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Synthesis and Characterization of Nanomaterials for Application in Cost-Effective Electrochemical Devices

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Abstract: Nanomaterials have gained significant attention as a remarkable class of materials due to their unique properties and the fact that they encompass a wide range of samples with at least one dimension ranging from 1 to 100 nm. The deliberate design of nanoparticles enables the achievement of extremely large surface areas. In the field of cost-effective electrochemical devices for energy storage and conversion applications, nanomaterials have emerged as a key area of research. Their exceptional physical and chemical properties have led to extensive investigations aimed at improving the performance and cost-effectiveness of electrochemical devices, including batteries, supercapacitors, and fuel cells. The continuous development and enhancement of these high-performance materials are driven by the demand for enhanced productivity, connectivity, and sustainability at a reduced cost. This review focuses on the electrochemical performance of electrodes, energy storage, and electrochemical sensors (ES) based on nanotechnology. It discusses the application of nanotechnology in electrochemistry for water purification and the fate of substances in water, while also introducing green nanotechnology and cost-effective, high-fidelity product creation through electrochemical methods. The study emphasizes the synthesis of novel nanomaterials, such as metal-organic frameworks (MOFs), covalent organic frameworks (COFs), and MXenes, with applications in electrochemical devices. Furthermore, it explores the integration of nanostructures with electrochemical systems in economically significant and future applications, along with the challenges faced by nanotechnologybased industries. The paper also explores the interplay between nanomaterials and biosensors, which play a vital role in electrochemical devices. Overall, this review provides a comprehensive overview of the significance of nanomaterials in the development of cost-effective electrochemical devices for energy storage and conversion. It highlights the need for further research in this rapidly evolving field and serves as a valuable resource for researchers and engineers interested in the latest advancements in nanomaterials for electrochemical devices.

Keywords: nanomaterials; green nanotechnology; electrochemical devices; cost-effective; biosensors; energy storage

1. Introduction

The development of cost-effective electrochemical devices is crucial for efficient energy storage and conversion, which is essential for sustainable development. Nanomaterials have demonstrated great potential in enhancing the performance of electrochemical devices, but the synthesis of these materials for large-scale applications remains a challenge [1]. Several synthesis methods have been developed to address this challenge, including solution-based, template-based, and microwave-assisted synthesis. These methods have shown promise in producing high-quality nanomaterials in large quantities, and they have the potential to reduce production costs. Integrating nanomaterials into electrochemical devices is also a significant challenge, as the properties of nanomaterials differ significantly from those of their bulk counterparts [2]. The interface between the nanomaterials and other components of the electrochemical device is critical, and it can significantly influence device



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Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). performance and efficiency. Therefore, optimizing the integration of nanomaterials into electrochemical devices is crucial for realizing their full potential.

The synthesis of nanomaterials on a large scale for practical applications remains a challenge. However, significant progress has been made in developing various synthesis methods, such as solution-based methods, template-based methods, and microwave-assisted synthesis [3,4]. These approaches show promise in producing high-quality nanomaterials in large quantities while also potentially reducing production costs.

Integrating nanomaterials into electrochemical devices presents another significant challenge. The properties of nanomaterials differ significantly from their bulk counterparts, necessitating careful consideration of their integration with other components of the device. The interface between nanomaterials and the device's other constituents critically influences device performance and efficiency [5]. Therefore, optimizing the integration of nanomaterials is crucial to fully exploit their potential in electrochemical devices [5].

One key aspect that distinguishes nanomaterials in electrochemical applications is their electrical properties. Nanomaterials can be broadly classified as conductive, semiconductive, or insulating based on their ability to conduct electricity. Conductive nanomaterials, such as carbon nanotubes and graphene, exhibit high electrical conductivity and find extensive use as current collectors, electrodes, and catalysts [6]. Semi-conductive nanomaterials, such as metal oxides and sulfides, possess moderate electrical conductivity and can serve as active materials for energy storage and conversion. Insulating nanomaterials, including metal–organic frameworks (MOFs) and covalent organic frameworks (COFs), offer low electrical conductivity but provide high surface area and large pore volumes, making them suitable support materials for electrochemical devices [7].

Moreover, the composition of nanomaterials also affects their electrical properties. Innovative nanomaterials like MOFs, COFs, and MXenes have gained considerable attention due to their tunable electrical properties and high surface areas, making them promising candidates for electrochemical device applications [7].

Overall, the unique properties of nanomaterials make them attractive for use in electrochemical devices, and their potential for cost-effectiveness makes them even more appealing. By understanding the different types of nanomaterials and their properties, researchers can continue to develop new and improved electrochemical devices with enhanced performance and cost-effectiveness. Therefore, this review will highlight electrochemical techniques, energy storage, as well as electrochemical sensors based on nanotechnology. As well as the integration of nanostructures with the electrochemical system in applications of economic interest. It will shed light on the future of these applications and the challenges of environmental sustainability. Moreover, innovative sustainable materials have been used in nuclear applications such as cellulosic waste in treatment [8,9] or cement mixed with plant waste [10,11], natural clay [12,13], bitumen [14,15] or with asphaltene and polymer [16], cement waste [17], and glass [18–20] for waste stabilization and radiation shielding.

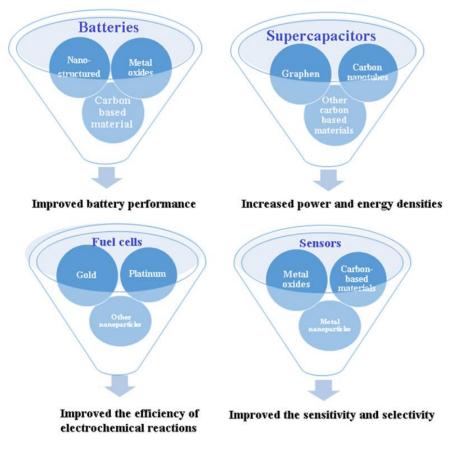
2. Classification of Nanomaterials

Nanomaterials can be classified based on their dimensions, such as 0D, 1D, and 2D structures. Zero-dimensional (0D) nanomaterials are spherical nanoparticles and are the simplest type of nanomaterials. Examples of 0D nanomaterials include metal nanoparticles like gold and silver, semiconductor nanoparticles like quantum dots, and oxide nanoparticles like titanium dioxide [21]. These nanomaterials have unique physical and chemical properties compared to their bulk counterparts, such as a high surface-to-volume ratio and quantum confinement effects.

Moving on to one-dimensional (1D) nanomaterials, they have one dimension in the nanoscale range. Examples of 1D nanomaterials include nanowires, nanotubes, and nanorods. Carbon nanotubes, silicon nanowires, and zinc oxide nanorods are some common examples of 1D nanomaterials. These nanomaterials are appealing for use in a wide range of electrical, photonic, and energy storage applications because of their excellent mechanical, electronic, and optical capabilities that result from their one-dimensional structure. Finally, two-dimensional (2D) nanomaterials have two dimensions in the nanoscale range. Examples of 2D nanomaterials include graphene, transition metal dichalcogenides (TMDs) like MoS₂ and WS₂, and black phosphorus [22]. These nanomaterials have unique electronic, mechanical, and optical properties that make them promising candidates for various applications, including sensors, energy storage, and electronic devices.

Nanomaterials can also be classified based on their electric properties, such as insulating, semiconducting, and metal-like properties. Insulating nanomaterials do not conduct electricity and have a wide band gap. Examples of insulating nanomaterials include metal oxides like titanium dioxide, zinc oxide, and aluminum oxide. These nanomaterials are widely used in various applications, including catalysis, energy storage, and environmental remediation.

Scheme 1 and Table 1 summarize the use of nanomaterials in various types of electrochemical devices.



Scheme 1. Nanomaterials in various types of electrochemical devices. Made by authors. .

Table 1. Nanomaterials Used in Various Electrochemical Devices.

Electrochemical Device	Nanomaterials Used	anomaterials Used Applications	
Batteries	Nanostructured metals, metal oxides, and carbon-based materials, MOFs, COFs, MXenes.	Electrode materials to improve performance, increase surface area, improve conductivity, and provide higher energy and power densities	[20,21]
Supercapacitors	Carbon nanotubes, graphene, and other carbon-based materials, MOFs, COFs, and MXenes.	Electrode materials to increase surface area, improve conductivity, and provide high power and energy densities	[22–26]
Fuel Cells	Platinum, gold, and other metal nanoparticles, MOFs, COFs, MXenes.	Catalysts to improve the efficiency of electrochemical reactions that generate electricity	[27]

Electrochemical Device	Nanomaterials Used	Applications	References
Sensors	Metal nanoparticles, metal oxides, and carbon-based materials, MOFs, COFs, MXenes	Sensing elements to improve sensitivity and selectivity due to their high surface area, high catalytic activity, and unique optical and electrical properties	[28]

Table 1. Cont.

Semiconducting nanomaterials have intermediate electrical conductivity, and their conductivity can be tuned by doping or changing their size. Examples of semiconducting nanomaterials include quantum dots, carbon nanotubes, and TMDs [29]. Various electrochemical devices can benefit from the use of certain nanomaterials because of their distinctive electrical and optical capabilities in the fields of electronics, photonics, and energy conversion. For example, metal nanoparticles like gold and silver have been used in electrochemical sensing and catalysis. Carbon nanotubes have shown potential as electrodes in batteries and fuel cells, while graphene has shown promise as a material for supercapacitors. Some studies focused on the use of graphene as a means of stabilizing the interface between silicon-based electrodes and electrolytes to achieve a stable cycle life. While coating graphene on the surface of nanostructured silicon electrodes is an effective approach, most of the graphene-supported anode reports involve silicon nanoparticle structures. The study by Yang et al. [30] suggested that coating graphene layers on silicon nanowires could be a viable option for stabilizing the interface between silicon-based electrodes and electrolytes. After 500 charge-discharge cycles, the resultant silicon nanowires showed excellent Coulombic efficiencies of over 99% and a huge reversible capacity of 1650 mA h g1. The nanowires were composed of silicon carbide nanocrystals and surrounded by a homogenous graphene shell. Furthermore, Si nanowires were formed in situ by a gold-catalyzed procedure (Figure 1). These nanowires, when backed by graphene, showed an initial reversible lithium extraction capacity of 2009 mA h g1, and after 30 cycles at 420 mA g1, a reversible capacity of 1400 mA h g1 was achieved. These findings demonstrate the potential of graphene-supported silicon nanowires and suggest a promising avenue for further research in this area. Furthermore, insulating nanomaterials like titanium dioxide have been used as anodes in lithium-ion batteries, while semiconducting nanomaterials like quantum dots have been used in solar cells [2].

To better understand and categorize the different types of nanomaterials that can be used in electrochemical applications, a classification based on different classes of materials has been proposed. These classes include carbon-based nanomaterials, metal oxides, and hydroxides, conducting polymers, and hybrid materials. Carbon-based nanomaterials such as graphene, carbon nanotubes, and fullerenes have been extensively studied and show great potential for improving the performance of electrochemical devices, particularly in energy storage applications such as batteries and supercapacitors [31].

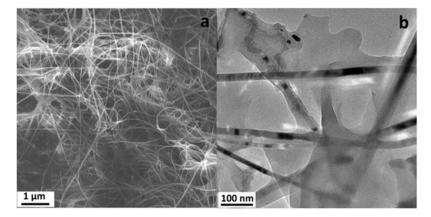


Figure 1. GNS-supported Si nanowires: (a) SEM image and (b) TEM image. Reprinted with permission from ref. [32] license 5535970520510. Copyright Elsevier in 25 April 2023.

