9 and 8, respectively. These densely packed surfaces typically show lower catalytic activity due to the higher coordination and thus lower reactivity of their surface atoms. Due to these reasons, metal-based electrocatalysts, particularly those involving precious metals, such as platinum (Pt), palladium (Pd), and ruthenium (Ru), are among the most effective catalysts due to their excellent catalytic activity and electron transfer properties [35].

However, their widespread adoption faces barriers due to their hefty price tags and vulnerability to deactivation, such as through carbon monoxide poisoning [36]. In response to these challenges, the scientific community has broadened its focus to explore more cost-effective alternatives. Materials based on non-precious metals—notably nickel, cobalt, and iron—have shown great promise. These substitutes deliver comparable catalytic performance at a fraction of the cost, making them highly attractive for industrial-scale implementation [37]. The gas-sensing domain has seen a surge in interest in metal oxide compounds. Materials, such as iron oxide (Fe2O3), nickel oxide (NiO), and chromium oxide (Cr2O3), are becoming increasingly common in sensor applications [38, 39]. Hematite (α- Fe_2O_3) has emerged as a particularly intriguing option, thanks to its natural abundance, chemical robustness, affordability, and favorable electronic properties. While bulk Fe_2O_3 often suffers from sluggish reaction rates and underwhelming gas-sensing capabilities, recent breakthroughs in nanotechnology have offered solutions. By engineering Fe_2O_3 at the nanoscale, researchers have successfully overcome these limitations, unlocking enhanced performance and expanding its potential in next-generation sensing devices [40]. For instance, Navale et al. investigated the gas-sensing capabilities of hematite (Fe_2O_3) nanoparticles across a range of different gases. Their experiments included exposure to nitrogen dioxide, ammonia, carbon monoxide, carbon dioxide, and methane. The results revealed a particularly strong sensitivity to nitrogen dioxide compared to the other gases tested. This heightened response was attributed to the strong chemical affinity between nitrogen dioxide molecules and the hematite nanoparticle surface, highlighting the potential of nanostructured Fe_2O_3 for selective NO_2 detection in mixed gas environments [41]. Similarly, some researchers have also demonstrated that nano-sized nickel oxide (NiO) exhibits enhanced surface catalytic properties for ammonia detection compared to its bulk counterpart. For example, Juang et al. employed a hydrothermal technique to synthesize well-dispersed, porous NiO nanosheets arranged in a spherical assembly. The unique structure of these nanosheets, featuring numerous holes, greatly increases the material's surface area, thereby enhancing its gas detection capabilities. The dispersed configuration of the nanosheets facilitates increased surface interaction with ammonia molecules, resulting in a more pronounced sensing response. In experimental trials, this porous NiO

nanosheet-based sensor displayed remarkable sensitivity, achieving an 87.17% response to a low concentration of 15 ppm ammonia at an operating temperature of 150 °C. Moreover, the sensor demonstrated rapid response times and high sensitivity even at low ammonia concentrations, underscoring its effectiveness for ammonia detection applications. This highlights the potential of nanostructured metal-based electrocatalysts as an advanced material for highly sensitive and efficient ammonia gas sensors [42].

As an inference, metal-based electrocatalysts, particularly those employing precious metals like platinum and palladium, offer excellent catalytic activity and electron transfer efficiency due to their open surface structures and high reactivity. However, their high cost and susceptibility to deactivation restrict broader applications. To address these limitations, non-precious metal alternatives, such as nickel, cobalt, and iron, as well as metal oxides like Fe_2O_3 and NiO, have shown substantial potential. Nano-structuring these alternatives significantly enhances their surface area and analyte interactions, boosting sensitivity and selectivity in gas detection applications, especially for gases like nitrogen dioxide and ammonia. Moreover, doping also helps in enhancing the catalytic property [43, 44]. In general, dopants can introduce new energy levels or modify existing ones within the catalyst's electronic structure, thereby influencing its interaction with reactants and facilitating electron transfer. Doping can also alter the internal electron distribution of the catalyst, affecting its ability to bind with reactants and stabilize key reaction intermediates. In the case of transition-metal catalysts, doping is particularly significant, as it enables tuning of the d-band center position. This directly impacts the strength of metal–reactant interactions, which further improves catalytic activity. The concept of heteroatomic doping is further elaborated in a review by Qi et al., where a P-doped M-NxC structure is discussed. In this system, phosphorus (P), which has low electronegativity compared to nitrogen, modifies the electronic environment of the single-atom catalyst (SAC). This P-doping alters the coordination structure around the metal center and optimizes the adsorption and activation of reaction intermediates at the active sites, which enhances the catalytic performance [45].

In summary, the discussion highlights the use of nanostructured metal-based electrocatalysts, especially non-precious metals (Ni, Co, Fe) and metal oxides (Fe_2O_3, NiO), along with trends like high-index planes, nanoscale engineering, and heteroatomic doping (e.g., P-doping) to enhance sensing performance in a cost-effective and scalable manner.

3.2 Metal-free electrocatalysts

Electrochemical sensing has traditionally relied on noble metals and transition-metal oxides as the most effective electrocatalysts, owing to their outstanding catalytic properties. However, these materials face significant drawbacks in practical applications. Noble metals are expensive and lack long-term stability, while transition-metal oxides suffer from poor electrical conductivity. These limitations have sparked a growing interest in developing alternative metal-free electrocatalysts. The ideal candidates would possess large surface areas, facilitate efficient charge transfer, and exhibit exceptional electrocatalytic performance, all without the use of metals [46]. In light of this, the field of electrochemical sensing is undergoing a significant shift toward metal-free electrocatalysts, with carbon nanomaterials at the forefront of this innovation. These materials, including graphene, carbon nanotubes, and carbon dots, offer a compelling alternative to traditional noble metal and transition-metal oxide catalysts. Carbon-based electrocatalysts boast several advantages that make them attractive for practical applications [47]. Their relatively low cost addresses the economic barriers associated with noble metals, while their excellent electrical conductivity overcomes the limitations of transition-metal oxides. Additionally, these materials are generally considered more environmentally friendly, aligning with growing sustainability concerns in scientific research and industrial applications. The performance of carbon nanomaterials can be further enhanced through heteroatom doping, a process that introduces elements like nitrogen, sulfur, or phosphorus into the carbon structure [48]. This modification creates beneficial defects and alters the material's electronic properties, often resulting in improved catalytic activity. For example, nitrogen-doped graphene has demonstrated exceptional performance in various electrochemical reactions due to increased electron density around the nitrogen atoms, facilitating more efficient electron transfer. These graphene-based sensors exhibit high affinity for the detection of analytes like H_2O_2, NO, glucose, dopamine, paracetamol, trinitrotoluene, phenol, etc. [49–51]. For instance, Chang et al., demonstrated the potential of nitrogen doping to

enhance the gas-sensing capabilities of reduced graphene oxide (rGO). The team employed a modified Hummers' method to produce graphene oxide, which was subsequently transformed into nitrogen-doped reduced graphene oxide (N-rGO) through a hydrothermal process using graphene oxide and ammonium hydroxide as starting materials. Morphological analysis revealed that rGO exhibited a smooth, flat sheet-like structure, while N-rGO displayed a folded surface topology. This structural modification in N-rGO was found to contribute to improved gas sensitivity. Moreover, they observed contrasting semiconducting behaviors between the two materials under ambient conditions. rGO sensors exhibited p-type characteristics, whereas N-rGO sensors showed n-type behavior. Both materials demonstrated responsiveness to nitric oxide (NO) gas at concentrations below 1000 parts-per-billion (ppb) at room temperature. Among these, N-rGO sensors showed superior performance, detecting NO gas at concentrations as low as 400 ppb. As shown in Fig. 4a, the detection of NO gas involves three main steps. First, when the material is exposed to air, oxygen molecules (O_2) are adsorbed onto its surface, forming adsorbed oxygen ions (O_2^- and $2O^-$) through interactions with free electrons, which fill the oxygen vacancies. In the second step, when NO gas is introduced, it reacts with the adsorbed oxygen, resulting in the formation of adsorbed NO species (NO(ads)) and subsequent reactions that create oxygen vacancies and release nitrogen gas (N_2). Finally, upon re-exposure to air, the NO adsorbates are replaced by new oxygen species, restoring the surface with adsorbed oxygen ions and preparing it for further NO detection cycles. This process enables the sensor to detect NO by monitoring changes in surface reactions and electron transfer, as represented by the equations (Eqs. 1, 2, and 3) inside the Fig. 4a. When exposed to 1000 ppb of NO, the N-rGO sensor exhibited a sensitivity of 1.7,

