

Nanoparticle-based electrochemical sensing

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Abstract:

Electrochemical sensing has emerged as a potent technique for detecting and quantifying various analytes due to its inherent sensitivity, simplicity, and potential for miniaturization. Leveraging their distinctive physicochemical properties, nanoparticles have sparked a revolution in electrochemical sensing, enhancing sensing platforms' sensitivity, selectivity, and stability. This paper presents the background and significance of nanoparticle-based electrochemical sensing, along with the diverse applications of nanoparticles in science and engineering. The fundamental principles of nanoparticles in electrochemical sensing are explored, highlighting their unique advantages in electrocatalysis, charge transfer facilitation, and signal amplification. The applications of various types of nanoparticles in electrochemical sensing are discussed, elucidating the role of nanomaterials in designing advanced conduction strategies such as impedance spectroscopy, cyclic voltammetry, and amperometry.

Furthermore, this review delves into the applications of nanoparticle-based electrochemical sensors across domains, including gas detection, environmental monitoring, and the field of biosensors. The practicality of these sensors in real-world scenarios is showcased through case studies, emphasizing their rapid response, high specificity, and potential for multi-analyte detection. The challenges and prospects of this field are also examined. Addressing nanoparticle stability, reproducibility, and scalability issues is crucial for the successful commercial translation of these sensing platforms. In conclusion, this review underscores the transformative impact of nanoparticles on the landscape of electrochemical sensing. Their multifunctionality, coupled with evolving conduction methodologies, promises to develop ultra-sensitive and reliable sensing platforms that contribute to advancements across various application domains. With the continuous evolution of nanoparticle synthesis techniques, electrochemical sensing is poised to achieve new frontiers of sensitivity and specificity.

Keywords: Electrochemical Sensing, Nanoparticles, Nanomaterials, Biosensors, Application Domains

1. Introduction

With the rapid advancement of nanotechnology, the applications of nanoparticles across various scientific domains continue to expand. In analytical chemistry, electrochemical sensing based on nanoparticles has emerged as a captivating area of research. Nanoparticles, owing to their distinct physical, chemical, and electronic properties and the advantage of increased surface area-to-volume ratio, offer extensive possibilities for electrochemical sensing. This paper primarily focuses on electrochemical sensing technology utilizing nanoparticles and explores their applications in fields such as analytical chemistry, biosensing, and environmental monitoring.

In this realm, metallic, semiconductor, and magnetic nanoparticles are widely employed as functional units for highly sensitive and selective analysis. Due to their excellent conductivity and catalytic performance, metallic nanoparticles enable the amplification and enhancement of electrochemical signals at electrode surfaces. Semiconductor nanoparticles, through photoelectrochemical mechanisms, facilitate efficient

detection of specific analytes. Simultaneously, magnetic nanoparticles find unique applications in biological analysis, contributing to the separation and amplified electrochemical sensing of biorecognition complexes, presenting novel avenues for biosensor development.

This paper introduces nanoparticles' fundamental principles and advantages in electrochemical sensing, encompassing their roles in increasing electrode surface area, catalytic reactions, and signal amplification. Subsequently, various applications of distinct types of nanoparticles in electrochemical sensing are explored, spanning fields such as gas sensors, biosensors, and environmental monitoring. Finally, current challenges and future directions are discussed, aiming to further propel nanoparticle-based electrochemical sensing technology's widespread application in practical scenarios.

Through in-depth research and exploration, we can better comprehend the potential of electrochemical sensing based on nanoparticles and the opportunities and challenges it presents in addressing intricate analytical and detection issues. These endeavors will contribute to the synergy between nanotechnology and electrochemical

sensing, fostering breakthroughs in scientific research and practical applications.

2. Basic principles of nanoparticles in electrochemical sensing

2.1 Electrochemical Aptamer Sensors

Electrochemical sensors are widely recognized for their cost-effectiveness, rapid response, excellent selectivity, high sensitivity, and simplicity. Among them, electrochemical aptamer sensors represent a prevalent class of sensors that combine the benefits of electrochemical sensing with the specificity of aptamer-based molecular recognition. These sensors hold immense potential in various applications, including environmental monitoring, pharmaceutical analysis, and disease diagnosis [1].