3. Applications of Nanomaterials in Electrochemical Devices

Metal oxides and hydroxides, including titanium dioxide, zinc oxide, and iron oxide, have also shown promise in electrochemical devices due to their high surface area and redox properties [33]. Conducting polymers such as polyaniline and polypyrrole offer unique properties such as high conductivity and tunable redox potential, making them suitable for a range of electrochemical applications. Hybrid materials, which combine two or more classes of nanomaterials, have also been investigated and offer the potential for improved performance and functionality. When electroactive materials are nanostructured, significant alterations are made to the devices' electro-chemical characteristics. Assembled from quasi-zero-dimensional structures or building blocks (nanoparticles), two- and threedimensional nanometer-sized structures like dye-sensitized solar cells (DSSC) are one example. Moreover, 0D nanoparticles and nanodots have attracted significant attention for their potential use in batteries due to their small size and high surface area. In particular, 0D nanoparticles and nanodots exhibit unique physical and chemical properties that make them promising candidates for electrode materials in batteries. These materials can significantly improve the performance of batteries by providing high capacity, fast charging and discharging rates, and a long cycle life [34].

One advantage of 0D nanoparticles and nanodots is their high surface area, which provides numerous active sites for electrochemical reactions. This property can enhance the capacity of batteries by increasing the amount of active material available for electrochemical reactions [35]. Additionally, the small size of 0D nanoparticles and nanodots enables fast diffusion of lithium ions, leading to faster charging and discharging rates. More efficient ion transfer and enhanced electrochemical performance are two benefits of using 0D nanoparticles and nanodots in batteries, which benefit from their high surface area-to-volume ratio [35].

Research has been conducted on various types of 0D nanoparticles and nanodots, including metal oxide nanoparticles, carbon nanodots, and quantum dots. For instance, studies have shown that tin oxide nanoparticles can improve the cycling stability and rate capability of lithium-ion batteries, while carbon nanodots have been proven to enhance the capacity and rate performance of sodium-ion batteries [36]. Quantum dots have also been investigated for their potential use in next-generation batteries because of their unique electronic and optical properties [36]. Moreover, 0D nanoparticles and nanodots have unique properties that make them attractive for use in various electrochemical devices beyond just batteries. For instance, their high surface area-to-volume ratio and tunable size and shape make them ideal for use in sensors, catalysts, and supercapacitors. Additionally, their small size can facilitate electron transfer and enhance ion diffusion, improving overall device performance. Recent research has focused on developing novel synthetic methods for producing highly uniform and monodisperse 0D nanoparticles and nanodots, as well as exploring their electrochemical properties and potential applications in various electrochemical devices [37].

Extensive research has been carried out on 1D nanowires and 2D nanosheets for their applications in various electrochemical devices, such as supercapacitors and fuel cells [38]. These materials exhibit high specific capacitance and fast charge/discharge rates in supercapacitors due to their large surface area and excellent conductivity. In fuel cells, 1D nanowires and 2D nanosheets have been used as catalysts to improve reaction kinetics and reduce the cost of electrochemical reactions. The unique properties of 1D and 2D nanomaterials, such as their mechanical strength, flexibility, and transparency, make them attractive for various electrochemical device applications [39].

Three-dimensional (3D) nanometer-sized structures, also known as nanoarchitectures or nanostructures, have also been investigated for their potential use in electrochemical devices. These structures can offer a high surface area and unique porosity, which can enhance electrochemical reactions and improve the performance of electrochemical devices [40]. Examples of 3D nanostructures include nanocubes, nanorods, and mesoporous silica. Researchers have explored their use in batteries, supercapacitors, and fuel cells with promising results. However, challenges remain in synthesizing and integrating 3D nanostructures into practical electrochemical devices, and more research is needed to overcome these challenges and fully exploit the potential of these materials.

Definite analytical techniques are used for nanoparticle detection and characterization. Advanced technologies include electron microscopes for scanning or transmission (SEM, TEM) [38,39,41,42]. Different optical procedures can provide various types of physicochemical information for nanoparticles, but their usefulness depends on the technology and particular material properties, such as composition and scale [43]. Electrochemical technologies can address some problems, such as low-cost, easy-to-use portable controllers and self-powered devices [44]. As many nanoparticles can generate electrical signals, have high biocompatibility and stability, and are simple to operate, nanotechnology may be a promising solution for many electrochemical technologies [45].

Quantitative techniques are required to quantify exposure to foreign organisms to support the creation and implementation of biomonitoring programs. The electrochemical phenomenon has many technological applications, such as chemical synthesis, electroextraction, metal refining, batteries, fuel cells, sensors, and surface modification by electrostatic precipitation, separation, and corrosion. Recent studies have emphasized the importance of nanotechnology in constructing promising electrode materials for high-performance supercapacitors [43–47]. The synthesis of nitrogen-rich activated nanosized carbon with hierarchical micro/mesoporous and ultra-high specific surface area was greatly simplified and chemical waste was reduced when Zheng et al. [48] revealed a template-free and one-step carbonization–activation technology. Activated carbon with a high nitrogen content and strong electrochemical characteristics was obtained by synthesizing chitin nanoparticles in a NaOH/urea solvent using a mechanically driven sol–gel transition process.

In Table 2, we compare different types of nanomaterials used in various electrochemical devices. Metal oxides and hydroxides, such as titanium dioxide, zinc oxide, and iron oxide, offer high surface area and redox properties, making them suitable for electrochemical applications. Conducting polymers like polyaniline and polypyrrole exhibit high conductivity and tunable redox potential, providing versatility in electrochemical devices. Hybrid materials, which combine different nanomaterial classes, offer improved performance and functionality due to synergistic effects. Furthermore, nanomaterials designed for electrode materials in electrochemical devices demonstrate tailored properties for high-performance devices. By understanding the distinctive characteristics and synthesis methods of these nano-materials, researchers can develop cost-effective electrochemical devices with enhanced performance.

Table 2. Comparative Analysis of Morphological Features and Properties of Nanomaterials Synthesized via Different Methods.

Nanomaterials	Synthesis Method	Morphological Features	Properties	References
Metal oxides and hydroxides	Various	High surface area, redox properties	Suitable for electrochemical devices	[46,47,49,50]
Conducting polymers (e.g., polyaniline, polypyrrole)	Various	High conductivity, tunable redox potential	0	
Hybrid materials	erials Combination of nanomaterial classes Improved performance and Enhanced proper functionality through synergy		Enhanced properties through synergy	[52]
0D nanoparticles and nanodots	Various synthesis methods	Small size, high surface area	Promising for batteries, sensors, catalysts, supercapacitors	[53]
1D nanowires and 2D nanosheets	Various synthesis methods	Large surface area, excellent conductivity	Suitable for supercapacitors, fuel cells, and other electrochemical devices	[54]

Nanomaterials	Synthesis Method	Morphological Features	Properties	References
3D nanostructures (nanocubes, nanorods, mesoporous silica)	Various synthesis methods	High surface area, unique porosity	Potential for batteries, supercapacitors, fuel cells	[55]
Nanomaterials for electrode materials	Various synthesis methods	Tailored properties for high-performance devices	Potential for cost-effective electrochemical devices	[56]

Table 2. Cont.

4. Morphology and Properties of Nanomaterials Prepared by Different Synthesis Methods

Both top-down and bottom-up methods are employed in the production of nanomaterials. Top-down methods reduce raw materials to their nanoscale building blocks. Mechanical milling, laser ablation, etching, sputtering, and electro-explosion are all examples of top-down processes. Manufacturing phase blends and nanocomposites can benefit greatly from mechanical milling as it is a low-cost method for developing nanoscale products from bulk materials [57]. Nanocomposite materials, such as those enhanced by oxides and carbides, wear-resistant spray coatings, and nanoalloys of aluminum, nickel, magnesium, and copper, have all been made using this technique. Carbon nanoparticles that have been ball-milled are a special kind of nanomaterial that has applications in environmental remediation, energy storage, and energy conversion [2]. To produce nanostructured materials, such as nanofibers, from a variety of materials, most notably polymers, electrospinning is a commonly used top-down approach. By using two coaxial capillaries and either two viscous liquids or one viscous liquid as the shell and one non-viscous liquid as the core, core–shell nanoarchitectures may be constructed in an electric field [58]. The generation of carbon-based nanomaterials relies heavily on bottom-up approaches, such as chemical vapor deposition techniques. This technique may be used to create a wide range of carbon-based nanomaterials by chemically reacting vapor-phase precursors to generate a thin coating on the surface of the substrate.

Despite these limitations, top-down techniques have several advantages, such as the ability to produce well-defined, monodisperse nanoparticles with precise control over size and shape. These techniques are also scalable and can produce large quantities of nanoparticles. Recent research has focused on improving the efficiency of top-down techniques and reducing their environmental impact, making them more sustainable for industrial-scale production [59]. Top-down and bottom-up methods of creating graphene have both recently been developed. Bottom-up manufacturing converts carbon precursors including carbon-bearing gases, aromatic hydrocarbons, and polymers into graphene by processes like chemical vapor deposition (CVD), laser and heat pyrolysis, epitaxial growth, and direct organic synthesis. These processes are challenging because they often need sophisticated equipment and elaborate surroundings. Graphite may be converted to graphene via a variety of top-down methods, including chemical oxidation–reduction, liquid-phase exfoliation (LPE), electrochemical exfoliation, solid-phase exfoliation, and arc discharge.

It is not unexpected that the majority of commercially available graphene is made using top-down methods due to its ease of usage and reduced production costs. However, because of its high production cost, graphene is not yet widely produced across the world. Successful commercialization and industrial adoption of graphene, therefore, remain critically dependent on the development of novel methodologies and improvements in existing top-down procedures. The remarkable acceleration of graphene preparation progress over the past several years is emphasized in Figure 2. This has allowed for the commercial production of various types of graphene in metric ton quantities.

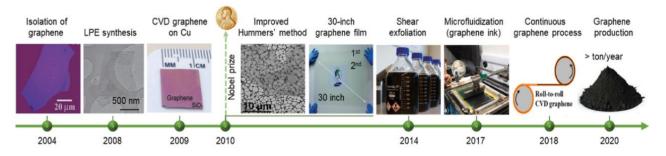


Figure 2. Timeline of main events in the research and development of graphene preparation, beginning with the isolation of graphene in 2004. Reprinted with permission from [59]. Copyright year, Elsevier, 15 April 2023. License Number 5530201217040.

Table 3 provides a comprehensive comparison of different classes of nanomaterials, highlighting their relevant properties and performance characteristics.

Table 3. Comparative Analysis of Nanomaterial Classes: Properties and Performance Characteristics.

Properties/Performance Characteristics	Carbon-Based Nanomaterials	Metal-Based Nanomaterials	Metal Oxide Nanomaterials	Semiconductor Nanomaterials	Composite Nanomaterials
High electrical conductivity	\checkmark				
Large surface area	\checkmark		\checkmark		
Excellent mechanical strength	\checkmark				
Unique catalytic properties		\checkmark			
High surface-to-volume ratios		\checkmark			
Semiconducting behavior			\checkmark	\checkmark	
Diverse functionalities			\checkmark		
Size-dependent optical and electronic properties				\checkmark	
Enhanced conductivity					\checkmark
Tunable bandgap				\checkmark	
Efficient charge separation			\checkmark		
Improved photocatalytic activity			\checkmark		
High electrocatalytic activity		\checkmark			
Fast charge transport	\checkmark				
High stability	\checkmark				
Efficient light absorption				\checkmark	
Mechanical strength					\checkmark

Note: (\checkmark): The sign refers to a specific characteristic that indicates the desired function of nanomaterial.