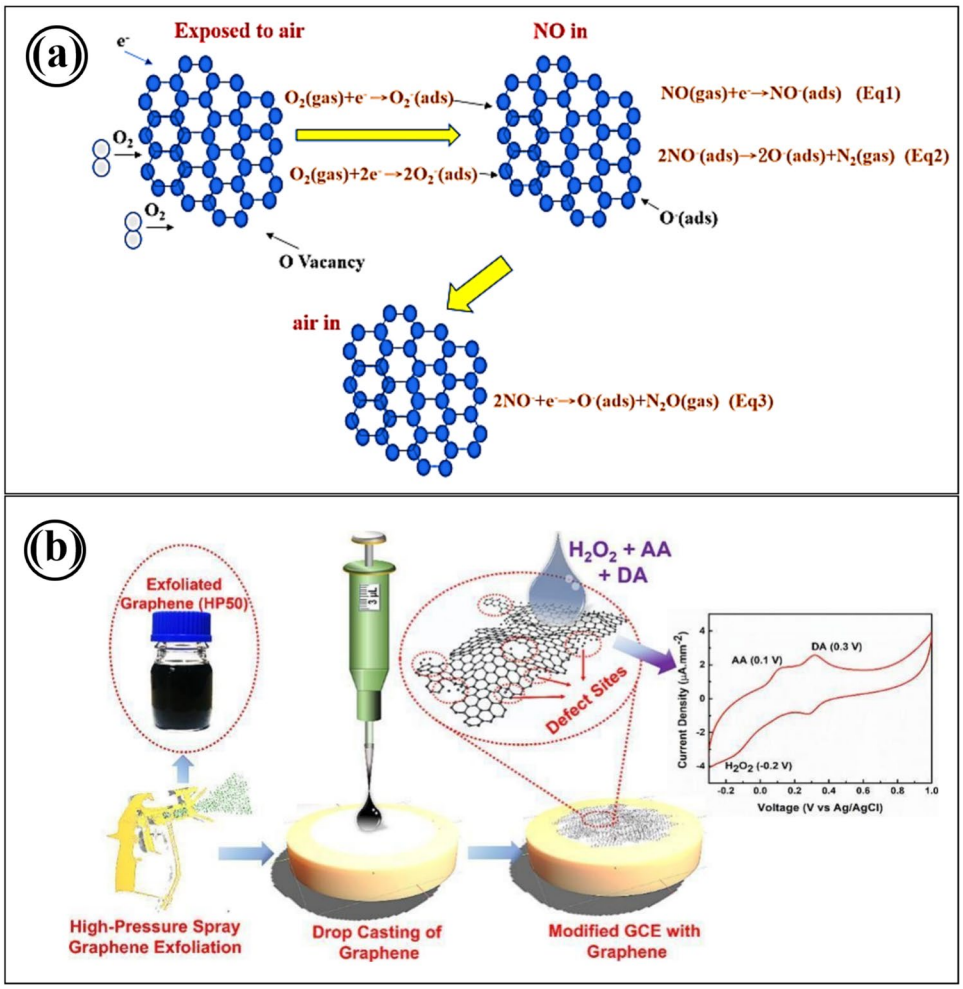

Fig. 4 **a** Illustration of three steps involved in NO sensing by N-rGO sensor (reused from [52] with permission from Nature under CC BY 4.0), **b** illustration of synthesis of modified Glassy Carbon Electrode (GCE) with graphene with defect sites (reused from [53] with permission from Elsevier under CC BY 4.0)

significantly outperforming the rGO sensor, which had a sensitivity of only 0.012 [52].

An innovative research by Ramu et al. introduces a groundbreaking approach to graphene-based material synthesis utilizing a high-pressure airless spray technique, as shown in Fig. 4b [53]. By exfoliating bulk natural graphite, they produced unique nanoscale flakes comprising both amorphous carbon and few-layer graphene structures, distinguished by abundant edge-plane defects. Their electrochemical sensing studies revealed distinctive voltage responses for three key analytes: hydrogen peroxide showed reduction at − 0.2 V, while ascorbic acid and dopamine displayed oxidation peaks at 0.1 V and 0.3 V, respectively (versus Ag/AgCl reference in 1 M solution). The material also demonstrated remarkable sensing capabilities with high-sensitivity values of 164, 739, and 3357 μA mM^{-1} cm^{-2} and impressively low detection limits of 15, 1.7, and 2.1 μM for hydrogen peroxide, ascorbic acid, and dopamine, respectively. Recent research has explored the potential of three-dimensional (3D) carbon architectures to address some limitations of conventional two-dimensional (2D) structures. While 2D materials have been revolutionary, their assembly also significantly impacts performance. This is influenced by the macromorphological arrangement of the 2D layers [54, 55]. By assembling 2D graphene sheets into 3D forms, such as hydrogels, aerogels, sponges, and foams, researchers aim to maintain the material's outstanding properties while mitigating issues like aggregation due to π–π interactions. For instance, Yavari et al. developed a macroscopic three-dimensional graphene foam (GF) network that offers high sensitivity and reversibility for gas detection, effectively addressing the limitations associated with two-dimensional structures. The GF, synthesized using a porous nickel foam scaffold, boasts an impressive specific surface area of about 850 m^2 g^{-1} and a low density of 5 mg cm^{-3}, resulting in 99.7% porosity. In gas detection tests, the foam demonstrated remarkable sensitivity, detecting NH_3 and NO_2 at concentrations as low as 20 ppm in air at room temperature. The sensor showed a normalized resistance change (ΔR/R) of approximately 30% for 1000 ppm NH_3 and 16% for 200 ppm NO_2. The robust 3D structure enabled rapid, reversible gas detection, marking a significant improvement over 2D graphene-based sensors [56].

As an inference, metal-free electrocatalysts, especially carbon-based materials like graphene and carbon nanotubes, offer a promising alternative to traditional noble metals for electrochemical sensing. Unlike traditional metal-based catalysts, they are cost-effective, corrosion-resistant, highly conductive, and environmentally benign, making them highly suitable for achieving stability and sustainability. With enhancements like heteroatom doping and 3D structuring, carbon nanomaterials achieve impressive sensitivity and selectivity, making them ideal for detecting gases (e.g., NO and NH_3) and biomolecules. This shift toward metal-free options provides a sustainable pathway for developing efficient, high-performance sensors suitable for diverse applications. This advancement aligns with broader research trends aimed at addressing global challenges in sustainable energy, green chemistry, and environmental protection.

3.3 Hybrid electrocatalysts

Hybrid electrocatalysts combine metal-based and metal-free materials to leverage the benefits of both systems. These catalysts often feature transition metals or metal oxides supported on carbon nanomaterials, which provide high surface areas and excellent catalytic properties. Hybrid catalysts can address the issues of metal scarcity and poisoning while simultaneously offering high catalytic performance. For example, graphene-metal nanoparticle hybrids are widely researched for their applications in sensing, as graphene provides excellent conductivity and a large surface area, while metal nanoparticles offer high catalytic activity. A significant contribution to hybrid nanomaterial development came from Qu and colleagues, who engineered a graphene-nickel nanoparticle (NiNP) composite using an innovative far-infrared-assisted reduction method. Their synthesis protocol involved the simultaneous reduction of graphene oxide and nickel (II) ions with hydrazine. While graphene-metal hybrids were well known, their use of far-infrared assistance in the reduction process represented a novel approach. The researchers utilized the material's magnetic properties to anchor it onto a magnetic electrode, which was subsequently employed for carbohydrate detection. The modified electrode showed remarkable catalytic efficiency in carbohydrate measurements. To validate its practical applications, the team analyzed human serum samples (20 mL of 0.1 M NaOH added to 200 mL serum) from healthy subjects at Shanghai Zhongshan Hospital. Using chronoamperometry at 0.5 V versus SCE, they conducted recovery studies by sequentially

adding 20 mL aliquots of 60 mM glucose solution at 25-s intervals. Their analysis revealed a serum glucose concentration of 5.2 mM, with recovery rates between 93.8 and 102.5%, demonstrating the sensor's reliability for clinical applications [57]. Notably, the same team previously developed another hybrid nanomaterial by combining graphene with copper nanoparticles (CuNPs). They synthesized this graphene–CuNP composite through an in situ chemical reduction process. The researchers integrated this hybrid material with capillary electrophoresis techniques to create an enhanced platform for carbohydrate detection and analysis [58]. Metal oxide–carbon nanotube composites have also emerged as effective materials for environmental sensing applications. One notable example comes from research conducted by Amit et al., who developed novel sensor platforms for cadmium detection in drinking water. Their approach combined multi-walled carbon nanotubes (MWCNTs) with either tin oxide (SnO_2) or manganese dioxide (MnO_2), along with an ionic liquid, deposited on indium tin oxide (ITO) substrates, as shown in Fig. 5a. The researchers employed an eco-friendly synthesis method, utilizing natural extracts from tomatoes and oranges to produce the metal oxides, which helped reduce production costs. The resulting sensors showed promising performance characteristics. The SnO_2-based composite demonstrated sensitivity in the 20–200 ppb range, with a detection limit of 3.61 ppb. Meanwhile, the MnO_2-based sensor operated effectively between 300 and 2000 ppb, achieving a detection limit of 41.34 ppb. These results underscore the potential of hybrid nanocomposites as effective tools for monitoring hazardous heavy metals in water supplies [59].