Electrochemical aptamer sensors incorporate aptamers as molecular recognition elements onto sensor surfaces. These aptamers interact specifically with target analytes, generating corresponding biological signals, which are then transduced into detectable electrochemical signals through a transducer (e.g., measuring electrodes, thermistors, and field-effect transistors). The change in electrochemical signals before and after the interaction between the aptamer and target analyte allows for quantitative detection of the target analyte.

2.2 Electron Transfer and Interface Charge Transfer Mechanisms between Nanoparticles and Electrode Surfaces

The electron transfer and interface charge transfer mechanisms between nanoparticles and electrode surfaces refer to the processes where electrons and charges are exchanged during electrochemical reactions. These mechanisms play a critical role in nanoscale electrochemistry, exhibiting distinct characteristics compared to conventional macroscopic electrochemical reactions. Electron transfer is influenced by the small size of nanoparticles, which enhances electron interactions between surface atoms and molecules. This can result in electron transfer between nanoparticles and electrode surfaces. These processes are pivotal in determining the reaction rates and mechanisms. Interface charge transfer involves the movement of charges between nanoparticles and electrode surfaces. These positive (cations) or negative (anions) charges play crucial roles in electrochemical reactions. Interface charge transfer affects the distribution of electrode surface potential and reaction kinetics. Understanding these electron transfer and interface charge transfer mechanisms is essential in enhancing the performance of nanoparticle-modified electrodes, leading to improved electrochemical sensors

with enhanced signal amplification, selectivity, and sensitivity [2].

2.3 Catalytic and Signal Amplification Mechanisms of Nanoparticles at the Electrochemical Interface

Researchers commonly utilize strategies to amplify detection signals in constructing electrochemical aptamer sensors to further enhance sensor sensitivity. There are generally two ways to amplify sensor signals. One approach involves amplifying specific signals at the sensor interface using nanoparticles and enzyme catalysis techniques. The other approach involves the secondary amplification of output signals through more sensitive detection instruments. Comparatively, the former approach involves proportional amplification of both detection signals and noise, making research into novel signal amplification technologies and combining various signal amplification techniques highly significant for enhancing sensor sensitivity.

Due to their unique physicochemical properties (quantum size effects, surface effects, small size effects, macroscopic quantum tunneling effects), Nanoparticles exhibit distinctive electrochemical and optical characteristics. They are widely employed as modifiers for sensor interfaces and solid carriers for biomolecules in constructing biosensors. The small size and surface effects of nanoparticles result in large specific surface areas and high surface free energies, which enhance chemical reactivity upon interaction with other molecules and facilitate higher loading of biomolecules. The macroscopic quantum tunneling effects of nanoparticles promote efficient electron transfer. When nanoparticles are used as solid electrode carriers, they effectively increase the electrode's specific surface area, enhance conductivity, and thus improve the sensor's response rate.

Furthermore, nanoparticles offer excellent biocompatibility, creating favorable microenvironments for biomolecules and maintaining their activity, thus enhancing sensor stability. Some nanoparticles have also shown strong catalytic activity towards specific substrates. Using the signal amplification effect of nanogold, a sandwich-type electrochemical aptamer sensor was developed for highly sensitive detection of platelet-derived growth factor (PDGF-BB) (Fig.1). Initially, a large amount of PDGF-BB aptamer is immobilized onto nanogold to form functionalized nanocomposites. A sandwich reaction introduces these onto a gold electrode surface, creating a capture aptamer-target analyte-nanogold composite structure. The negatively charged aptamers on the nanogold surface facilitate the adsorption of a significant amount of electroactive $[\text{Ru}(\text{NH}_3)_5\text{Cl}]_2$. As the target analyte concentration increases, the

electrochemical signal gradually rises, allowing the detection limit of PDGF-BB to reach 0.01 pmol/L [3].