To create nanostructured materials, the hydrothermal process is one of the most wellknown and commonly utilized techniques. The hydrothermal method involves carrying out a heterogeneous reaction in an aqueous medium at high pressure and temperature close to the critical point in a hermetically sealed tank, resulting in nanostructured materials. The solvothermal strategy is functionally equivalent to the hydrothermal one. The only difference is that it is performed in an organic solvent rather than water [60]. Closed systems are typically utilized for hydrothermal and solvothermal processes. The sol–gel process is a common wet-chemical strategy for fabricating nanomaterials [61]. This method is employed to fabricate a wide range of superior metal-oxide-based nanomaterials. Because the liquid precursor is first transformed to a sol before the metal oxide nanoparticles are synthesized, this procedure is referred to as the sol–gel technique.

Morphology control is a promising and successful technique for changing surface atomic active sites and thereby increasing intrinsic electrocatalytic activity and selectivity. A change in morphology might result in the appearance of new physical and chemical characteristics in the same material [62]. Because of the varied atom distribution, the form of the nanomaterial might influence the electroanalytical properties. The intrinsic morphology, crystal morphology, and aspect ratio of 1D transition metal oxides strongly influence their electrochemical performance. Studies aimed at optimizing the electrochemical performance of supercapacitors by controlling the aspect ratio and morphologies of 1D nanomaterials are being conducted on transition metal oxides used in supercapacitor electrodes [63]. Mao et al. [64] manufactured core-shell CuCo₂S₄/CoMoO₄ nanorods by a hydrothermal and calcination process. Core-shell-structured CuCo₂S₄/CoMoO₄ nanorods were generated on nickel foam using a combination of the hydrothermal technique and calcination in this work. The hydrothermal method was employed to grow the $CuCo_2S_4/CoMoO_4$ nanorod heterostructure on the NF substrate (Scheme 2). In Figure 3, the morphologies of $CuCo_2S_4$, $CoMoO_4$, and $CuCo_2S_4/CoMoO_4$ are displayed. $CuCo_2S_4$ exhibits a porous cluster structure with interlaced nanowires, as depicted in Figure 3a,b. During the hydrothermal reaction, $CoMoO_4$ forms a nanosheet on the NF, as shown in Figure 3c,d. Meanwhile, Figure 3e-h display the SEM images of CuCo₂S₄/CoMoO₄, which demonstrate that it has highly uniform nanorods with a diameter of approximately 100 nm. Interestingly, this is in stark contrast to the morphologies of $CuCo_2S_4$ and $CoMoO_4$.



Scheme 2. Illustration of the synthesis of the CuCo₂S₄/CoMoO₄ nanorod array on the NF substrate. Reprinted with permission Ref. [64] with permission license 5536080833324. Copyright Elsevier. 25 April 2023.

The electrochemical performance of the $CuCo_2S_4/CoMoO_4$ heterostructure is enhanced. The $CuCo_2S_4$ nanorods provided pathways for quick electron transport and efficiently disseminated $CoMoO_4$ particles, while the outer $CoMoO_4$ shell hindered the inner $CuCo_2S_4$ nanorods' expansion during the redox reaction and boosted the composite's stability [64,65].

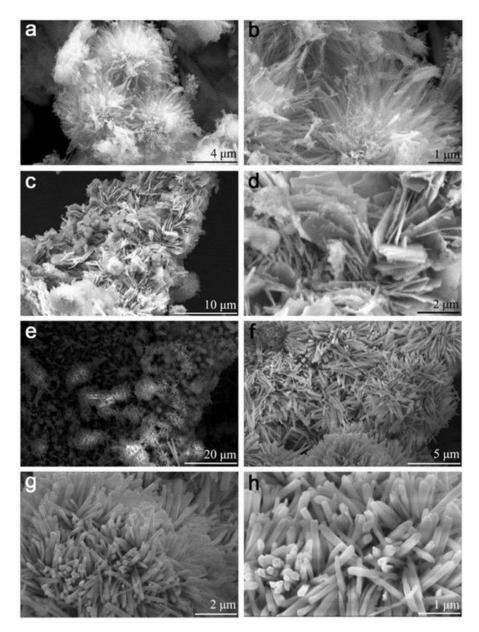


Figure 3. SEM images of $CuCo_2S_4$ (**a**,**b**); $CoMoO_4$ (**c**,**d**); and $CuCo_2S_4/CoMoO_4$ nanorod structures (**e**-**h**). Reprinted with permission from Ref. [64] with permission license 5536080833324. Copyright Elsevier. 25 April 2023.

Titania nanostructures with low dimensions (such as 0D, 1D, and 2D structures) have intriguing properties, including a high surface-to-volume ratio, clearly defined surfaces in terms of structure and geometry, small curvature radii, and enhanced electrical, thermal, and chemical stability. Electrochemical capacitors, photocatalysis, sensors, and energy storage devices are just some of the many surface-related and interfacial applications that might benefit from TiO₂ nanostructures because of their unique features [66]. Incorporating 0D and 2D materials into a composite may be performed in four different ways (Figure 4a–d): The 0D component is used as decoration for the bottom surface of the 2D material (Figure 4a). The 0D part is placed around the boundaries of the 2D substance (Figure 4b). A 2D substance encloses the nanoparticle (Figure 4c) [67]. as Additionally, the dopants can be incorporated into the 2D material (Figure 4d) if the zero-dimensional component consists of single atoms. In a composite where the particle is located on the basal plane (as shown in Figure 4a), the nanoparticle or fullerene must be securely attached to the two-dimensional material host to maximize catalytic activity, prevent nanoparticle ripening, or at the very least reduce the likelihood of the zero-dimensional component detaching from the composite under practical conditions. By understanding the interaction processes between the zero-dimensional and two-dimensional components and properly capping the nanoparticle or treating the two-dimensional material host in advance, we can better design our materials. Engineering functional groups for the zero-dimensional component and/or the two-dimensional material that take advantage of one of four types of interaction (Figure 4e–h) may improve its adherence, including van der Waals attraction (Figure 4e), hydrogen bonds, electrostatic repulsion, and dipole–dipole interactions (Figure 4f), as well as p interaction with aromatic molecules (Figure 4g) or a covalent bond (Figure 4h) [68].

In metal oxide supercapacitors, the charge storage mechanism is generally governed by faradaic processes (reversible redox reactions) at the electrode surface [69]. Non-faradaic supercapacitors, which are dominated by electrostatic charge diffusion and accumulation at the electrode-electrolyte interface via an electric double-layer capacitor formation, can be integrated into the metal oxide matrix to significantly improve hybrid supercapacitors' capacitive performance [69]. In terms of electrochemical performance, TiO_2 demonstrates electrochemical double-layer capacitance (non-faradaic) with significantly lower specific capacitance values than standard carbonaceous non-faradaic capacitors. Because titania is semiconducting, its conductivity is limited, and the interfacial states/defects between the titania and metal electrodes slow down charge transport, preventing the electrodes from achieving their full electrochemical potential [70]. The hydrothermal procedure, which is simple and inexpensive, is widely used to create various titania/titanate nanostructures [71]. In particular, the alkali-controlled hydrothermal treatment of titania is extremely effective in controlling the surface shape of titania/titanate nanostructures. However, the Schottky barrier, which is expressed by the Fermi level mismatch between the semiconducting titania and the metallic electrode, is typically characterized by the interfacial contact between the nanostructured titania and the metal electrode for electrochemical device applications.

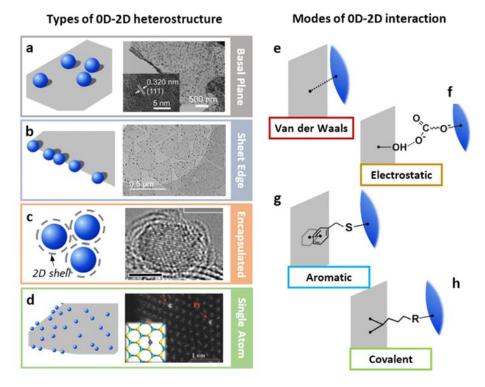


Figure 4. The different morphologies and potential interaction mechanisms for 0D-2D hybrids. (a) Nanoparticles are arranged on the basal plane. A TEM image of CoS_2 nanoparticles decorating a reduced graphene oxide (r-GO) nanosheet [72]. (b) Nanoparticles are preferentially situated along the nanosheet edge. A TEM image of triangular MoS₂ with Au nanoparticles along their edges [73].

(c) Nanoparticles encapsulated in 2D sheets. A TEM image of a Pt nanoparticle encapsulated in several graphene layers. Scale bar 2 nm [74]. (d) Single-atom sites within, on top of, or along the edge of the nanosheet. High-angle annular dark-field (HAADF) scanning-mode transmission electron microscopy (STEM) image of Pt substitutions in MoS₂ [75]. (e) A van der Waals interaction, such as between graphene and a ligand-free nanoparticle. (f) Electrostatic dipole–dipole interaction, such as between a hydroxyl functional on GO and a citrate ligand on a nanoparticle. (g) An aromatic/p-p interaction, such as between graphene and a phenylethanethiol-capped nanoparticle. (h) A covalent bond is typically formed between a functional group on GO and a ligand. Permission from Elsevier, License Number 5527170984812. 13 April 2023.

The self-sourced Ti surface is directly modified into titania nanostructures in situ and then used as-is as an electrochemical microelectrode, which is novel [71]. As a result, the transition between metallic Ti and semiconducting TiO₂ is greatly enhanced. The neardefect-free interfacial feature of the Ti-TiO₂ microelectrode has demonstrated outstanding super-capacitive performance for enhanced charge storage devices, demonstrating the importance of interfacial states and defects in controlling the charge transport characteristics of electronic and electrochemical devices.

To create hollow carbon nanocages, scientists use graphene microcrystals as building blocks to enclose curved carbon nanosheets with sub-micrometer gap space. A large number of sub-nanochannels (1 nm) or purpose-built nanopores (1-10 nm) connect the inside and outside of carbon nanocages [76]. This unique carbon nanocage material has a number of advantages over previously developed nanocarbon materials, including a readily accessible inner cavity, the coexistence of micropores, mesopores, and macropores, a high specific surface area, and the ability to be easily doped and modulated. Also, the arch arrangement of carbon layers in the cage-like carbon nanomaterials' curved nanosheet structure may significantly reduce anisotropy by obstructing interlayer sliding and guaranteeing structural stability [76]. Designing interfacial composites based on hollow carbon nanocages may be conducted in two ways: (a) by constructing metal or metal compounds enclosed by hollow carbon nanocages and (b) by generating hollow carbon nanocages coated with metal or metal compounds. Furthermore, the electrochemical energy storage capabilities of hollow interfacial materials may be improved by the compact interfacial effect of carbon nanocages' metal compounds. Molybdenum phosphide nanoparticles and nitrogen-doped carbon (MoP-NC) complex hollow nanocages were successfully synthesized by Liu et al. [77] by combining non-template and annealing techniques with both organic and inorganic composite precursors. The findings suggest that there is a complementary relationship between nitrogen-doped carbon and hydrogen production in alkaline electrocatalysis. The use of hollow nanocages and molybdenum phosphide nanoparticles (metal-carbon) can further enhance this relationship, resulting in faster hydrogen production. Therefore, these materials can improve the performance of electrocatalysis for hydrogen production by optimizing the interaction between nitrogen-doped carbon and the hydrogen production process [77].