Metal oxide–carbon composites represent another significant advancement in sensor development, as demonstrated by Xiao et al. Their team engineered a novel metal/metal oxide@carbon composite (M/MO@C) through the carbonization of a copper/nickel-based metal–organic framework (Cu/Ni–MOF), as shown in Fig. 5b. This synthesis approach resulted in metal/metal oxide nanoparticles being evenly dispersed throughout a porous carbon framework. Through detailed materials' characterization using X-ray diffraction and X-ray photoelectron spectroscopy, they confirmed the formation of multiple active species, including Cu_2O, CuO, Ni, and NiO. The composite material, designated as M/MO@C-800, showcased exceptional glucose sensing capabilities, attributed to the synergistic effects of the Cu_2O/CuO

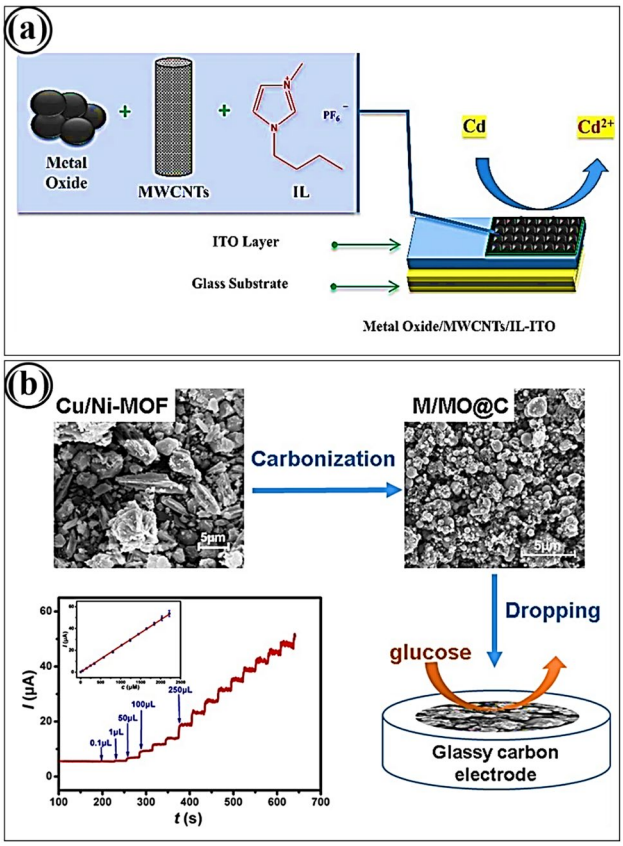

Fig. 5 **a** Representation of deposition of metal oxide and MWCNTs on ITO substrate (reproduced from [59] with permission from Elsevier), and **b** preparation process of electrocatalyst from Cu/Ni-MOF and the results of glucose sensing (reproduced from [60] with permission from Elsevier)

and Ni/NiO components combined with the porous carbon structure. The sensor achieved impressive analytical performance with a broad linear response range (0.1 μM to 2.2 mM) and high sensitivity (detection limit: 0.06 μM). Additionally, the device demonstrated robust reproducibility, long-term stability, and strong selectivity in real sample analysis, establishing its viability as an advanced sensing platform [60].

As an inference, hybrid electrocatalysts, combining metal-based and carbon-based materials, offer a powerful synergy that enhances both catalytic activity and conductivity. By leveraging the high surface area and conductivity of carbon materials alongside the catalytic efficiency of metal nanoparticles, hybrid systems address limitations like metal scarcity and stability issues.

Particularly this section highlights hybrid electrocatalysts that combine metal or metal–oxide

nanoparticles (e.g., Ni, Cu, SnO_2, and MnO_2) with carbon-based materials, such as graphene and carbon nanotubes (CNTs). Key trends include green synthesis methods, MOF-derived carbon composites, and the use of magnetic or porous structures to enhance sensitivity, selectivity, and stability for clinical (e.g., glucose detection) and environmental (e.g., heavy metal detection) sensing applications. These advancements suggest that hybrid electrocatalysts hold significant potential for versatile, high-performance sensors applicable to both clinical and environmental monitoring.

4 Emerging design and engineering strategies for electrocatalysts

Several emerging designs and engineering strategies are being developed for the purpose of sensing in electrocatalytic sensing applications. They include nano-structuring of materials and specifically controlling the morphology and functionalizing the surface of the electrocatalysts, along with doping and alloying approaches and mechanistic insights into electrocatalyst–surface interactions.

4.1 Nano-structuring and morphology control

Generally, nanostructures are known to offer high surface areas due to their large surface-to-volume ratios, which increases the number of active sites available for detecting various compounds. This nanostructuring of electrocatalysts enhances accuracy and proves especially beneficial in medical applications. Numerous metal nanoparticles have been explored for sensing purposes. For example, silver nanoparticles on NiOOH nanorods can detect urea in urine and serum [61], while iridium oxide nanoparticles can sense PBDE in distilled water [62]. Additionally, platinum nanoparticles of 4 and 20 nm have been used to detect hydrogen peroxide [63], and graphene–bimetallic nanoparticle composites are applied in bisphenol A detection [64]. Recent research focuses on further enhancing material morphology, with 2D nanostructures proving effective for constructing advanced 2D sensing platforms using materials like TMD, MOFs, and MXenes [65]. A study by Cogal et al. explored the development of novel electrochemical sensors using 2D hybrid carbon nanofibers. These sensors, which incorporated cobalt-doped 2D-$MoSe_2$ and polypyrrole (PPy), were designed for the concurrent detection of three important biological molecules: ascorbic acid (AA), dopamine (DA), and uric acid (UA).

The researchers employed a three-step synthesis approach:

(i) initial hydrothermal synthesis of 2D transition-metal dichalcogenide (TMD) nanosheets;
(ii) followed by electrospinning and thermal treatment to produce carbon nanofibers;
(iii) finally, the construction and testing of the electrochemical sensing platform.

Microscopy analysis, as shown in Fig. 6A, revealed that Co/$MoSe_2$/PPy@NF exhibited uniform, smooth surfaces with fiber diameters in the 395–469 nm range. The nanosheet structure was confirmed through TEM analysis, as depicted in Fig. 6B and C. After further processing, SEM imaging (Fig. 6D) showed that the Co/$MoSe_2$/PPy@CNF demonstrated increased surface roughness and porosity, with fiber diameters expanding to 850 nm–1 μm. Additional TEM examination (Fig. 6E and F) further validated the successful integration of Co/$MoSe_2$/PPy nanosheets in a layered arrangement [66].

Researchers have also explored 2D metal–organic frameworks (MOFs) for sensing applications, as demonstrated in a study by Ma et al. Their work focused on developing a non-enzymatic sensor for hydrogen peroxide (H_2O_2) detection by combining silver nanoparticles with a two-dimensional copper-porphyrin MOF framework. The synthesis process for the Ag NP/Cu-TCPP nanocomposite is illustrated in Fig. 7a. The research team employed multiple analytical techniques to characterize the material, including scanning electron microscopy, transmission electron microscopy, X-ray diffraction, X-ray photoelectron spectroscopy, and Fourier-transform infrared spectroscopy. These analyses confirmed the successful formation of a two-dimensional sheet structure with silver nanoparticles evenly distributed across the MOF surface. Microscopy images provided detailed structural insights, with Fig. 7b revealing the morphology of the 2D Cu-TCPP nanosheets and Fig. 7c showing the integrated Ag NP/Cu-TCPP nanocomposite structure. The MOF demonstrated peroxidase-like catalytic behavior, effectively reducing H_2O_2. The sensor's practical applicability was validated using a commercial disinfectant containing 3% H_2O_2, achieving impressive recovery rates ranging from 97.8 to 100.2%, demonstrating its reliability for H_2O_2 concentration measurements [67].

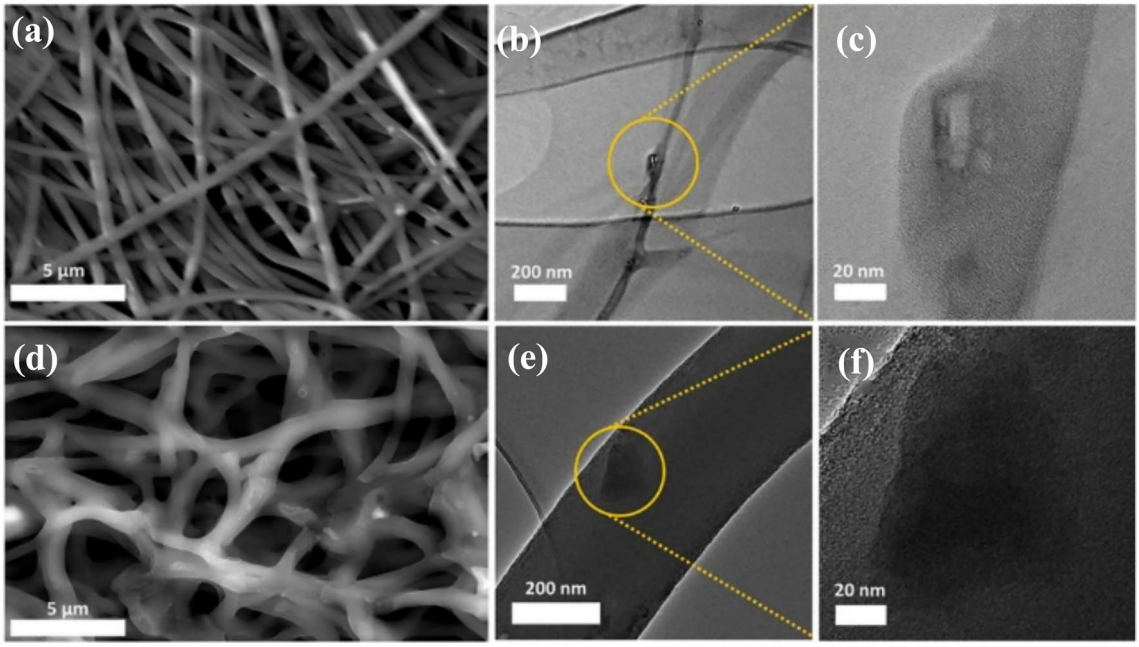

Fig. 6 SEM (**A**) and TEM (**B**) and **C** images of Co/$MoSe_2$/PPy@NF, SEM (**D**) and TEM (**E**) and **F** images of Co/$MoSe_2$/PPy@CNF (Reproduced from [66] with permission from Springer under CC by 4.0 license)