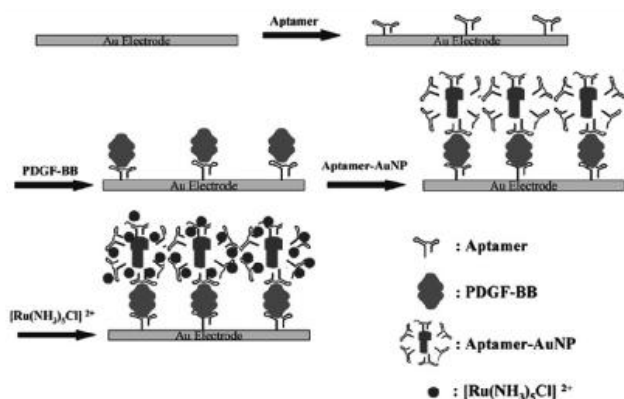


Fig.1 Schematic diagram of the principle for the electrochemical apt sensor with gold nanoparticles for amplification [4]

Moreover, carbon nanotubes' unique hollow tubular structure endows them with favorable properties such as large surface area, heat and thermal conductivity, excellent strength, and good biocompatibility. Consequently, they are widely utilized in the construction of electrochemical aptamer sensors. Graphene, composed of carbon atoms in a honeycomb lattice plane with sp² hybridization, is a two-dimensional material with just one carbon atom thickness. It exhibits a larger specific surface area and excellent conductivity. A sandwich-type coagulation factor aptamer sensor was developed by combining multiple nanoparticle materials for signal amplification. Graphene's large surface area is initially used to immobilize coagulation factor aptamer 1. A sandwich reaction combines the aptamer two complexes following specific binding with the coagulation factor. This complex employs SiO₂-modified carbon nanotubes for amplification, effectively increasing the loading capacity of the electroactive substance thionine. The cooperative catalytic effect of surface-nano-platinum and porphyrin iron enhances the detection limit of the target coagulation factor to 50 fmol/L [5].

Furthermore, carbon nanotubes with excellent conductivity and large surface area are employed to immobilize a considerable amount of alkaline phosphatase (ALP) and labeled coagulation factor aptamer. This composite is introduced onto the electrode surface through a sandwich reaction. Multiple catalytic amplifications of the detection signal occur through ALP and cardiac troponin in the detection solution, with two substrates present, enabling the sensor to achieve a detection limit of 8.3 fmol/L for the coagulation factor. In summary, nanoparticles primarily enhance electrochemical response signals [6].

3. Applications of Different Types of Nanoparticles in Electrochemical Sensing

3.1 Metallic nanoparticles

Catalytic electrochemical sensors can detect the conversion of chemical compounds, which can be directly or indirectly correlated with the concentration of analytes in a sample. The type of catalyst used to drive the chemical reaction categorizes sensors as either enzymatic or non-enzymatic. Despite many enzymatic biosensors offering high sensitivity and selectivity, they often exhibit significant drawbacks, limiting their scope of application. Common limitations include a short shelf life due to enzyme fragility, stringent operating conditions stemming from enzymes' low tolerance to environmental fluctuations, low reproducibility, repeatability, and high production costs. Consequently, research focus has shifted towards non-enzymatic sensors, including devices that exploit the unique properties of metal nanoparticles. It has been demonstrated that certain nanoparticles, also referred to as enzymes, can catalyze chemical reactions due to their activity resembling oxidases, peroxidases, catalases, and/or superoxide dismutase (Fig. 2). Consequently, they serve as a more affordable, robust, easy-to-handle, and stable alternative to enzymes. The thermal stability of nanoparticles becomes particularly advantageous when designing devices for storage in resource-constrained settings, such as regions with limited access to refrigeration, like rural areas in sub-Saharan Africa. The heightened stability of biosensors enables prolonged storage and facilitates distribution to off-grid communities, for instance, in rural parts of sub-Saharan Africa.