5. Nanotechnology-Based Electrochemical Sensors

The synthesis and characterization of nanomaterials have opened up new opportunities for the development of cost-effective electrochemical biosensors. These biosensors have the potential to revolutionize various fields, including healthcare, environmental monitoring, and food safety. Biosensors that detect analytes with high sensitivity and specificity have been made possible by the use of nanomaterials due to their unique characteristics and high surface-to-volume ratio.

The use of nanomaterials in biosensors has led to the development of various novel sensing mechanisms, such as surface-enhanced Raman scattering (SERS), localized surface plasmon resonance (LSPR), and electrochemical impedance spectroscopy (EIS). These mechanisms offer several advantages, including improved detection limits, enhanced selectivity, and reduced analysis time. Moreover, the use of nanomaterials in biosensors has

enabled the development of portable, low-cost, and disposable devices, which are highly desirable in resource-limited settings.

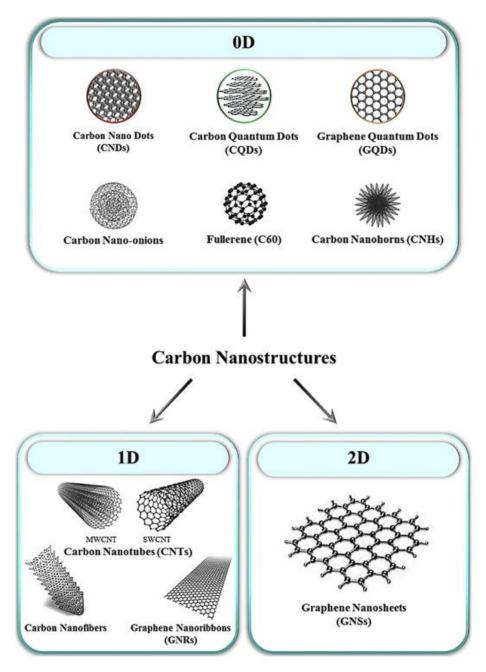
Electrochemical sensors are based on enzymes, such as oxidation and reduction enzymes, that stimulate chemical reactions that produce or consume electrons. The sensor consists of three electrodes: the reference electrode or signal, the precipitation, and the reserve electrode [78]. The target analyte participates in the reaction on the active electrode's surface, and the ions produced generate an electrical potential from that electrode to trigger a signal. The electron flow rate can be measured in proportion to the analyte concentration when the voltage is constant, or it can be measured at the zero point (this provides the logarithmic response). It should be noted that a sensitive charge capacitance is used in the working voltage or active electrode. Direct electrophoresis of small peptides and proteins may be made possible by their intrinsic charges used for bio-functionalized ions in the influencing field of the transistor. The potentiometer is a type of position sensor that works as an adjustable voltage divider. The sensors are print screens, cabling embedded polymers, and open circuit sensors that rely on conjugated polymers for immunoassays [79]. These appliances have only hypersensitive and long-lasting electrodes. The alleles can only be detected by HPLC, LC/MS, and without preparing a simple model at existing levels. The signal is generated by electrochemical energy and physical alterations to the conductive polymer layer due to variations on the sensor surface [80]. These changes are attributed to ionic force, water pH, and redox reactions as the latter bends the enzyme around the base. An existing measurement detector estimates the current when a solvent is actively in contact with a working electrode, which is fixed to a constant tension for a reference electrode [81].

An electrode solution is used to determine the voltmeters that produce a characteristic electrode signal, called waveforms, which are then expressed. Electrode current data are quantified over time using voltmeter and ammeter detectors. Reversible pulse potentials between high and low values are useful for voltmeter measurements because they provide the distinctive current response for inverse interactions. In order to measure the voltage flowing through the electrode during its lifetime, the signal must be pulsed with extra voltage at short intervals between high and low values. An oxidized or reduced product can be created on the electrode surface, resulting in a positive or negative current by applying pulses of potential voltage between high and low voltage levels, respectively.

Recent technological developments have made it easier to design electrodes that are sensitive to certain analytes while still being cost-effective, dependable, and selective. Electrodes with screen printing, integrated nanomaterials, and EC immunoassays are three examples of how these advancements might be used together to create electrical sensor (EC) technology. These new technologies have led to the creation of heterogeneous systems that combine their best features [81]. Chemical exposure analysis has several potential uses in modern environmental and biological monitoring thanks to the development of low-cost and selective EC sensors. Chemicals, metabolites, proteins, metals, inorganic ions, organic compounds, and more may all be identified using these methods in a wide range of biological matrices [82].

Nanomaterials provide several advanced analysis technologies, including more sensitive, selective, and biomonitoring electrochemical sensors.

Carbon nanomaterials are favored in enhancing the performance of electrochemical sensing [83]. Carbon nanomaterials can increase electrode surface area, accelerate electron transfer, and act as catalysts, enhancing electrochemical processes such as detection, adsorption, and removal. Several carbon nanomaterials, including graphene, mesoporous carbon, carbon nanotubes, and carbon dots, have demonstrated strong adsorption and detection capacities (Figure 5). Electrochemical methods using anodic stripping voltammetry with modified carbon electrodes are commonly used for detecting heavy metals due to the excellent electrocatalytic properties of carbon and its derivatives. Surface modification of the working electrode (GCE) increases the sensitivity of metal electrode detection. Electro-



chemical sensors can be made much more effective by first coating the working electrode with a layer of active electrocatalytic species.

Figure 5. Different types of carbon nanostructures in zero, one, and two dimensions. Reprinted with permission from Ref. [84] with permission license 5537630623042. Copyright Elsevier. 29 April 2023.

Electrochemical platforms with enhanced electrical conductivity and sensitivity can be designed and fabricated using CNPs due to their excellent characteristics. In this context, establishing a stable modifier coating on the electrode's surface is crucial. Electrode surface modification using CNPs has been accomplished in different ways, as illustrated schematically in Figure 6. Drop-casting modification is a quick and simple approach to creating customized electrodes. When a CNP suspension is dropped over the surface of bare electrodes, stable films are generated due to the CNPs' strong dispersibility in water. This method is practical for carbonaceous electrodes like glassy carbon electrodes (GCE) due to the strong contacts between the electrode surface and CNPs or CNP composites with other carbon nanomaterials (such CNTs and graphene). However, the modifier film's stability might be a problem for non-carbonaceous electrodes. Carbon paste electrodes (CPEs) are fabricated using a similar process, except that a combination of carbon (microparticles) powder and an appropriate binder (such as Nujol or ionic liquids (ILs)) is used to create an electrode. Carbon nanoparticle (CNP) composites can be used as modified carbon paste electrodes for electrochemical platforms. CNPs have excellent characteristics for surface functionalization, and their composites with hydroquinone, sodium dodecyl sulfate (SDS), and halloysite nanoclay (HNC) have been proposed for modified electrodes due to their low background current, large potential window, low cost, ease of preparation, electrode surface renewal, and potential for miniaturization. The layer-by-layer (LBL) assembly approach has been used to manufacture multilayer uniform thin films with regulated architecture and composition, which can address stability difficulties related to the drop coating method. The LBL method involves alternating adsorption of polyelectrolytes and/or nanomaterials onto a charged substrate with an opposing charge.

The electrode surface modification with the LBL technique has been used to assemble negatively charged phenyl sulfonated carbon nanoparticles onto positively charged substrates, such as chitosan, poly (diallyldimethylammonium chloride) (PDDAC), imidazolium salt ionic liquid (IL) precursor, and sol–gel-processed functionalized silicate.

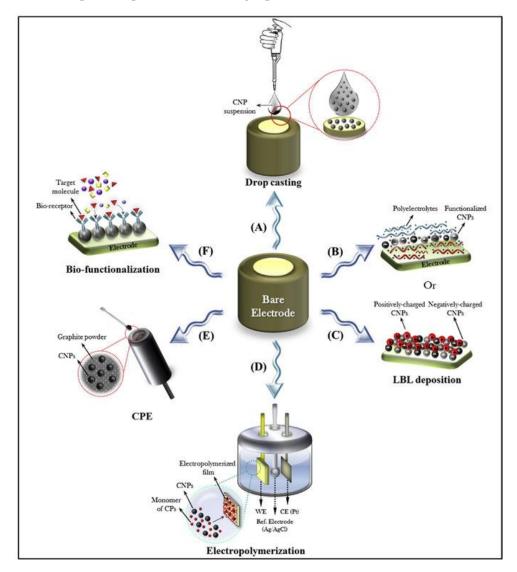


Figure 6. Schematic representation of various methods for electrode surface modification using CNPs. Reprinted with permission from Ref. [84] with permission license 5537630623042. Copyright Elsevier. 28 April 2023.

The modification of carbon nanoparticle (CNP) electrodes with organic compounds significantly improves their electrochemical sensitivity to both organic and inorganic analytes. Electrochemical polymerization of conducting polymers (CPs) is an effective method for creating modified electrodes with excellent electrical conductivity. One such method involves the electro-polymerization of CNPs/pyrrole aqueous solution on the surface of a glassy carbon electrode (GCE) by cycling the voltage between 0 and 0.8 V. This process creates a sensing platform where the synergistic impact of polypyrrole (PPy) and CNPs enhances electrochemical performance beyond the sum of their separate surface area effects.

CNPs are responsible for high electrochemical activity because they can act as liquid– liquid interface stabilizers and interfacial conduits for electrons. Another method for the formation of CNPs on the surface of electrodes is through the cyclic voltammetry (CV) technique, which was used to achieve surface nanocrystallization of carbonaceous surfaces like glassy carbon electrodes in anhydrous N, N-dimethylformamide (DMF) containing tetra-n-butylammonium bromide (TBAB) [85]. As shown in Figure 7, in situ synthesis of CNPs on the surface of GCE occurred due to redox reactions and the insertion of Bu_4N^+ ions and electrolytes into the surface layer of the GC electrode during continuous cathodic and anodic scanning.

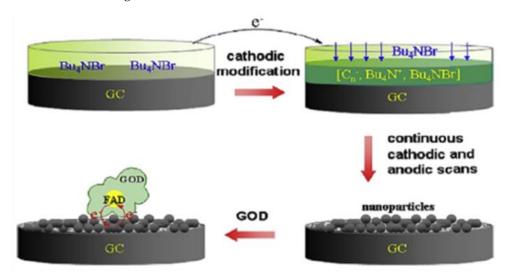


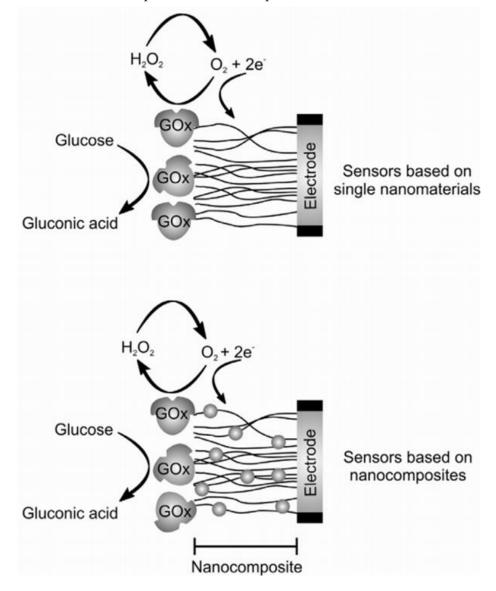
Figure 7. Mechanism of in situ formation of CNPs on the surface of GC electrode by surface nanocrystallization process used for investigation of the direct electrochemistry of glucose oxidase enzyme (GOD), Reprinted with permission from Ref. [85]. With permission license 5537631494458. Copyright Elsevier. 28 April 2023.