Similarly, two-dimensional transition-metal carbides and nitrides, known as MXenes, have emerged as significant materials in recent research. Their synthesis involves a complex process that begins with MAX phase materials, from which specific elements (Al, Si, or Ga) are selectively removed through etching. Various etching agents can be employed, including hydrofluoric acid, fluoride salts, other acids, non-aqueous solutions, halogens, and molten salts. The choice of etchant plays a crucial role in determining the surface chemistry of the resulting MXene. Following the etching process, the multi-layered structure undergoes exfoliation to produce individual MXene sheets. The versatility of MXenes is demonstrated through their various fabrication forms, which include powders, inks, films, fibers, and spherical structures. These materials possess several advantageous properties—notably their excellent conductivity, compatibility with biological systems, and water-loving nature—making them particularly suitable for electrochemical sensing applications. Their utility extends to detecting various substances, from environmental contaminants to biological markers and pharmaceutical compounds. A notable example is found in the work of Rasheed and colleagues, who developed a sensing platform combining platinum nanoparticles with $Ti_3C_2T_x$ MXene, as illustrated in the Fig. 7d. This sensor effectively detected Bisphenol A (BPA), achieving a detection threshold of 32 nM and maintaining linearity across concentrations from 50 nM to 5 µM [68]. Additionally, various 2D sensing platforms based on materials like g-C_3N_4 and h-BN are also widely utilized. Table 1 provides a list of some of the electrochemical sensing platforms developed using g-C_3N_4, $Ti_3C_2T_x$, MOF, and h-BN materials.

As a summary, the concept of nano-structuring and morphology control underscores the essential role of nanotechnology in advancing the field of electrochemical sensing. By engineering materials at the nanoscale, researchers can achieve high surface-to-volume ratios, which significantly increases the number of active sites available for detecting various compounds. This is particularly advantageous in medical and environmental applications, where sensitivity and specificity are paramount. For instance, silver nanoparticles on NiOOH nanorods have been successfully used to detect urea and graphene–bimetallic nanoparticle composites are employed in bisphenol A detection. Each of these materials has been tailored to exhibit unique interactions with target molecules, demonstrating the versatility of nanostructures in creating highly selective and efficient sensors. Furthermore, the development of two-dimensional (2D) nanostructures, such as MOFs and

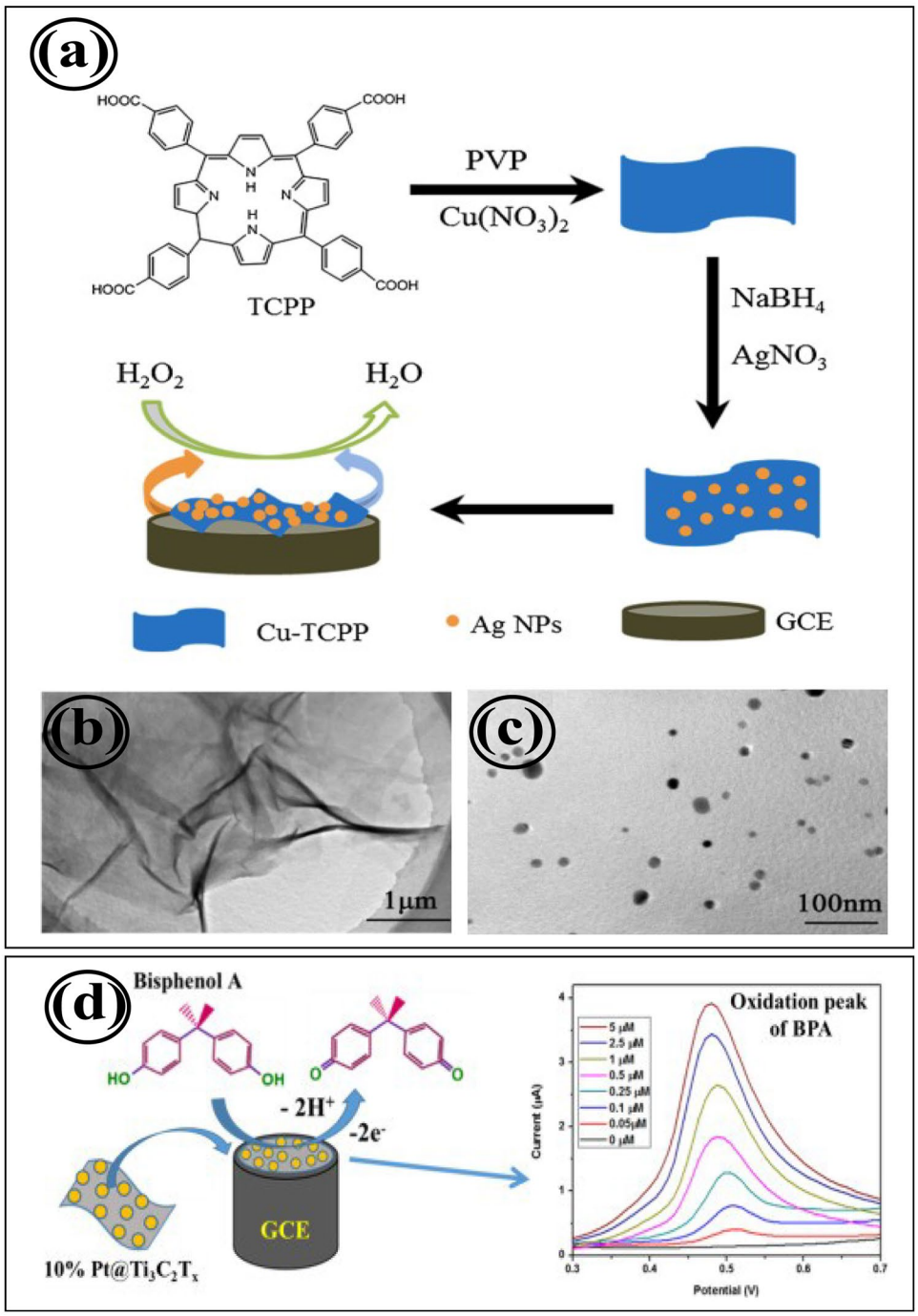

Fig. 7 **a** Schematic illustration of the preparation process for Ag NP/Cu-TCPP nanocomposite, **b** SEM image of 2D Cu-TCPP nanosheets, **c** SEM image showing the integrated Ag NP/Cu-TCPP nanocomposite structure (reproduced from [67] with permission from Springer Nature) **d** schematic representation of the sensing platform incorporating platinum nanoparticles with $Ti_3C_2T_x$ MXene for BPA detection. (Reproduced from [68] with permission from Elsevier)

MXenes, marks a significant advancement in sensor technology. The examples of Co/$MoSe_2$/PPy@NF sensors for multi-molecule detection and Ag NP/Cu-TCPP nanocomposites for hydrogen peroxide sensing reveal that precisely controlled synthesis processes can yield materials with enhanced catalytic properties and high sensitivity. The flexibility of 2D materials, which can be fabricated in various forms like films and powders, makes them suitable for detecting a wide range of substances. Such advancements suggest that the continued evolution of nanostructured sensors will lead to highly adaptable, robust, and sensitive detection platforms across diverse fields, addressing critical needs in healthcare and environmental monitoring.

Table 1 List of electrochemical sensing platforms developed using modified electrodes with g-C_3N_4, $Ti_3C_2T_x$, MOF, and h-BN

Material	Modified electrode	Linear range	Sensitivity	Stability	Target	Detection limit	References
g-C_3N_4	g-C_3N_4/FE_3O_4-CPE	14–120 µM	0.16µA/µM	96.8%(2 weeks)	Tramadol	0.1 µM	[69]
	g-C_3N_4/GO/Fc-TED/GCE	0.045–213 µM	–	94.7%(30 days)	metolcarb	8.3 nM	[70]
	TiO_2-g-C_3N_4@AuNPs/GCE	0.5–3 nM	–	95.5%(20 days)	AMX	0.2 nM	[71]
	CdO/PANI/mpg-C_3N_4/GCE	0.05–80 µM	0.0393µA/µM	94.5%(30 days)	Ep	0.011 µM	[72]
	g-C_3N_4/β-CD/GCE	1–100 µM	0.2µA/µM	–	TNT	68 ppb	[73]
$Ti_3C_2T_x$	$Ti_2C_2T_x$/GCE	0.015–10 mM	–	90% (5 weeks)	DA	3 nM	[74]
	Ti_3C_2Tx/GCE	50 nM–100 µM	0.024µA/µM	–	CBZm	10.3 nM	[75]
	H–C_3N_4/Ti_3C_2Tx/GCE	0.5–1.5 µM	49.91µA/µM	90.9% (15 days)	Pb^{2+}	0.6 nM	[76]
	20%Pd@Ti_3C_2Tx/GCE	0.5–10 µM	5.71µA/µM.cm^2	–	L-Cys	0.14 µM	[77]
	AuNPs-Ti_3C_2-NTO-PEDOT/GCE	0.0001–20 ng/mL	–	84.23% (2 weeks)	PSA	0.04 pg/mL	[78]
h-BN	D-h-BN/GCE	0.01–30 µM	0.0848µA/µ	96.55% (4 weeks)	4-AP	0.003 µM	[79]
	h-BCN	10–500 µM	0.32µA/µM	–	UA	2 µM	[80]
	PtNPs/POM/h-BN	0.1–300 µM	–	–	NHS	60 nM	[81]
	$h-_{BN}$/HNTs	0.009–173 µM	0.459µA/µM.cm2	–	FZ	0.001 µM	[82]
	MIP/GQDs/2D-hBN/GCE	1 pM–10 nM	30.014µA/nM	–	SER	0.2 pM	[83]
MOF	NiCo-MOF	1 µM–8 mM	0.6844µA/ mM.cm^2	–	Glc	0.29 µM	[84]
	Ni-MOF400	5 µM–4.1 mM	2918.2µA/ mM.cm^2	–	Glc	0.92 µM	[85]
	Ni-MOF/GCE	0.5–8 mM	2412µA/µM.cm^2	–	Glc	0.23 µM	[86]
	Ni-MOF/GO/GCE	0.10–300.0 µM	2.411µA/µM	–	4-CP	8 nM	[87]
	MOF-818@RGO/MWCNTs-3/GCE	0.2–7Mm and 7–50 µM	12.89µA/µM	95% (1 week)	CA	0.0052 µM	[88]
	Ni-MOF@CNTs/GCE	0.001–1 µM	284.64µA/ mM.cm^2	96.0% (7 days)	BPA	0.35 nM	[89]