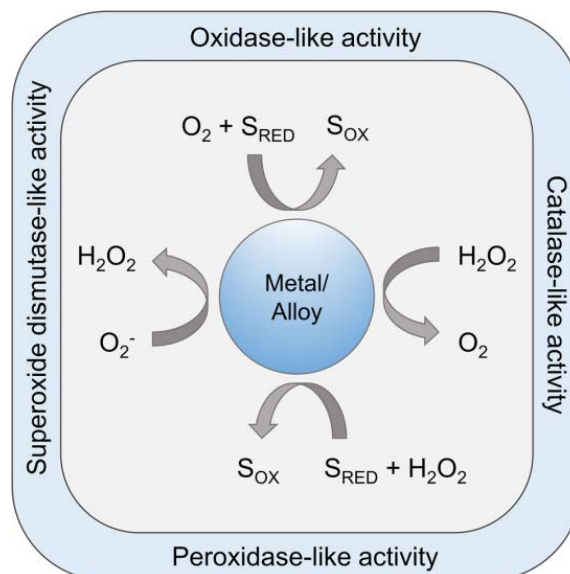


Fig.2 Possible enzyme-like catalytic activities of metal/metal alloy nanoparticles. Figure adapted with permission from ref. [7]; copyright © 2015, American Chemical Society

The size, shape, morphological uniformity, chemical purity, dispersity, and environmental factors of metal nanoparticles dictate their catalytic performance and applicability. These parameters can be adjusted by altering synthesis conditions, such as temperature, type and quantity of metal precursors, type and quantity of reducing agents, and stabilizers (capping agents). In some cases, microporous carriers can control the catalytic processes involving embedded nanoparticles.

Metal nanoparticles can assume various roles in sensing devices. For example, they can substitute enzymes like horseradish peroxidase (HRP) in sandwich-like assays (Fig.3a). The utilization of nanoparticles in immunosensors and immunoassays in this context has been subject to review. In other applications, the activity of metal nanoparticles is modulated by an analyte, enabling the correlation of catalyzed reaction rates with analyte concentrations (Fig. 3b). In this scenario, the substrate for the nanoparticle-catalyzed reaction is abundant. In contrast, the analyte is an inhibitor or activator (enhancer). Sensors based on this mechanism can be employed for heavy metal detection. Lastly, metal nanoparticles can directly catalyze analyte conversions, facilitating their electrochemical detection [8].

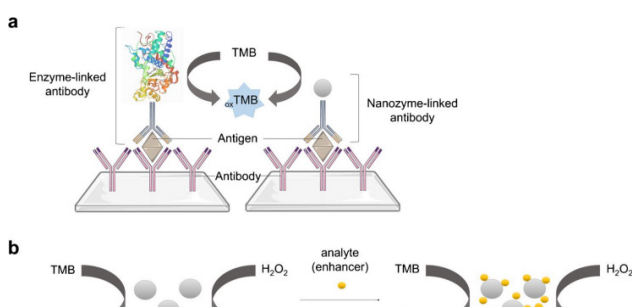


Fig. 3 Examples of metal NP application in sensors. a Metal NP as a signal amplifier.

b detection of an analyte based on its modulation effect on metal NP catalytic activity. [9]

3.2 Semiconductor nanoparticles

Semiconductor nanomaterials have extensive applications in gas sensing due to their high electron mobility, large surface-to-volume ratio, charge confinement capability, crystallinity, chemical stability, and thermal stability. Semiconductors or nanostructured metal oxides exhibit high reactivity towards target pollutant gases, leading

to electrical resistance changes. However, they lack specificity and selectivity, operate at high temperatures, involve high-energy interactions, and may not be universally suitable for all environments. Therefore, searching for high-performance semiconductor sensors with high sensitivity and selectivity is crucial and has garnered significant interest.

Colloidal quantum dots containing materials such as zinc oxide, PbSe, tin oxide, tungsten trioxide, and lead sulfide have been employed for the selective detection of substances or gases at room temperature, such as ammonia, nitrogen dioxide, LPG, methane, ethanol, hydrogen sulfide, etc. Nano-label sensors decorated with tin oxide can be used to detect drunk driving and in food quality and safety systems. The combination of tin oxide with reduced graphene oxide (rGO) has been applied for electrochemical detection of heavy metal ions in drinking water, offering high selectivity. The non-toxic properties of titanium dioxide and zinc oxide nanostructures make them suitable for medical and pharmaceutical sensing applications, as they exhibit biocompatibility and easy binding with biological entities. As biosensors, they can detect dangerous levels of chronic diseases, Zika virus in urine, and even coronaviruses in liquid samples.