Biosensors are analytical devices that combine a biological component, such as an enzyme or antibody, with a physicochemical transducer to convert a biological response into a measurable signal. Electrochemical biosensors, in particular, have emerged as a promising tool for healthcare applications, as they can detect biomarkers in body fluids such as saliva, interstitial fluid, and sweat. By functionalizing carbon nanomaterials with chemical and biological components, biosensors can be made to work at the same scale as natural biological processes, providing high sensitivity and specificity for the detection of target analytes.

Electrochemical sensors are used in the medical sector; for instance, a glucometer (glucose meter) is used to monitor blood levels of glucose in diabetic patients [86]. Modern electrochemical biosensors are promising tools for the healthcare sector, because they can detect biomarkers in body fluids such as saliva, interstitial fluid, and sweat. Blood is the standard biofluid for glucose monitoring. For example, the blood glucose level is well known to be used for warning patients with diabetes. Blood glucose levels are about 4.9–6.9 mM, while patients can have a high (even 40 mM) or a low glucose level (2 mM) [87].

Two types of electrochemical glucose sensors are available, enzyme and non-enzyme. The electron redox mediators are organic and organometallic compounds like ferrocene derivatives, ferrocyanide, organic salt conducting agents, and quinones. In contrast, the electrochemical properties of glucose sensors still depend on GOx properties, and it has been more complicated to manufacture glucose sensors [87].

The schematic and operational mechanisms of glucose detection utilizing single nanomaterial and nanocomposite sensors are depicted in Scheme 3 [88].



Scheme 3. Mechanics of sensing glucose using single nanomaterial and nanocomposite glucose biosensors. Reprinted with permission from Ref. [88], with permission license 5537761217387. Copyright Elsevier 28 April 2023.

The third generation of glucose sensors has lower detection features and a smaller linear range compared to the first- and second-generation sensors. Non-enzymatic glucose sensors are based on direct glucose oxidation on the electrode surface and can use methods such as potentiometry and amperometry to measure the electrode's potential. Certain metals such as Pt, Au, Cu, Ni, and Co, metal oxides like CuO and CO_3O_4 , and carbon materials are promising candidates for non-enzymatic glucose sensors due to their electrocatalytic activity. An ideal glucose sensor should be selective for glucose and have a sensitivity of more than 1 μ A/mMcm². Additionally, the sensor should remain stable for at least 6 months during application.

Bare carbon electrodes have inadequate sensitivity and an excessive level of electrochemical insulin detection. To improve electrode sensitivity, various types of nanoparticles such as SiO₂NPs, NiONPs, SiCNPs, CNTs, RuOCNTs, Ni (OH)₂, and GO have been used for electrode surface modification. The electrocatalytic activity towards insulin has been found to increase when nanoparticles are modified with metal and graphene oxide. Researchers have used a chemical method and polyaniline conductance polymer as a support holder to develop a flexible glucose sensor that can be synthesized and deposited into the cotton material substrate using nickel phosphide (NiP) and copper oxide (CuO) nanoparticles [89].

Scientific studies can use additional electronic measurements for glucose detection besides resistance- and conductance-based methods. In the existence of glucose, the ConA dextran displacement, immobilized with gold-coated nanostructures on an electrode, changes capacity throughout the electrode. Potentiometrically, ion-selective electrodes can measure free silver ions (Ag⁺), which in GOx-generated hydrogen peroxide are released from silver nanoparticles. After glucose oxidation, it can be detected in nanotubes that function on palladium, although it is several orders of magnitude below physiological blood glucose with a linear range of detection [90].

To improve the sensitivity of electrochemical flavonoid sensors, modified materials such as carbon nanomaterials, metal nana-particles, and the liquid combination of these materials were employed [91]. The sensing elements could deliver more active sites, increase the active electrical surface area, and improve mass transport rates and the speed of electron transfer rates. A new electrochemical platform based on ionic nanocomposite nanoparticles CoFe₂O for the analysis showed an excellent result because the ionic liquids have high conductivity and quick mobilization The electrochemical flavonoid biosensors have attracted attention because of their outstanding electric conductance, biocompatibility, and significant stability. Electrochemical sensor construction with carbon-based nanomaterials has substantial properties, such as high chemical stability, large thermal and electrical conductivity, and a high surface-to-volume ratio [92]. The carbon set as C6-C3-C6 is derived from 2-phenyl chromone as the core of the flavonoid compounds.

The natural flavonoid compound mother's core has hydroxyl, methoxy, oxygen hydrocarbon, isoprene oxygen, and other substitutes. Because of the location and difference between substituents, flavonoids are divided into six subunits of isoflavone, flavonols, and anthocyanin. The flavonoid structure has a powerful linkage to its redox characteristics [93].

Nanomaterials are significant to the sensor for the functionality of biological systems, subject to fluctuating conditions. Nanomaterials can be developed to adjust the desired electrocatalytic activity and improve the sensor's stability. Non-enzymatic glucose sensors have reported materials based on copper oxide, tin oxide, or titanium dioxide [94].

Table 4 shows some sensors based on nanomaterials, such as CNTs, and their methods of manufacturing are chemical vapor deposition, arc discharge, laser ablation, and gasphase catalytic growth [95]. It also shows polymer nanowires with their methods [96], graphene nanostructures [97], metallic nanowires [98], and silicon (Si) nanowires [99].

Avoiding drift and fouling, developing reproducible calibration methods, applying preconcentration and separation methods to achieve a proper analyte concentration that avoids saturation, and integrating the nanosensor with other elements of a sensor package in a reliable manufacturable manner are all challenges for nanosensors [100]. Because nanosensors are a new technology, there are numerous unsolved problems with nanotoxicology, which restricts their usage in biological systems for the time being [100].

Table 5 provides a summary of the most common types of nanomaterials used in electrochemical biosensors. Carbon nanotubes are highlighted for their high surface area, good electrical conductivity, and excellent mechanical properties. These properties make them a popular choice for detecting biomolecules such as glucose, cholesterol, and DNA. Graphene is also used in biosensors to detect biomolecules such as glucose, dopamine, and DNA. Metal nanoparticles are then described for their high surface area and excellent catalytic activity. This property makes them useful in the detection of biomolecules such as glucose, cholesterol, and DNA. Quantum dots are highlighted for their unique optical and

electronic properties. They are used in biosensors to detect biomolecules such as proteins and DNA. By functionalizing these nanomaterials with chemical and biological components, biosensors can be made to work at the same scale as natural biological processes, providing high sensitivity and specificity for the detection of target analytes.

Nanostructure	Method of Manufacture	References	
Carbon nanotubes	Chemical vapor deposition Arc discharge Laser ablation Gas-phase catalytic growth	[95]	
Polymer nanowires	Electrochemical deposition Template filling Reactive ion etching	[96]	
Graphene nanostructures	Exfoliation Chemical vapor deposition Epitaxial growth	[97]	
Metallic nanowires	Template-assisted electrodeposition Electrochemical deposition Electroless deposition 4- Template filling	[98]	
Si nanowires	Reactive ion etching Photolithography	[99]	

Table 4. Nanosensors and methods of manufacture with one-dimensional materials.

 Table 5. A summary of the nanomaterials commonly used in electrochemical biosensors.

Nanomaterials	Properties	Application	References
Carbon nanotubes (CNTs)	High surface area, good electrical conductivity, and excellent mechanical properties The development of (bio)sensors capable of tackling future biosensing challenges in clinical diagnostics, environmental monitoring, and security control represents a very good alternative when the special properties of CNTs are combined with the potent biomolecule recognition properties and the known benefits of the electrochemical techniques.	Detection of glucose, cholesterol, and DNA	[101]
Graphene (GR)	With high sensitivities, broad linear detection ranges, low detection limits, and long-term stabilities, GR-based biosensors displayed exceptional performance.	Detection of glucose, dopamine, and DNA	[102]
Metal nanoparticles	High surface area and excellent catalytic activity	Detection of glucose, cholesterol, and DNA	[103]
Quantum dots	Unique optical and electronic properties	Detection of biomolecules such as proteins and DNA	[104]
Metal oxide nanoparticles	High surface area and excellent catalytic activity	Detection of glucose, cholesterol, and DNA	[105]

6. Nanostructured Materials for Enhanced Electrochemical Performance in Energy Storage Devices

Providing robust electrochemical energy conversion and storage systems is one of our society's most difficult issues [103,104,106,107]. Because of their nanoscale size impact, nanomaterials have numerous attractive features for electrochemical energy storage devices that are considerably different from bulk or micron-sized material [108]. Constrained

dimensions, in particular, play crucial roles in regulating the attributes of nanomaterials, including ion transport kinetics, strain/stress magnitude, and active material consumption. Because they can sustain electron transport along the longitudinal plane and have a confinement effect throughout the diameter, nanowires, as one of the characteristic onedimensional nanomaterials, offer significant potential for realizing a range of applications in the disciplines of energy storage. Using renewable energy and avoiding conventional fossil-fuel-related environmental issues has become critical for achieving a globally sustainable energy future. Electrochemical conversion and storage methods for "clean energy" (e.g., fuel cells, electrolyzers, photo-electrolyzers, metal-air batteries, metal-ion batteries, and supercapacitors) have played critical roles in this regard [109]. To rival existing fossilfuel-based energy supply systems, the performance of renewable energy devices must be greatly increased. Electrochemical energy conversion and storage typically involve many intricate chemical reactions and physical interactions at the surface and inside of electrodes/electrolytes, and the kinetics and transport behaviors of different carriers (e.g., electrons, holes, ions, molecules) are closely associated with the electrode materials and electrode structures [110].

To qualify nanostructures as excellent electrodes for energy conversion and storage that meet industry standards for device applications, it is essential to achieve well-defined nanostructures with accurately controllable geometries of four key structural parameters: size, structure, hetero-architectures through combining different nano-units, and spatial arrangement [111].

Non-rechargeable primary batteries are essential in various applications, including wristwatches, remote controls, and electronic keys, as they store energy for a long time, up to ten years, and self-discharge less than rechargeable batteries [112].

Several types of batteries have been marketed for practical electrochemical applications [113]. The first portable, non-spill battery was the zinc dioxide and manganese dry cell, which made light bulbs and other devices more convenient. However, mercury batteries of zinc and mercury oxide, which had a higher energy level, were phased out due to potential contamination and widespread mercury from discarded batteries. Lead-acid batteries were the first practical rechargeable batteries that could be recharged from an external source, and their reversible electrochemical reaction allows for the exchange of electrical and chemical energy. They contain sulfuric acid and water, as well as lead plates, with the most commonly used blending being 30% acid. However, if the acid remains unchanged, it can crystallize inside the battery's lead plates and render it useless. The average lifespan of these batteries is around three years [114]. They are widely used in cars, but their electrolytes of water limit the maximum voltage for each cell, and freezing water can limit their performance in low temperatures [115]. In comparison, lithium batteries do not use water in their elements. Rechargeable lithium-ion batteries are an essential factor in many mobile devices. Experimental flow batteries provide a wider option to regenerate reactants from external tanks [116]. Fuel cells have much greater efficiency than any combustion process and can convert the chemical energy of hydrocarbon gases into electric power. They are used to power spacecraft and for public energy grid storage [117].