4.2 Surface functionalization techniques

The modification of electrocatalysts through various functionalization approaches has become a key strategy in materials science research. This is particularly evident in the development of carbon-based catalytic materials, where functionalization enables researchers to engineer specific physical and chemical attributes [90]. Among the diverse functionalization strategies employed, acid-based oxidation has proven effective for incorporating oxygen-containing functional groups onto carbon surfaces. Additionally, researchers have successfully implemented ultrasonic techniques, utilizing sound waves to enhance the functionalization process and improve material properties. In this view, Balram et al. made notable contributions to pharmaceutical sensing technology through their work on detecting acetaminophen (APAP) with exceptional sensitivity. Their innovative approach centered on the development of a hybrid material that combined functionalized carbon nanotubes with metal–oxide nanostructures. Specifically, they created a composite by integrating zinc oxide nanoparticles with multi-walled carbon nanotubes that had been modified with amine groups (NH_2-MWCNTs). The synthesis protocol involved two key steps: first utilizing ultrasonic treatment to facilitate amine functionalization of the nanotubes, followed by an arginine-based solution method that generated uniquely structured ZnO particles resembling begonia flowers. Ultrasonic treatment is commonly employed to aid the amine functionalization of nanotubes by

utilizing acoustic cavitation, which helps in dispersing the nanotubes and improving their interaction with functionalizing agents. This cavitation generates localized zones of high temperature and pressure, effectively breaking apart nanotube agglomerates, increasing the accessible surface area, and facilitating the grafting of amine groups onto the surface [91, 92]. The sensing platform, detailed in Fig. 8a, was constructed by depositing this novel nanocomposite onto a screen-printed carbon electrode (SPCE). Performance testing revealed impressive capabilities, with the sensor able to detect APAP concentrations as low as 1 nM and exhibiting high sensitivity (89.64 μA μM^{-1} cm^{-2}). The research included extensive performance validation, examining crucial parameters, such as long-term stability, measurement reliability, and the

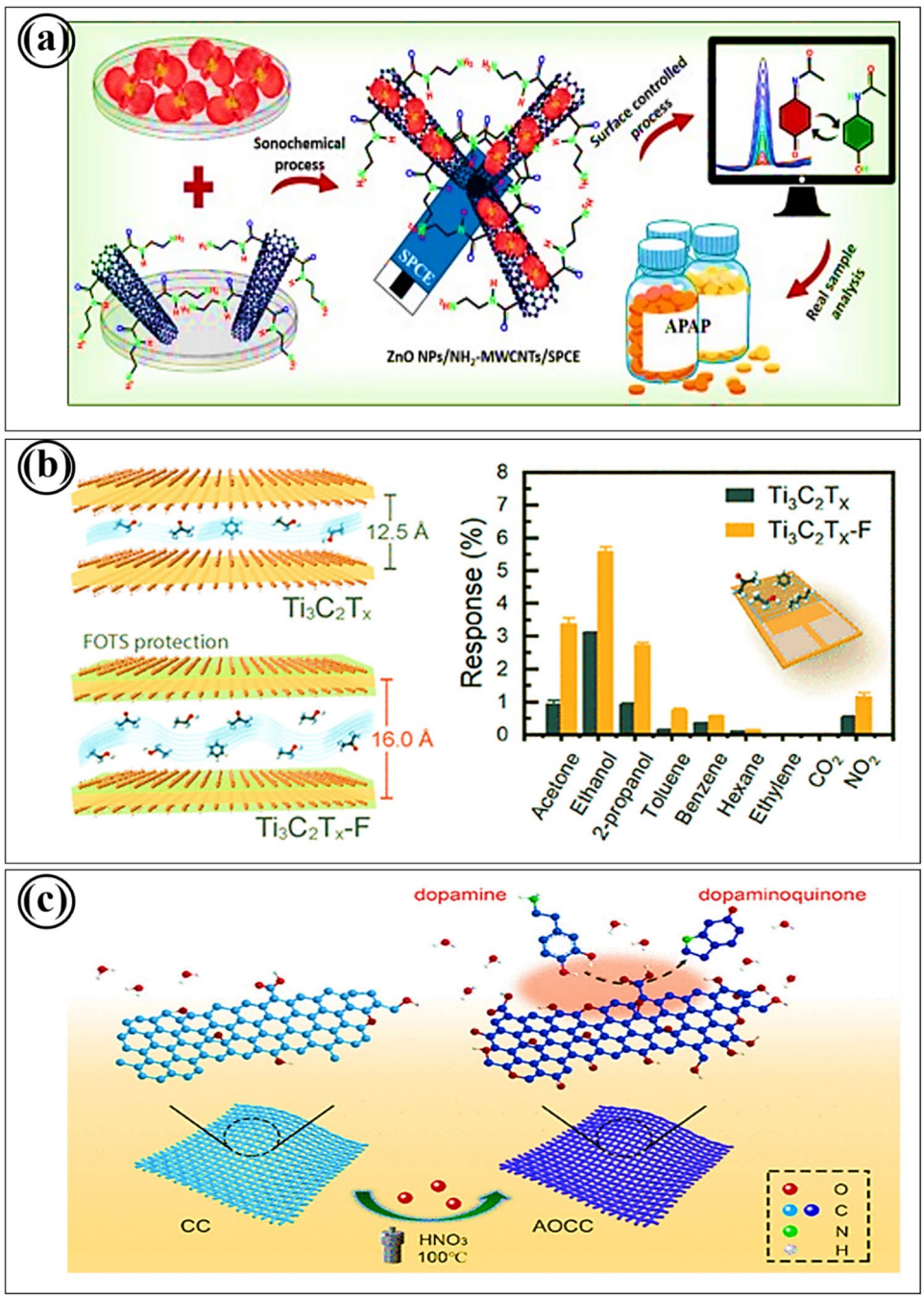

Fig. 8 **a** A detailed schematic illustrating the preparation of ZnO/NH_2-MWCNTs on SPCE for analysis (Reproduced from [93] with permission from Elsevier), **b** depiction of performance of functionalised-$Ti_3C_2T_x$ MXene systems in various gas detection systems (adapted with permission from [94]. Copyright 2020 American Chemical Society), **c** Illustration of process of modification of carbon cloth through acid-based surface treatment (Adapted with permission from [95]. Copyright 2020 American Chemical Society)

ability to function accurately in the presence of potential interfering substances. To demonstrate real-world applicability, the team successfully tested their sensor using various practical samples, analyzing APAP content in pharmaceutical tablets and complex biological matrices including human serum and urine specimens [93]. Similarly, in their work, Chen et al., focused on $Ti_3C_2T_x$ MXene systems. Although these materials naturally possess excellent sensing properties, their effectiveness typically diminishes when exposed to humid conditions, primarily due to moisture-induced degradation and oxidative processes. The team tackled this challenge by implementing a novel functionalization strategy that employed fluoroalkyl silane compounds (FOTS). This surface treatment successfully created moisture-resistant properties while enhancing the material's overall performance and longevity. When tested these functionalized materials in gas detection systems, as documented in Fig. 8b, revealed impressive capabilities across multiple applications. The sensors proved effective in detecting an array of chemical compounds, demonstrating versatility in identifying both organic vapors and gas molecules, including but not limited to acetone, ethanol, toluene, carbon dioxide, nitrogen dioxide, and ethylene. Performance analysis revealed multiple strengths including exceptional detection limits, measurement consistency, extended functional lifespan, specific molecular recognition, and rapid sensing dynamics [94].