Due to their various morphologies, these nanomaterials exhibit different behaviors and responses towards pollutants or molecules, influenced by the structure or morphology's impact on the electron transfer rate for pollutants or molecules. Some metal oxide semiconductor nanoparticles have shown biosensing behavior influenced by their crystalline nature, electronic band structures, and molecule interactions. P-type nickel oxide nanostructures have also demonstrated efficient sensing of formaldehyde gas due to the enhanced electrochemical activity brought about by the large surface area of the semiconductor electrode material within the electrolyte system [10].

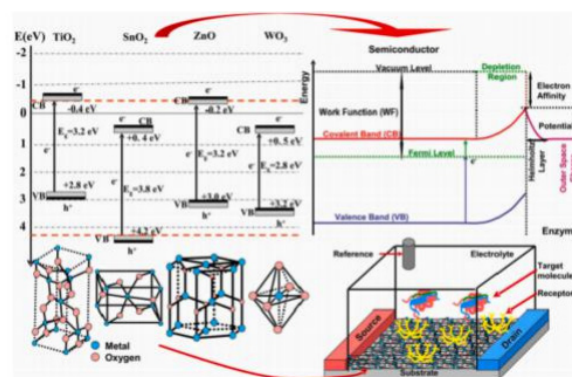


Fig.4 Biosensing properties of several semiconductors, showing crystal structures, band structures, and biosensing interactions with biomolecular [11]

3.3 Magnetic nanoparticles

Magnetic nanoparticles play a crucial role in biomolecule recognition, separation, and analysis. These nanoparticles are widely employed as carriers for encapsulating enzymes, proteins, peptides, antibodies, antigens, and other biomolecules, thus finding extensive applications in biomedicine and immunodiagnostic fields. Particularly noteworthy is their significant role in the separation and amplification of electrochemical signals in biorecognition complexes.

In this process, magnetic microspheres serve as carriers, enabling specific biomolecules to be immobilized on their surfaces. These modified nanoparticles can recognize and capture specific biomolecules, facilitating their separation from complex mixtures. This technique leverages the properties of magnetic nanoparticles and an external magnetic field to achieve efficient separation. What's more, these biorecognition complexes facilitate the isolation of target molecules and amplify electrochemical signals.

By immobilizing biomolecules onto magnetic nanoparticles, signal amplification in electrochemical detection can be achieved. When these modified nanoparticles bind with target molecules, the electrochemical signal experiences significant enhancement. This amplification effect enables us to detect target biomolecules more sensitively, even at extremely low concentrations. As such, the ability of magnetic nanoparticles to amplify electrochemical signals provides a powerful tool for detecting and analyzing biomolecules, holding significant promise for disease diagnosis and biological research.

In conclusion, applying magnetic nanoparticles in recognizing and separating biomolecules is a captivating field characterized by the ability to capture and separate biomolecules while achieving high-sensitivity detection through the amplification of electrochemical signals. This technology has broad applications in biomedicine, immunology, and biochemical analysis, offering substantial potential for various fields [12].

4. Applications of Nanoparticle-based Electrochemical Sensing in Various Fields

4.1 Utilization of nanoparticles in gas sensors

Air quality has a significant impact on human health and socio-economic well-being, making the control of environmental gas pollution increasingly important. Novel gas-sensitive devices based on nanostructured thin films promise to offer higher sensitivity, selectivity, and faster response times. Techniques such as mass spectrometry,

infrared spectroscopy, and laser absorption spectroscopy can accurately detect gas pollutants within mixtures. However, using these conventional analytical instruments often proves time-consuming, expensive, and generally unsuitable for remote areas and outdoor conditions.

In recent years, polymer-based gas sensors have been developed with the advancement of organic electronics. Configuring conductive polymers, such as polyaniline and polypyrrole, in a pattern similar to organic field-effect transistors on an insulating substrate can detect extremely low concentrations of various organic compounds. This includes compounds like trinitrotoluene and other nitroaromatic explosives.

The sensitivity of polymer-based gas sensors depends on the nanostructure of the sensing area. Introducing metal oxide nanoparticles, graphene sheets, or carbon nanotubes can achieve tunable selectivity of the sensor.

A practical solution for high-sensitivity gas sensing lies in carbon-based nanomaterials. Pristine graphene, an atom-thick carbon material with zero bandgap and semi-metallic electronic properties, typically requires further surface functionalization to enhance its ability to detect gas molecules [13].