A flow battery system generates energy using an electrochemical cell and electrically active solutions, typically mineral salts [118]. This non-metallic material with electric activity provides large-scale energy storage, which is crucial for utilizing renewable and intermittent energy sources like solar and wind energy, which can account for over 20% of the total energy capacity when paired with storage systems for electrical power. These applications are used in electrical grids and remote generating stations that require inexpensive and flexible storage systems [119]. There are options for using water pumping from a reservoir to high elevations as a power source, but these can be expensive and limited to specific geographic locations, such as conventional batteries and superconducting electromagnetic storage [120]. Flow batteries, on the other hand, require two active and dissolved components—compounds that can participate in the electrochemical reaction that occurs at the electrode [115]. In the electrochemical cell, chemical energy is transformed

into electrical energy and vice versa via an ionic conducting membrane. Electrically active substances are pumped through the cell into a fluid and kept in separate tanks outside the cell, as opposed to conventional electroactive materials (Figure 8). This design allows for optimum separation of the energy stored, depending on the size of the electrochemical cell or the cell group [121].

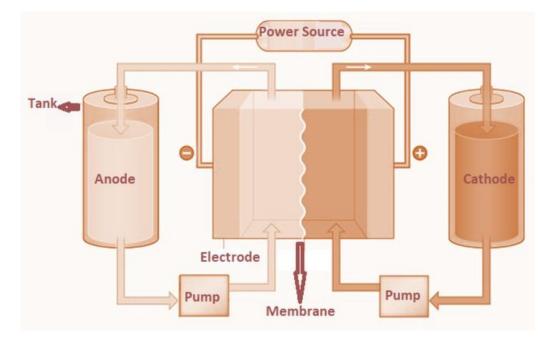


Figure 8. Illustration of Flow Battery. Adapted from [122]. with permission license 5583830752137, Copyright Elsevier 7 July 2023.

Nanostructured materials are of interest for electrochemical energy conversion and storage devices due to their large surface area, new size effects, and increased kinetics and other properties. Two types of nanoscale size effects have been discovered in these materials: "trivial size effects," which rely solely on the increased surface-to-volume ratio, and "true size effects," which also require changes in local material characteristics [123]. The field of "nanoionics" has established a significant place in this area, comparable to nanoelectronics in semiconductor physics, and its advancement may lead to future generations of clean energy devices.

To significantly improve the electrochemical efficiency of electrode materials, their dimensions can be decreased to a nanoscale level to benefit from the nanoparticle effect, which results in a higher surface area and shorter ion spreading time [124]. Various nanoparticle materials with improved electrochemical performance have been fabricated, although controlling the size, especially when it is less than 10 nm, can be a difficult problem [125].

Some studies have developed the nano-molecule flow battery, which can store electric and hydrogen gas for supplying hybrid energy storage systems. This battery adopts a nano-battery design, "hybrid hydrogen energy flow," to store energy and release on-demand electricity or hydrogen gas for use as fuel [126]. When a condensed liquid containing nanoparticles is used, the volume of stored energy is increased by around ten times. This system allows for flexible operation of the equipment, requiring either fuel or electricity [125].

Rechargeable batteries must meet several main parameters to be useful in hybrid electric vehicles and electric vehicles, including cost, higher energy capacity, longer cycle life, safety, and environmental viability [125].

Nanometer-sized electrode materials may improve electroactivity toward Li insertion and high rate capability, enabling high power performance [127]. The high rate capacity results directly from the transport benefits of tiny particle sizes, such as shorter transport lengths for both e- and Li⁺ transport and a greater electrode/electrolyte contact area due to the higher surface area [128]. The former allows for complete Li diffusion in a short storage period, i.e., at high charge/discharge rates, while the latter considerably lowers the specific current density of the active material [128]. In addition to the "conversion" mechanism, an interfacial Li storage mechanism has recently been proposed to explain the additional Li⁺ storage capacity in nanometer-sized transition metal oxides at low potential. Li surface storage may play a key role in total capacity in nanostructured devices. This technique may be more energy-efficient for nanometer-sized particles than for bulk insertion.

In recent years, metal oxide nanostructures have been investigated for various applications such as sensors, energy-efficient coatings, and semiconductor devices [129]. These nanowires are covered with a gold-palladium alloy that functions as an existing conductor. Manganese oxide is deposited on the alloy surface in the electrochemically active form [130]. The result is a hundred-fold increase in the surface area of the brush-like layer compared to the copper cable because the broad surface is an essential factor for storing energy. The nanowire acts as the sheath that covers the copper wire and is the supercapacitor's first electrode. Some researchers have investigated environmentally friendly batteries that can provide cars and mobile phones with the necessary energy, using nanotechnology and genetically modified viruses. These viruses infect bacteria, do not harm humans and have been used to build the two charged terminals (poles) of lithium-ion batteries, which have the same power, capacity, and performance as the latest rechargeable models [131]. As a result, they operate hybrid energy cars and personal electronic devices. When tested in laboratories, the new cathode material could be charged and discharged more than a hundred times without losing any part of its capacity or electrical capacity. This technology enables lithium-ion batteries to charge in seconds, not hours. This achievement will lead to smaller, faster-charging batteries for mobile phones and other devices.

The incorporation of nanoscale silicon in lithium-ion batteries can increase their energy density by up to 10 times due to the high surface area of the silicon nanoparticles, which can store more lithium ions. Similarly, the use of nanostructured sulfur as the cathode material in lithium–sulfur batteries has been shown to improve their energy density and cycle life.

Lithium-sulfur batteries offer three times higher energy density than standard lithiumion batteries, thanks to the low atomic weight of lithium and moderate weight of sulfur [132]. During discharge, lithium dissolves from the cathode layer and reverses this process on the anode surface during charging. Lithium-sulfur batteries can be refilled at a temperature of 60 °C and exhibit good cold–hot discharge characteristics. However, they face challenges such as limited cycle life (40–50 charge/discharge cycles) and poor stability at high temperatures [133–135]. Despite these challenges, lithium–sulfur technology shows promise for use in battery experiments with nanowires. Researchers have developed a new high-performance lithium battery anode-electrode structure using carbon and silicone nanomaterials. This material contains potent and durable silicon circuits with irregular channels that increase molecular mass–lithium battery receptivity [131,132]. The research has shown stable performance and capacity with graphite cathodes five times larger than regular lithium batteries. Manufacturers tout it as a simple, cost-effective, safe, and widespread technology [136]. However, structural problems limit the cycle life and extraction of lithium at higher volumes. When compared to the more traditional method of oxidizing and reducing organic molecules in organic synthesis, organic electrophoresis is a more eco-friendly and reliable option. Electro-oxidation processes have various advantages over conventional reagent-based reactions [137], including functional tolerance, moderate conditions, scalability, and sustainability. Careful applications of electrochemical oxidation to electrophoresis have been made [138]. Sustainable methods and tactics including oxidation mediators for direct re-operation of anabolic and inactive C-H in massive industrial contexts have also been created by researchers [139]. Some researchers have conducted electrochemical anodic oxidation using the galvanizing method without catalysts and oxidation. Recent studies have also focused on anode/cathode electrodes modified with

electrolytic oxidation/reduction nanomaterials [140]. Using electrodes can expand the electrode synthesis range and improve electrode synthesis efficiency. Therefore, developing modified electrodes with special electrical synthesis functions is essential [141].

Nanotechnology provides new opportunities for designing, synthesizing, and modifying cathode materials to overcome capacity limitations and significantly improve battery performance. The nano-template approach allows the creation of nanostructure materials with complicated morphology in a single step while maintaining high control over size and form, which would otherwise be challenging to achieve [142]. Solvothermal/hydrothermal and coprecipitation synthesis techniques are widely used, easy, and low-cost approaches for producing cathode nanostructures with high yield, but they lack control over the final nanostructure size and form [143]. The nanostructural fabrication of cathode materials has resulted in improved battery performance due to their enhanced structure and shorter Li⁺ diffusion route lengths, allowing for faster Li⁺ insertion/extraction processes [144]. Advances in nanosynthesis processes have led to precise control over size, form, and repeatability at low to medium processing costs, and the limitations are quickly closing.

Supercapacitors, on the other hand, rely on the fast charge–discharge of ions at the electrode-electrolyte interface, making them ideal candidates for nanomaterials. Nanomaterials such as graphene, carbon nanotubes, and metal oxides have been used as electrode materials in supercapacitors to enhance their energy and power densities, as well as their cycle life [145]. The use of these nanomaterials can increase the specific capacitance of the electrodes by several orders of magnitude, due to their large surface area and high conductivity. Supercapacitors can store a lot of energy and charge quickly; their working voltage is between 1 and 3 V, which is suitable for both organic and aqueous electrolytes. Batteries have a high energy density so our gadgets may be used all day, but they might take a long time to charge. Electrochemical capacitors known as supercapacitors are utilized for quick power supply and recharging (i.e., high power density). Nanomaterials are particularly well suited for use in supercapacitors because of their high surface area-to-volume ratio, which provides a large interface area for ion adsorption and desorption. The fast charge-discharge rate of supercapacitors is dependent on the rapid transfer of ions at the electrode-electrolyte interface, and nanomaterials with their large surface area and short diffusion distance are ideal for facilitating this process. Unless basic performance parameters are carefully considered, the fuzziness between these two electrochemical techniques might lead to misunderstanding and even inappropriate claims (Figure 9).

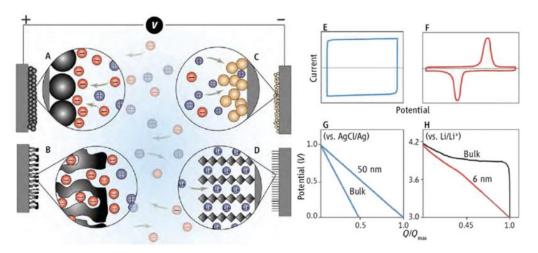


Figure 9. Comparing batteries and supercapacitors. (**A**,**D**) The different mechanisms of capacitive energy storage are illustrated. Double-layer capacitance develops at electrodes comprising (**A**) carbon particles or (**B**) porous carbon. The double layer shown here arises from the adsorption of negative ions from the electrolyte on the positively charged electrode. Pseudocapacitive mechanisms include

which exhibits faradaic redox peaks (F). Galvanostatic discharge behavior (where Q is a charge) for a MnO_2 pseudocapacitor is linear for both bulk and nanoscale material (G) [146,147], a LiCoO₂ nanoscale material exhibits a linear response while the bulk material shows a voltage plateau (H). Permission from Elsevier, License Number 5526570094637. 12 April 2023.

Table 6 provides an overview of the best-performing materials in various electrochemical devices, highlighting their material class, synthesis method, performance parameters, and applications. These materials play a crucial role in advancing the field of electrochemistry by offering enhanced performance and enabling the development of high-performance devices.

Table 6. Best-Performing Materials in Electrochemical Devices.