In addition, a novel approach to electrochemical sensor development was demonstrated in research by Ma et al., who explored the modification of carbon cloth through acid-based surface treatment, as shown in Fig. 8c. While acid oxidation has been traditionally employed for enhancing carbon materials in capacitors, catalysts, and current collectors, its potential in sensing applications remained largely unexplored. The research team developed an innovative metal-free sensing platform by subjecting carbon cloth (CC) to controlled acid oxidation, resulting in the incorporation of oxygen-containing functional groups. This modification transformed the material's surface characteristics, creating enhanced accessibility for target molecules like dopamine through improved hydrophilic properties. Comparative analysis revealed that the functionalized carbon cloth (AOCC) substantially outperformed its unmodified counterpart in electrochemical sensing applications. The optimized sensor exhibited remarkable performance metrics, including high sensitivity (9320 μA mM^{-1} cm^{-2}), exceptional detection capabilities down to 10 nM, and broad-range functionality spanning from 0.1 to 104.5 μM. The device demonstrated robust performance across key parameters, including target specificity, operational longevity, and measurement consistency [95].

This highlights how modifying electrocatalysts through targeted functionalization methods has emerged as a key strategy in enhancing the efficiency, sensitivity, and specificity of electrochemical sensors. In particular, carbon-based materials have benefited greatly from such approaches, as functionalization allows for precise control over their surface properties, which in turn influences their chemical reactivity and sensing capabilities. Techniques, such as acid-based oxidation and ultrasonic treatment, have been instrumental in achieving desired functional groups on carbon surfaces, which enhance interactions with specific target molecules. Overall, the mentioned literature of examples illustrates how surface functionalization techniques enable tailored electrocatalyst performance across various sensing applications, pushing the boundaries of sensitivity, stability, and material longevity in electrochemical sensor development.

4.3 Alloying approaches

In electrocatalytic sensing applications, researchers utilize various metallic alloys that fall into three distinct categories. Noble Metal Alloys exhibit remarkable stability and catalytic efficiency, though their widespread adoption faces constraints due to cost and availability limitations. Noble metal–non-noble metal alloys represent a strategic combination, merging precious metals with more common elements to achieve an optimal balance between performance and cost-effectiveness. Non-noble metal alloys, while initially displaying lower catalytic activity, can achieve enhanced performance through targeted modifications of their surface properties and structural characteristics [96]. Cost-effective alternatives to precious metals have gained significant attention in sensing technology, with nickel-based alloys emerging as particularly promising materials [97]. These non-noble metal systems combine affordability with desirable characteristics, such as high conductivity, thermal robustness, and resistance to corrosive elements [98]. Based on this approach, Sasikumar et al. developed an innovative sensor for detecting fenitrothion (FNT), a concerning organophosphate-based pesticide. Their design incorporated a bimetallic oxide combining nickel and

cerium (NiCeO), which underwent extensive testing in both environmental waters and agricultural produce, specifically eggplant and bitter gourd samples. The researchers found that their NiCeO-based platform demonstrated enhanced performance through optimized charge transfer and abundant active sites for electrochemical reactions. Performance evaluation under controlled pH environments revealed impressive capabilities: the sensor exhibited a detection sensitivity reaching approximately 104.383 (± 0.002) μA μM^{-1} cm^{-2}, could identify FNT at concentrations down to 1.8 nM, and maintained accuracy across concentrations spanning from 1 to 5747.3 μM. Field testing confirmed the sensor's reliability through successful analyte recovery studies in complex sample environments [99]. Also, Panda's et al. introduced an innovative single-use electrochemical sensing platform incorporating platinum-nickel alloy nanostructures (PtNi NPs) for monitoring hydrogen sulfide emissions from breast cancer cells. The research team synthesized PtNi nanoparticles using hydrothermal techniques, achieving uniform particles approximately 5.6 nm in size. While detecting H_2S in cellular environments presents significant challenges due to the dynamic equilibrium between various sulfur species (H_2S, HS^-, S^{2-}) under biological conditions, the team found that their PtNi NPs efficiently promoted H_2S oxidation reactions in neutral phosphate buffer solutions (pH 7.0). Performance analysis revealed impressive capabilities: the sensor functioned accurately across concentrations from 0.013 to 1031 μM, achieved detection thresholds as low as 0.004 μM (at S/N = 3), and demonstrated sensitivity of 0.323 μA μM^{-1} cm^{-2}. Additional testing confirmed the device's durability, measurement consistency, and ability to function accurately despite potential interfering compounds. The sensor proved effective in analyzing diverse sample types, including environmental water, biological fluids, such as urine and saliva, and demonstrated particular utility in monitoring H_2S production from both breast cancer cells and mouse fibroblasts, benefiting from its biocompatible design. Figure 9 depicts a schematic of an electrochemical sensing process using platinum–nickel (PtNi) alloy nanoparticles to monitor hydrogen sulfide (H_2S) emissions from breast cancer cells [100].

Studies have also highlighted molybdenum carbide (Mo_2C) as a promising alternative to platinum-based systems, offering comparable electronic properties in a non-noble metal format. A significant breakthrough in this field emerged from Kokulnathan et al., who

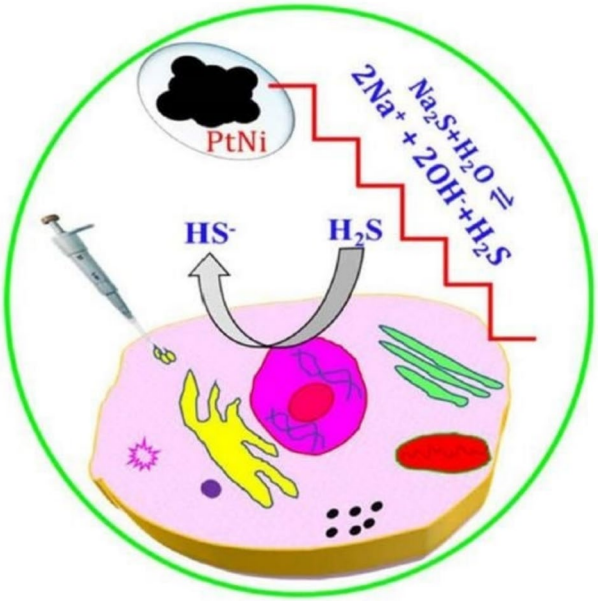

Fig. 9 Schematic representation of an electrochemical sensing platform using PtNi nanoparticles for the detection of hydrogen sulfide emissions in breast cancer cells (reproduced from [100] with permission from MDPI under CC by 4.0 license)

engineered a flexible sensing device targeting parathion-ethyl (PE). Their electrochemical studies demonstrated the superior catalytic activity of Mo_2C-enhanced electrodes over traditional versions in PE reduction. This improved functionality was linked to the material's distinctive 2D configuration, which creates an optimal combination of conductive properties, expansive reaction surfaces, and numerous catalytically active regions. The testing revealed impressive analytical capabilities of the Mo_2C sensor, with detection sensitivity reaching 10 μA μM^{-1} cm^{-2}, detection limits as low as 0.004 μM, and reliable performance across concentrations from 0.02 to 43 μM. The device exhibited multiple beneficial characteristics for practical applications, including sustained performance over time, selective response in complex mixtures, consistent results across multiple tests, prolonged storage durability, and successful application in environmental water analysis for PE detection [101].

From the mentioned works of literature, we can infer that, in electrocatalytic sensing applications, alloying strategies offer significant benefits by enhancing performance. Noble metal alloys provide high catalytic efficiency but are often limited by cost. In contrast, noble-non-noble metal alloys achieve an optimal balance between performance and affordability.

Non-noble metal alloys, like nickel- and molybdenum-based systems, offer cost-effective alternatives with improved conductivity and durability, making them highly promising for sustainable sensing applications. Research in this area includes practical applications, such as Sasikumar et al.'s NiCeO sensor for detecting pesticide residues and Panda et al.'s PtNi alloy-based platform for monitoring hydrogen sulfide emissions in cancer cells. These studies demonstrate that alloying enhances electrocatalytic performance by improving charge transfer and increasing active sites, while also expanding the range of detectable substances, from environmental contaminants to biological markers. The findings indicate that strategic metal combinations can yield highly sensitive, stable, and versatile sensors, capable of performing effectively across diverse sample types and conditions. Alloy-based sensors thus hold strong potential for addressing complex, real-world challenges in a cost-effective and biocompatible manner.

5 Advanced sensing platforms and technologies

5.1 Techniques used in electrochemical sensors

Electrochemical sensing commonly employs two major detection approaches: voltammetric and amperometric measurements, both of which monitor changes in oxidation–reduction currents. These techniques differ fundamentally in their applied potential strategies—voltammetric methods utilize changing electrical potentials, while amperometric approaches maintain a fixed potential throughout the measurement process. In general, several analytical methods, including cyclic voltammetry, stripping voltammetry, impedance spectroscopy, and amperometric measurements, provide comprehensive information about the characteristics of target substances being studied. [102, 103]

5.2 Food sensors

The integration of electrochemical detection systems in food safety monitoring has expanded due to their exceptional ability to identify minute quantities of contamination at the nanomolar scale. An illustrative example of this technology's advancement can be found in the research of Zabihpour's team, who engineered an innovative detection platform for vanillin analysis. Their approach utilized a specialized electrode combining carbon paste with $NiFe_2O_4$ nanostructures (produced through co-precipitation) and an ionic liquid, specifically 1-hexyl-3-methylimidazolium chloride (1H3MCl). Through cyclic voltammetry analysis, the team observed oxidation signatures for vanillin at distinct potentials: + 690 mV for the unmodified carbon paste electrode and + 650 mV for their enhanced $NiFe_2O_4$/1H3MCl/CPE system. Further investigation using differential pulse voltammetry highlighted the superior catalytic performance of their modified electrode, which produced clear oxidation responses at 640 and 1050 mV [104]. Table 2 provides a comprehensive overview of some of the electrochemical techniques currently employed for specific food industry applications and target compounds.