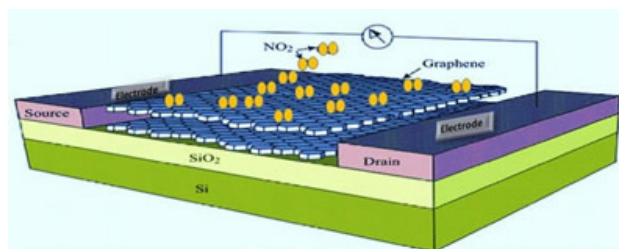


Fig 5 A Schematic diagram of the graphene-based thin-film gas sensor [14]

Graphene derivatives, such as functional GO and carbon nanotubes, have great prospects in gas detection because of their extremely high surface-to-volume ratio so that the entire thin layer of atoms can be affected by gas molecules. Furthermore, chemically modified surfaces of functional GO enhance the absorption of gas molecules and improve delivery to sensor electrodes [15].

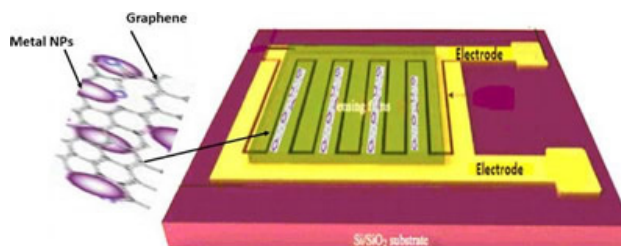


Fig.6 Schematic representation of the gas sensor with fork finger electrodes and metal nanoparticles modified graphene channels[16]

4.2 Nanoparticle-based biosensors

The accurate detection of small quantities of disease-related target proteins, particularly cancer and tumor biomarkers in early stages, is crucial for various applications such as clinical diagnosis, biomedical research, food quality control, and environmental analysis. Immunosensors are being developed to identify these target substances in bodily samples and biological matrices. The goal is to create immunosensors that combine selectivity and sensitivity in a multiplexing capacity while remaining simple and cost-effective.

Different electroanalytical techniques, including electrochemical, optical, and microgravimetric methods, are commonly used. Electrochemical sensing platforms combined with nano signal amplification strategies offer advantages such as high selectivity, sensitivity, potential for miniaturization, portability, reduced complexity, and quick operation. Various nanomaterials like gold nanoparticles, carbon nanostructures, magnetic nanoparticles, and semiconductor quantum dots are utilized to construct these immunosensors. Due to their ease of preparation, electrocatalytic effects, and stability, carbon nanostructures are ideal for electroanalysis. Their large surface area, abundant functional groups, and biocompatibility make them suitable for immobilizing biomarkers for specific antigen-antibody recognition.

An electrochemical immunoassay was developed in 2009 to detect the carcinoembryonic antigen (CEA) cancer marker precisely. A sandwich-type sensing platform was created using anti-CEA antibodies immobilized on a screen-printed graphite electrode modified with carbon nanoparticles/poly (ethylene imine) (CNP-PEI). The increased active surface area due to CNPs and the amino groups in PEI polymer chains facilitated antibody loading, enhancing electrode surface accommodation. Cadmium sulfide quantum dots (CdS QDs) labeled with secondary α CEA antibodies were used as bio tracers to enhance the electrochemical signal and detection level. CNPs not only improved electron transfer rates but also enhanced sensitivity. The detection range for CEA was 0.032 to 10 ng/mL, with a detection limit of 32 pg/mL, achieved using square wave anodic stripping voltammetry (SWASV). This modified electrode was effectively used to measure urinary CEA levels, making it suitable for CEA analysis in cases of urothelial carcinoma [17].