Material Name	Material Class	Synthesis Method	Performance Parameters	Application	References
Lithium Cobalt Oxide (LiCoO ₂)	Metal Oxide	Solid-state reaction	High specific capacity, good cycling stability	Lithium-ion batteries	[148,149]
Lithium Iron Phosphate (LiFePO ₄)	Metal Phosphate	Sol-gel method	High energy density, long cycle life	Lithium-ion batteries	[150]
Silicon/Graphene Composites	Composite	Chemical vapor deposition (CVD)	High specific capacity, enhanced stability	Lithium-ion batteries	[151]
Sodium-ion Intercalation Materials	Metal Oxide	Hydrothermal synthesis	Good rate capability, low cost	Sodium-ion batteries	[152]
Graphene	Carbon-based Material	Mechanical exfoliation	High specific capacitance, fast charge-discharge rate	Supercapacitors	[153]
Activated Carbon	Carbon-based Material	Chemical activation	High energy density, long cycle life	Supercapacitors	[154]
Polyaniline	Conductive Polymer	Chemical oxidation	High capacitance, good stability	Supercapacitors	[155]
Carbon Nanotubes	Carbon-based Material	Chemical vapor deposition (CVD)	High power density, excellent cycling stability	Supercapacitors	[156]
Proton Exchange Membrane (PEM)	Polymer Electrolyte	Solution casting	High proton conductivity, Low permeability	Polymer electrolyte fuel cells	[157]
Platinum-based Catalysts	Noble Metal	Wet chemical synthesis	High catalytic activity, good durability	Polymer electrolyte fuel cells	[158]
Solid Oxide Electrolyte	Ceramic	Solid-state sintering	High ionic conductivity, stable at high temperatures	Solid oxide fuel cells	[159]
Perovskite Oxides	Metal Oxide	Sol-gel method	Good oxygen reduction reaction, thermal stability	Solid oxide fuel cells	[160]
Metal Oxide Semiconductors	Metal Oxide	Chemical vapor deposition (CVD)	High sensitivity, selective detection	Gas sensors	[161]
Enzymes	Biocatalyst	Enzyme immobilization	High specificity, rapid response	Biosensors	[162]
Nanomaterials (e.g., Carbon nanotubes, Graphene)	Various	Various synthesis methods	High sensitivity, versatile applications	Biosensors	[163]

In the realm of lithium-ion batteries, Lithium Cobalt Oxide (LiCoO₂) demonstrates high specific capacity and good cycling stability, while Lithium Iron Phosphate (LiFePO₄) excels in energy density and cycle life. Silicon/graphene composites exhibit a high specific capacity and enhanced stability. These materials have found significant applications in lithium-ion batteries due to their favorable electrochemical properties.

For alternative energy storage systems, sodium-ion intercalation materials have gained attention for their good rate capability and low cost, making them promising candidates for sodium-ion batteries. Additionally, supercapacitors, which offer rapid charge–discharge capabilities, find valuable materials in graphene, activated carbon, polyaniline, and carbon nanotubes.

Polymer electrolyte fuel cells rely on Proton Exchange Membranes (PEMs) with high proton conductivity and low permeability, while platinum-based Catalysts provide excellent catalytic activity and durability. In the realm of solid oxide fuel cells, solid oxide electrolytes with high ionic conductivity and stability at high temperatures, along with perovskite oxides with good oxygen reduction reaction and thermal stability, contribute to their efficient operation.

Metal oxide semiconductors find applications in gas sensors due to their high sensitivity and selective detection capabilities. Enzymes, immobilized for enhanced stability, offer high specificity and rapid response in biosensor applications. Nanomaterials like carbon nanotubes and graphene exhibit versatile applications and high sensitivity in biosensor technology.

The mentioned materials are synthesized using various methods such as solid-state reactions, sol–gel methods, chemical vapor deposition (CVD), and solution casting. These synthesis techniques play a crucial role in tailoring the properties and performance of the materials.

However, there are still some challenges that need to be addressed in the application of nanomaterials to energy storage devices. One of the key challenges is the scalability of the production of nanomaterials, which can be a complex and expensive process. In addition, the stability and durability of nanomaterials in harsh operating conditions need to be improved to ensure their long-term stability and performance. Furthermore, the cost-effectiveness of using nanomaterials in energy storage devices needs to be evaluated against traditional materials. Despite these challenges, the potential of nanomaterials for improving the electrochemical performance of energy storage devices makes them an important research area for the development of advanced energy storage technologies.

There are various factors that contribute to the performance of materials in electrochemical devices. Understanding these factors is crucial for optimizing the design and performance of these devices. The composition of the material plays a critical role in its electrochemical performance. Different materials exhibit varying chemical and physical properties, which directly impact their charge storage capacity, energy density, and stability. For example, in lithium-ion batteries, materials like lithium cobalt oxide (LiCoO₂) and lithium iron phosphate (LiFePO₄) are widely used due to their high specific capacity and long cycle life.

The method of synthesis greatly affects the structural characteristics and performance of materials. Various synthesis techniques, such as sol–gel, hydrothermal synthesis, chemical vapor deposition (CVD), and solid-state reactions, result in materials with different morphologies, crystal structures, and surface areas. These factors influence the electrochemical properties, including specific capacitance, energy density, and power density.

The surface area and porosity of materials significantly impact their electrochemical performance. Materials with high surface areas provide more active sites for electrochemical reactions, leading to improved charge storage capacity and faster reaction kinetics. The porosity of materials affects ion diffusion and electrolyte accessibility, influencing the overall device performance.

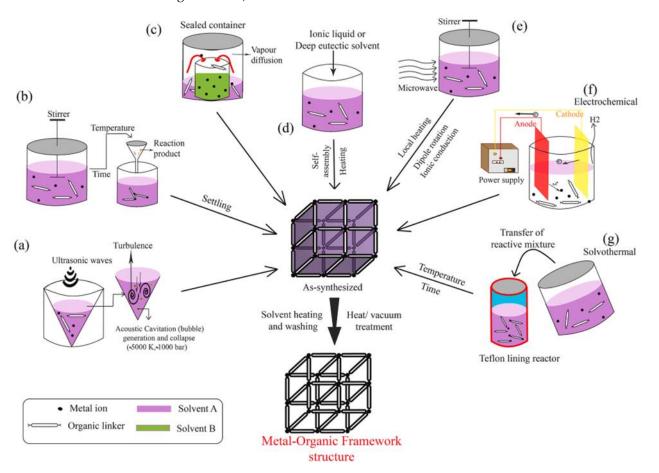
The morphology and nanostructure of materials play a vital role in determining their electrochemical properties. Nanomaterials, with their unique size-dependent properties,

offer enhanced electrochemical performance due to their large surface-to-volume ratio and shortened diffusion paths for ions and electrons. Materials with well-defined nanostructures, such as nanowires, nanotubes, and nanoparticles, exhibit improved electrical conductivity, ion diffusion, and stability.

The interaction between the electrode and electrolyte interface significantly impacts the performance of electrochemical devices. A well-designed interface with good electrode– electrolyte compatibility, minimized interfacial resistance, and effective charge transfer enhances the device's overall performance. Surface modification techniques and the use of specific electrolytes can improve the electrode–electrolyte interface and boost device performance. Understanding these factors and their interplay is essential for tailoring material properties to meet specific device requirements. By optimizing composition, synthesis methods, morphology, and interface characteristics, researchers can develop high-performance materials for electrochemical devices with improved energy storage capacity, cycling stability, power density, and overall efficiency.

7. New Types of Nanomaterials for Electrochemical Devices

Recent advances in nanotechnology have led to the development of new nanomaterials designed specifically for electrochemical devices. Among these emerging classes of nanomaterials are metal-organic frameworks (MOFs), covalent organic frameworks (COFs), and MXenes, which hold tremendous promise for the next generation of electrochemical devices. COFs are a type of crystalline porous material composed of light components bonded reversibly via covalent bonds in two to three dimensions. These materials have gained attention due to their unusual characteristics and wide range of potential uses [164]. Previously, it was challenging to create organic polymer networks with distinct pores of varying sizes, but the advent of reticular chemistry changed this. Reticular chemistry was initially used to manufacture MOFs, which are also porous materials. MOFs with very small dimensions, known as micro/nano MOFs, have better conductivity, a shorter diffusion distance for guest transport, and more accessible surface-active sites compared to bulk MOFs, without sacrificing any fundamental features of MOFs [165]. The structure of MOFs can vary depending on the reaction duration, particle size, yield, and morphology, even when using the same reactive combination (metal source, organic ligand, and solvent). Some approaches of synthesis lend themselves particularly well to implementation on a massive scale. Conventional solutions, diffusion synthesis, solvothermal synthesis, microwave synthesis, sonochemical synthesis, electrochemical synthesis, and iono-thermal synthesis are all employed to create MOFs, as shown in Figure 10. In addition, specific structural designs of micro/nano MOFs may confer novel functionalities and characteristics on conventional MOFs [165]. For instance, MOF nanowires oriented in a particular direction offer advantages for catalytic applications due to improved reactant and electron transport and the exposure of surface locations. When utilized as electrode materials, ultrathin metal–organic framework (MOF) nanosheets with high surface area-to-volume ratios may assure electrolyte penetration and improve the performance of energy storage devices [166]. Micro/nano MOFs have desirable features that make them attractive as electrode materials and high-performance catalysts for use in energy conversion and storage devices (such as rechargeable batteries, supercapacitors, and catalysis) [167]. The coordination modulation method allows for the expansion of MOF frameworks in specific directions by selectively coordinating modulators on particular crystal planes. This results in the formation of MOFs with different shapes, such as nanocubes, nanorods, and nanosheets. Researchers Sikdar et al. discovered that lauric acid can be used as a modulator to control the size and morphology of Zn-MOF. Without lauric acid, the MOF has an irregular shape due to metal ions combining with organic ligands. However, with the addition of lauric acid, the balance between the ligand and modulator coordination changes, leading to growth along a specific crystal plane and resulting in an anisotropic shape (as shown in Figure 10a). Varying concentrations of lauric acid enhance the competitive coordination, leading to



the formation of hexagonal nanoparticles, 1D nanorods, and 2D nanosheets (as shown in Figure 10b–d).

Figure 10. Different methods of MOF synthesis, (**a**) sonochemical synthesis method, (**b**) conventional solution method, (**c**) diffusion synthesis method, (**d**) iono-thermal process method, (**e**) microwave synthesis method, (**f**) electrochemical synthesis method, and (**g**) solvothermal synthesis method. Reproduced from Ref. [168] with permission from Elsevier, April 25, 2023. License Number 535901430300.

With 4-methoxy benzoic acid (MBA) as a modulator, Cu-MOF nanosheets were produced by Zhou et al. [169]. The MBA and BDC competed synergistically to generate highly distributed CuBDC-MBA-X when a Cu²⁺ and MBA combination was dripped into a mixture containing terephthalic acid (BDC). Two-dimensional nanosheets had their homogeneity altered by MBA. MOFs were created by the synthesis of a network of clusters containing polyatomic inorganic metal atoms connected by polytopic linkers [170]. Wang et al. [171] successfully synthesized a new hexagonal MOF nanoplate by using pyridine as a modulator. This MOF is composed of two Ni²⁺ cations, four BDC anions, and the nitrogen-containing terminals of 1,4-diazabicyclo[2.2.2]octane (DABCO) molecules. A paddlewheel secondary construction unit (SBU) is generated when pyridine and DABCO molecules fight for coordination, and this SBU subsequently self-assembles into 2D nanoplates. Because pyridine replaces some of the DABCO on the crystal's surface, a robust 2D growth trend is seen in its presence. As concentrations of pyridine were lowered, changes occurred in the shape of the nanocrystals and hexagons.