5.3 Bio-sensors

The field of biosensors has experienced a remarkable transformation since the creation of the first glucose sensor over half a century ago. Today, these highly sensitive analytical tools are essential in various domains, from healthcare and environmental monitoring to diagnostics and research, fundamentally changing the way we gather and interpret crucial data. Glucose sensing, in particular, remains a major application of these sensors. In a recent study, Kusior and colleagues investigated enzyme-free glucose detection using copper oxide (Cu_2O) nanoparticles, examining how different crystal facets influence electrochemical performance. By controlling the addition of surfactants during synthesis, they were able to produce Cu_2O nanoparticles with precise exposure of {100} and {111} facets. When applied to glassy carbon electrodes, the {100} facet demonstrated optimal sensitivity and a broad detection range, while the {111} facet initially displayed higher activity but underwent surface alterations during glucose oxidation. Further analysis using amperometry revealed a lower response compared to cyclic voltammetry, suggesting limited active sites for glucose interaction, with distinct anodic peaks varying by facet orientation [110]. As biosensor technology continues to advance, these devices become increasingly powerful and versatile, offering greater precision, heightened sensitivity, and new capabilities that extend beyond traditional limits. Such advancements hold vast potential to transform

Table 2 Overview of electrochemical techniques used in food industry applications for target compound detection

Technique	Samples	Target	Electrode	Detection level	References
Differential pulse anodic stripping voltammetry (DPASV)	Rice	Cadmium	CPE modified with Gold nanoparticle	1.94 nmol l^{-1}	[105]
Cyclic voltammetry (CV) and Differential pulse anodic voltammetry (DPAV)	Tomato	Copper, lead and cadmium	PGE/MWCNTs	1.03 µg l^{-1} for Cd, 2.12 µg l^{-1} for Cu, and 1.62 µg l^{-1} for Pb	[106]
Differential pulse voltammetry (DPV)	Chicken	Roxarsone	Lanthanum molybdate-modified SPCE	12.4 nmol l^{-1}	[107]
Square-wave voltammetry (SWV)	Apple	Cadmium	PGE/bimetal oxide nanoparticles/graphene oxide	1.85 ng^{-1}	[108]
Linear sweep anodic stripping voltammetry (LSASV)	Beans	Copper, lead and cadmium	GCE	0.1 mol l^{-1}	[109]

disease monitoring, pollutant analysis, and medical research, paving the way for impactful breakthroughs that can significantly improve lives.

5.4 Environmental sensors

Environmental monitoring has become increasingly reliant on electrochemical sensor technologies, which play a vital role in rapidly and sensitively detecting a wide range of pollutants across air, water, and soil. These analytical tools enable the accurate identification of contaminants, including heavy metals, nitrates, and greenhouse gases like CO_2 and NO_2, facilitating real-time, on-site evaluations that bolster environmental preservation efforts. The incorporation of advanced nanomaterials, such as metal oxides and carbon-based nanostructures, has further enhanced the effectiveness of these environmental sensors. By increasing available surface area and providing abundant active sites for pollutant interactions, these nanoscale modifications have led to significant improvements in sensor sensitivity and selectivity. Recent advancements, particularly in the development of modified electrode designs and the application of voltammetric techniques, have driven additional enhancements in the detection of metal contaminants in critical matrices like drinking water and biofuels. For instance, Del Valle's team reported the use of crown ether compounds immobilized on graphite–epoxy composite electrodes, enabling simultaneous detection of cadmium, lead, and copper in synthetic water samples at the parts-per-billion (nanomolar) level through differential pulse anodic stripping voltammetry. Table 3 further outlines the analysis of metal traces in biofuel samples using similar electrochemical approaches. As environmental monitoring continues to evolve, these electrochemical sensor technologies remain at the forefront, providing rapid, sensitive, and reliable data that empowers informed decision-making and drives progress in preserving our natural ecosystems.

As an inference, advanced electrochemical sensing technologies have transformed applications across diverse fields, with each platform optimized to meet the unique demands of its specific use case.

Table 3 Analysis of metal traces in biofuel samples using some of the voltammetry techniques

Technique	Samples	Target	Electrode	Detection level	References
Differential pulse anodic stripping voltammetry (DPASV)	Biodiesel	Copper	Mercury electrode	4.69 $nmol^{-1}$	[111]
Linear sweep stripping voltammetry (LSSV)	Bio-ethanol	Iron	Nafion electrode	2.0 μmol^{-1}	[112]
Square-wave voltammetry (SWV)	Biodiesel	Calcium	Glassy carbon electrode	1.6 nmol l^{-1}	[113]
Square-wave anodic stripping voltammetry (SWASV)	Bio-ethanol	Zinc	Gas diffusion electrode	5 µg l^{-1}	[114]

One important benefit of using electrochemical sensing over spectroscopic methods is its ability to offer rapid, on-site detection with minimal instrumentation and lower cost. Voltammetric and amperometric techniques, central to electrochemical analysis, allow precise monitoring of redox reactions, aiding in the sensitive detection of contaminants within the food industry. Research studies, like Zabihpour's nanostructured electrodes for vanillin detection, demonstrate the effectiveness of electrochemical systems in identifying trace contaminants. In biosensing, advancements such as facet-engineered Cu_2O nanoparticle platforms by Kusior et al. underscore the potential of biosensors. Similarly, environmental sensors incorporating nanomaterials have driven forward real-time pollutant detection, demonstrated by Del Valle's functionalized electrodes for heavy metal detection in water. As these technologies continue to advance, electrochemical sensing stands poised to support breakthroughs in environmental preservation, healthcare, and public safety.

6 Opportunities and challenges

The field of electrochemical sensing has witnessed remarkable advancements, enabling the development of highly versatile multi-analyte detection systems. These innovative technologies are designed to simultaneously monitor a range of target analytes, a capability that has significantly broadened their applicability across diverse domains. One area that has particularly benefited from this multi-faceted approach is healthcare diagnostics. By allowing for the concurrent measurement of multiple biomarkers from a single sample, these integrated sensor platforms enhance the prospects for early disease identification and facilitate the delivery of more personalized treatment strategies. As an example, Lee et al. have engineered sensor arrays incorporating individual needle-like microelectrodes, each tailored to detect specific parameters, such as oxidation–reduction potential, dissolved oxygen levels, and phosphate concentrations, all within a compact, battery-powered device, as illustrated in Fig. 10a [115]. The continued evolution and remarkable versatility empower these technologies to deliver more holistic and informative datasets, ultimately supporting more effective decision-making and problem-solving in complex real-world scenarios.

Additionally, the push toward miniaturization has resulted in compact, high-performance sensors. This development is essential for both point-of-care (POC) diagnostics and wearable electrochemical sensors. Wearable sensors allow for continuous tracking of vital signs and biomarkers, providing individuals with real-time health insights and personalized health management. By integrating these sensors into POC systems, diagnostic procedures become more streamlined, efficient, accessible, and cost-effective, particularly in remote or resource-constrained regions. For example, He et al. recently introduced an integrated wearable device that monitors six key biomarkers in sweat simultaneously, enhancing real-time health data availability, as depicted in Fig. 10b [116]. A recent trend in point-of-care diagnostics involves the development of compact electrochemical sensors. Researchers like Kelani et al., have pioneered a disposable screen-printed sensor designed specifically for therapeutic drug monitoring, with a focus on detecting ofloxacin in biological samples, as shown in Fig. 10c. To enhance selectivity, the sensor incorporates a supramolecular calixarene ionophore. A graphene nanocomposite layer acts as an efficient ion-to-electron transducer, contributing to the sensor's stability and minimizing signal drift. Rigorous characterization, adhering to IUPAC standards, revealed a linear response range of 1 micromolar to 10 mM, with a sensitivity of 59.0 millivolts per decade. The sensor exhibited excellent recovery rates, averaging 100.18%, and a low detection limit of 600 nM. Stability studies confirmed reliable performance over an 8-week period [117]. While electrochemical sensors are making strides, there are still hurdles to overcome in terms of their stability, sensitivity, selectivity, and scalability. One major issue is stability. Real-world conditions can be tough on sensors, and factors like fouling, environmental changes, and material degradation can lead to a decline in performance over time. Therefore, boosting long-term stability is key to getting accurate and reliable results. For instance, certain MOFs used in electrocatalytic sensing applications suffer from limited chemical and structural stability. MOFs constructed with divalent metal ions, such as Cu^{2+}, Ni^{2+}, or Zn^{2+}, in combination with carboxylate linkers often demonstrate poor resistance to moisture due to the relatively weak coordination bonds they form. A well-known example is Zn^{2+}-based MOF-5, which tends to degrade upon exposure to water or humid conditions [119].