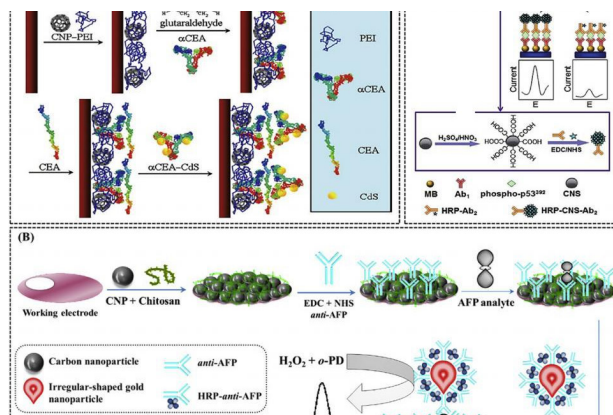


Fig.7 Schematic illustrations of (A) the assembly and amplified strategy for the detection of CEA using the α CEA-CdS bio tracer on the surface of CNP-PEI/SPGE [18]

5. Challenges and Prospects

The electrochemical sensing technology based on nanoparticles holds potential across various application domains, yet it also faces stability, selectivity, and reproducibility challenges. However, through future development directions, these challenges can be effectively addressed.

Firstly, the significance of nanoparticle material design cannot be overlooked. By meticulously designing nanoparticles' shape, size, and surface properties, sensor performance can be optimized, enhancing selectivity and sensitivity.

Secondly, innovative sensor structures are key to improving sensor performance. Novel structures such as core-shell configurations and porous materials can offer greater surface area, augmenting the adsorption and transmission of target molecules, thus elevating sensor signal response and selectivity.

Additionally, interdisciplinary collaboration is pivotal for advancing nanoparticle-based sensor technology. Collaboration among experts from different disciplinary domains can amalgamate knowledge and collectively tackle intricate issues, fostering innovation in sensing technology.

The advancement of intelligent processing and data analytics will enhance sensor analytical capabilities. Through technologies like machine learning and artificial intelligence, pattern and trend recognition will reduce the impact of interference, facilitating real-time data analysis and decision-making.

Lastly, developing portable and wearable sensors will bring sensing technology closer to practical applications. These sensors can facilitate real-time monitoring in medical diagnosis and environmental monitoring, enabling

continuous data collection and analysis.

In conclusion, through meticulous nanoparticle material design, innovative sensor structures, interdisciplinary collaboration, intelligent data processing, and the development of portable sensors, electrochemical sensing technology based on nanoparticles will continually overcome existing challenges, offering more precise, stable, and practical sensing solutions across diverse domains.

6. Conclusion

Electrochemical sensing based on nanoparticle technology showcases remarkable advantages and extensive potential applications in contemporary scientific research. This method harnesses the unique properties of nanoscale materials, offering new avenues for catalytic performance and signal amplification effects.

Regarding catalytic performance, the standout feature of nanoparticles is their substantial surface area-to-volume ratio. By modulating nanoparticles' size, shape, and crystal structure, researchers can create highly active surface sites, enabling precise control over catalytic reactions. For instance, metal nanoparticles, such as platinum nanoparticles, are commonly used as catalysts, excelling in redox reactions. Ingeniously designed nanoparticles like platinum ones can enhance the efficiency of oxygen reduction reactions in fuel cells, thereby improving energy conversion efficiency.

Signal amplification constitutes another pivotal advantage. Nanoparticles can magnify signals in electrochemical sensors, converting weak detection signals into visibly distinct ones. This phenomenon finds application in biosensing, where nanoparticles can be labeled onto biomolecules like proteins and nucleic acids, enabling highly sensitive detection of low-concentration molecules. As these nanoparticle labels participate in electrochemical reactions, their conductive properties lead to significant signal amplification, lowering the detection limits of target molecules.

The future application prospects are exceedingly broad. In medicine, nanoparticle-based electrochemical sensing can be employed for early disease diagnosis, drug screening, and personalized treatment. This technology can be used in environmental monitoring to track pollutants, monitor water quality, and assess air quality, among others. In energy conversion and storage, the catalytic capabilities of nanoparticles will aid in developing more efficient fuel cells, solar cells, and battery energy storage systems.

In conclusion, electrochemical sensing based on nanoparticle technology finds crucial applications in existing domains and holds immense potential in emerging

fields. Utilizing nanoscale effects, meticulously designed nanoparticles enhance catalytic performance and achieve signal amplification, revolutionizing scientific research and practical applications. With ongoing technological advancements, we can anticipate continuous innovation in this field, providing cutting-edge solutions for addressing complex challenges. Practical applications

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