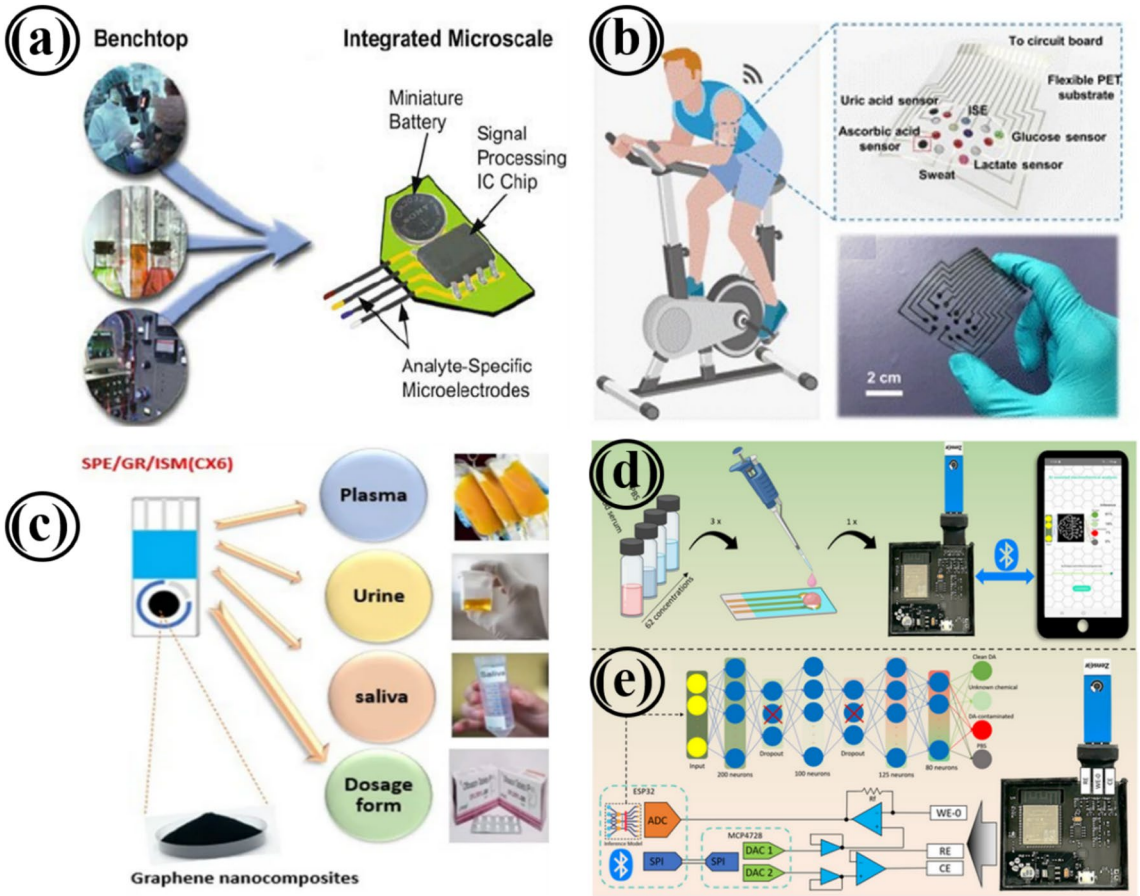

Fig. 10 **a** Illustration of transition from benchtop setup to integrated microscale device with analyte-specific microelectrodes (reproduced from [115] with permission from Springer Nature), **b** depiction of an integrated wearable device that monitors six key biomarkers in sweat (Reproduced from [116] with permission from Science under CC by 4.0 license), and **c** illustration of screen-printed/graphene nanocomposite (C-SPE/GNC/ISM) sensor 3 for detecting ofloxacin in various biological samples (reproduced from [117] with permission from SpringerOpen under CC by 4.0 license). (**d**) Edge-AI mobile-controlled device for DA, AA, and UA concentration measurements and (e) its corresponding potentiostat circuit diagram (reproduced from [118] with permission from Wiley under CC by 4.0 license)

Besides stability, sensitivity and selectivity are equally important. Sensors need to be able to detect even tiny amounts of the target substance while ignoring other stuff that might interfere. Recent breakthroughs in materials science, like using nanomaterials and specially treated electrodes, are helping to address these challenges by increasing surface area, creating more active sites, and improving the sensor's responsiveness. Scalability is another big factor, especially for mass production. It is crucial to find ways to produce sensors efficiently and cost-effectively on a large scale without sacrificing performance. This is essential for their widespread use in both consumer and industrial settings. The integration of artificial intelligence (AI) and machine learning (ML) with electrochemical sensors is opening up new possibilities for advanced sensing capabilities. AI and ML can enhance the interpretation of sensor data, improve real-time decision-making, and provide predictive analytics, thereby making electrochemical sensing more efficient and accurate. For example, a study by Filho et al. demonstrated the use of TinyML, an embedded AI model, in low-power portable systems for electrochemical applications. They explored how TinyML could distinguish between the interference of uric acid and ascorbic acid, two common electrochemically active substances, in neurotransmitter detection. This approach achieved accuracy rates of 98.1% with a 32-bit floating-point unit and 96.01% after 8-bit quantization. The research suggests that TinyML, with its

balance between memory usage and accuracy, could be pivotal in future medical devices, enabling real-time data processing with greater reliability. This innovation includes an Edge-AI device controlled by a mobile application for measuring multiple concentrations of dopamine (DA), ascorbic acid (AA), and uric acid (UA), along with the corresponding custom potentiostat circuit diagram, as illustrated in Fig. 10d and e [118]. Moreover, in environmental monitoring, AI can assist in identifying and tracking pollution sources, predicting air or water quality trends, and providing early warnings for environmental hazards. In industrial applications, AI-powered electrochemical sensors can be used for quality control, supply chain monitoring, and ensuring the safety of manufacturing processes, offering significant potential for automation and optimization. As AI, ML, and electrochemical sensing technologies continue to converge, they will undoubtedly drive innovations in diverse sectors, revolutionizing healthcare, environmental protection, and industrial processes. The future of electrochemical sensors holds exciting possibilities, with ongoing advancements in materials science, sensor design, and integration with cutting-edge technologies like artificial intelligence. Emerging materials, such as flexible and wearable sensors, along with innovations in nanomaterials, promise enhanced sensitivity, selectivity, and versatility, expanding the scope of applications from healthcare to environmental monitoring. Additionally, the integration of machine learning algorithms will enable real-time data analysis and predictive capabilities, paving the way for personalized medicine, early disease detection, and proactive environmental protection [120–122]. Moreover, AI and ML can play a pivotal role in optimizing sensor fabrication processes, enabling automated design workflows, reducing trial-and-error in material selection, and streamlining data-driven manufacturing, thereby lowering production costs and improving scalability. As these technologies evolve, electrochemical sensors will continue to transform industries, offering more accessible, efficient, and sustainable solutions for a wide range of challenges.

7 Summary and conclusion

In summary, the area of electrocatalysis has experienced major progress, largely fueled by the need for sophisticated sensing technologies that cater to essential demands in healthcare, environmental monitoring, and various industrial sectors. As scientists work diligently to investigate and enhance the design of electrocatalysts, the main objective remains to strike a balance among high sensitivity, selectivity, stability, and scalability, all while promoting environmental stewardship. This review has emphasized current developments in electrocatalyst materials and design tactics, providing insightful information for those looking to expand sensor performance capabilities. The results presented indicate that carbon-based nanomaterials, notably doped graphene and carbon nanotubes, exhibit outstanding electron transfer characteristics. These materials are becoming excellent options for high-sensitivity applications where detecting minute analytes is essential. Hybrid electrocatalysts, particularly those that merge metal nanoparticles with carbon nanostructures, also offer combined advantages by integrating the catalytic properties of metals with the strength and conductivity of carbon materials. These hybrids show major potential to improve sensor performance in detailed and challenging environments. For those engaged in this area of research, concentrating on nano-structuring methods, such as creating 3D porous frameworks or 2D layered structures through effective fabrication methods like freeze-drying and template-assisted synthesis, can greatly enhance the surface area and active sites of electrocatalysts, thereby increasing their responsiveness and effectiveness. The surface functionalization with heteroatoms (like nitrogen and sulfur) or the addition of metal oxides can further optimize pathways for electron transfer and improve selectivity, especially in the analysis of multi-component samples. Moreover, keeping scalability and cost considerations in mind, exploring alternatives to precious metals is critical. Transition metal oxides, alloy-based systems, and innovative materials like MXenes are promising and economical options for broader usage. Besides, using sustainable synthesis techniques, such as room-temperature or microwave-assisted methods, can enhance the economic and environmental viability of these materials.

As advances are made in electrocatalyst technology, incorporating AI and machine learning (ML) algorithms can considerably improve data analysis, assisting predictive insights and immediate responsiveness. Using these technologies, electrocatalysts can evolve into smart, adaptable sensors capable of tackling complex analytical challenges with precision.

In conclusion, the pursuit of optimizing electrocatalysts for sensing applications is both exciting and multi-dimensional. Recent research has increasingly focused on materials synthesized through green methods, using eco-friendly agents, such as polyphenols, citric acid, vitamins, and bio-based substances like cellulose and silk. There is also a growing interest in producing graphene from waste materials**.** By focusing on innovative materials, eco-friendly production methods, and state-of-the-art data processing, researchers can forge a pathway toward next-generation sensors that will play an important role in addressing some of the most urgent scientific and societal issues of our time.

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Author contributions

Balaji Chettiannan: investigation, conceptualization, methodology, data curation, and writing—original draft. Gowdhaman Arumugam, Stanleydhinakar Mathan, Manickam Selvaraj, and Mohammed A. Assiri: data curation and review, Ramesh Rajendran: conceptualization, visualization, methodology, review & editing, and supervision.

Data availability

No datasets were generated or analyzed during the current study.

Declarations

Conflict of interest The authors declare that they have no competing interests.

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