Using polyvinylpyrrolidone (PVP) as a regulator, Zheng et al. [172] synthesized Co-MOF of varying sizes. While both Co-MOFs had identical chemical and physical characteristics, the ultrathin 2D Co-MOF NS demonstrated superior performance in supercapacitor tests due to its greater exposure of active sites and faster electron transport. In addition, high-performance energy storage and conversion applications (such as rechargeable batteries, supercapacitors, and fuel cells) rely heavily on materials having hybrid micro/nanostructure. These multicomponent structures are difficult, yet they can potentially outperform their simpler single-component analogues. Because of its hybrid nature and the wide range of components available, the performance of any given application may be finetuned [173]. To date, several different synthesis methods have been developed to generate micro/nano hybrid structures; these approaches often include the use of precursors and sacrificial templates to build the complicated hybrid structures. Because the surface energy plays a role in the thermodynamic and kinetic processes of various re-actions happening at the interface of micro/nanocrystals, the design and management of different morphologies of MOF nanocrystals has been a popular issue in recent years [174].

Metal–organic frameworks (MOFs) are crystalline porous materials that are constructed from metal ions or clusters, which are linked together by organic ligand molecules. The modular approach used to create MOFs allows for the production of three-dimensional (3D) frameworks of varying topologies. MOFs are distinguished from their less-ordered counterparts such as activated carbon, metal oxide, and silica by their well-defined pore system [175]. MOFs have a wide variety of potential uses, including catalysis, the capture of greenhouse gases, and electronic device manufacturing, among others, which are enabled by their well-defined crystal structure. The storage and transformation of energy are among the many possible applications for metal–organic frameworks. Redox-active metals are particularly promising for electrochemical energy applications as they provide better charge transfer between the ligand and metal nodes. In the field of electrochemistry, MOFs have recently garnered much attention due to their high specific surface area, tunable shape, numerous pores, and adaptability. Publications on the energy-related uses of MOFs have increased significantly in the past two years [176]. Researchers have employed MOFs as building blocks to create nanomaterials with a wide range of shapes and sizes, including nanosheets, nanoparticles, nanospheres, and nanorods. However, the use of metal-organic frameworks (MOFs) in electrochemical devices poses several challenges. One significant challenge is their relatively low electrical conductivity, which can limit their use as electrode materials in electrochemical devices. This can be addressed by incorporating conductive materials into the MOF structure or by designing MOFs with higher intrinsic conductivity. Another challenge is the stability of MOFs in the presence of moisture, which can cause their structure to degrade and their performance to deteriorate. Researchers are working on developing MOFs with improved moisture resistance or implementing protective coatings to address this issue [177]. Additionally, the synthesis of MOFs can be complex and time-consuming, which can hinder their commercial application. Therefore, efforts are underway to develop simpler and more scalable synthesis methods for MOFs to overcome this challenge. Furthermore, although MOFs have exhibited potential in electrochemical devices, their electrochemical properties remain partially understood, and further research is necessary to optimize their utilization in practical applications.

COFs, which are the initial form of porous organic frameworks (POFs), have been successfully developed to incorporate organic building blocks into a systematically arranged structure [178] and may be created utilizing the principle of dynamic covalent chemistry, which involves linking together organic building blocks [179]. Through reversible thermodynamic bond formation, COF crystallization is facilitated via error correction and self-healing, whereby structural abnormalities are dynamically rectified [180]. Due to their composition of light elements linked via robust covalent bonds, COFs have exceptional thermal stability and possess low mass densities and permanent porosity [180]. COFs may be produced utilizing various energy sources, including heat, mechanical agitation, light irradiation, ultrasound, electron beams, and microwaves. Microwave-assisted synthesis has garnered significant attention due to its beneficial reaction features, such as reduced energy consumption, faster reaction speeds, and increased yields [181]. COF-5, a Boronate ester-linked COF, was synthesized in under 20 min at 100 °C under microwave irradiation by condensing 2,3,6,7,1,11-hexahydroxytriphenylene and 1,4-benzenediboronic acid, two hundred times quicker than the standard solvothermal method [182]. Compared to the

solvothermal synthesis, COF-5's Brunauer-Emmett-Teller (BET) surface area is higher at $2019 \text{ m}^2\text{g}^1$, whereas the solvothermal synthesis yielded only $1590 \text{ m}^2\text{g}^1$ [183]. Additionally, Bein's team utilized microwave-assisted synthesis to produce mesoporous Boronate ester-linked COF (BTD-COF), yielding a highly crystalline BTD-COF with a BET surface area of 1000 m²g¹ after two successive heating periods of 40 min [184]. Sonochemical synthesis drastically reduced the synthesis time by avoiding the induction phase, using less energy and, as a result, less money, and generating higher pressures and temperatures (>1000 bar and >5000 bar, respectively) due to the accelerated crystallization rate [185]. Sonochemically synthesized COF-5's yield is better, with a BET surface area of 2122 m²g¹, than a solvothermally manufactured equivalent [186]. While mechanochemical synthesis (MS) has been employed to create a wide range of porous materials, including metal oxides, porous carbon, metal–organic frameworks, and graphene derivatives, COFs remain a work in progress in terms of MS synthesis. MS is a low-cost, simple, and environmentally friendly technique. The photochemical synthesis method is useful for generating various functional materials, and Choi, Lim, and their colleagues established a photochemical method for synthesizing COF-5 (UV-COF-5) at room temperature in the presence of UV light, resulting in a 48-fold increase in growth rate and a BET surface area of 2027 m^2g^1 .

The production of functional materials was achieved by utilizing high-energy radiation, where Imine-based covalent organic framework EB-COF-1 was synthesized in just 160 seconds. This was accomplished by condensing 2,4,6-tris-(4-formylphenoxy)-1,3,5triazine and TAPB at room temperature in the presence of a 1.5 MeV electron beam. The rapid synthesis of the EB-COF-1 framework demonstrates the potential of high-energy radiation as a powerful tool for producing advanced materials with specific properties and functionalities [129]. This technology has opened up avenues for industrial-scale COF production and ensures their rapid synthesis.

Covalent organic frameworks (COFs) also face several challenges in their application in electrochemical devices. One significant challenge is their low electrical conductivity, which can limit their use as electrode materials in electrochemical devices. This can be addressed by incorporating conductive materials into the COF structure or by designing COFs with higher intrinsic conductivity [187].

Another challenge is the limited stability of COFs under acidic or basic conditions, which can cause their structure to degrade and their performance to deteriorate. This can be addressed by developing COFs with improved stability under harsh conditions or by implementing protective coatings.

The practical use of COF materials in energy-related devices is hampered by their poor stability or limited endurance. The collapse of COF structures in high aqueous acidic/alkaline electrolytes may make active centers inaccessible and hinder mass and charge transfer. Because most COFs are synthesized by solvothermal reactions of precursors in sealed glass tubes at elevated temperatures, resulting in completely insoluble powders comprised of randomly aggregated crystallites, poor process ability may be another major issue for their applications in electrocatalysis. This complicates the use of COF materials in energy-related equipment.

Consequently, it is preferable to create synthetic processing methods for building COFs with improved stability and process ability, which allows shaping, positioning, and orienting COFs as required, while integrating them with other components of the devices for stable functioning. Improving COFs' electrical conductivity is further required for enhancing their electrocatalytic performance and energy storage performance. One way to do this is to grow oriented COF thin films on a conducting support or to produce composites [188]. Finally, employing the novel stable and completely conjugated COFs made of cyanovinylene or olefin linkages would be beneficial for improving charge transport characteristics, which are required for electrochemical applications, and for increasing the recyclability (robustness) [188]. Additionally, the synthesis of COFs can be complex and time-consuming, which can limit their commercial application. Efforts are underway to develop simpler and more scalable synthesis methods for COFs to overcome this challenge.

Moreover, while COFs have shown promise in electrochemical devices, their electrochemical properties are not yet fully understood, and more research is needed to optimize their use in practical applications [189].

MXenes are a new type of 2D material composed of transition metal carbides, nitrides, or carbonitrides. They exhibit impressive electrochemical properties such as rapid specific capacitance, rapid charge–discharge rates, and great cycle stability [190]. Due to their high conductivity, MXenes are suitable as electrodes in electrochemical devices [107].

Despite graphene's significant contributions to scientific and technological advancements, its basic structure and chemistry limit its further development compared to other 2D materials. MXenes are a class of novel materials that have recently gained attention across various disciplines due to their many layers and adjustable features, which allow for a wide range of applications such as energy storage devices, catalysts, sensors, and biomedical applications [191,192]. Recent studies have shown that, like graphene, MXenes' surface functional groups can be fine-tuned to alter their electrical and electrochemical properties. MXenes exhibit high electrical conductivity, electrochromic behavior, antimicrobial characteristics, and transparency, making them attractive for various applications [189,190]. The exfoliation of the layered transition metal carbide Ti_3AlC_2 to produce MXenes using the top-down method involves immersing Ti_3AlC_2 powders in 50% hydrofluoric acid (HF). During exfoliation, OH and F groups are added to the surface, while Al layers are selectively scratched away [193]. MXenes can be broken down into ML powder by exploiting their inherent limitations. However, the search for inexpensive, environmentally friendly alternatives to traditional metals and plastics for use in high-capacity batteries and supercapacitors is ongoing. MXenes have exceptional electrical and electronic properties, such as a large specific surface area, a low energy barrier for electron transport, and a short iondiffusion route due to their atomic thickness, crystalline nature, and layered structure [194]. MXenes' electrical properties can be tuned by adjusting their surface terminations [194]. To develop effective energy storage devices in the future, researchers have been exploring novel applications of MXenes and MXene-based materials. MXenes face challenges in their application in electrochemical devices, including the difficulty of large-scale synthesis, susceptibility to oxidation in air, and sensitivity to the pH of the electrolyte, which can limit their electrochemical performance in certain devices.

At high current densities, the rate capability of MXene-based electrode materials is severely limited by the extremely high ionic diffusion resistance in the vertical direction that arises from stacking the layers of MXenes [195]. A possible way to improve MXenes' electronic conductivity, stability, and ion/electron transport is to structure them and create hybrid materials based on them. Improved electrochemical performance may result from the increased interlayer distances, which allow for the rapid passage of ions and electrolytes. Accordingly, further research has to be conducted to strike a good compromise between high-rate electrochemical performance and high-volume capacitance [196]. The influence of surface termination on the functional characteristics of MXene is poorly understood [197]. The optimization of the MXenes' surface terminations has great promise for controlling their characteristics, but a thorough study is needed to demonstrate the necessary relationships [198].

The key obstacles for MXene manufacturing and the limiting constraints for commercial development are the availability and expense of MAX phase powders and the usage of a significant volume of intrinsically dangerous high concentrated HF [199]. To reduce the environmental impact of MXene-based products, a concerted, multifaceted effort is necessary and desired [199].

The oxidation rate of MXenes may be slowed by removing the dissolved oxygen with dry nitrogen and storing the compound at low temperatures. Thus, the electrochemical charge storage mechanism of MXene-based electrode materials has been significantly impacted by aggregation of the produced MXene and impacts of MXenes' structural properties like basal spacing, surface chemistry, etc. [197].

One of the biggest obstacles to making flexible supercapacitor devices is striking a compromise between the mechanical features of MXenes (such as mechanical strength, toughness, flexibility, etc.) and their electrochemical properties [196].

Colloidal MXenes created by solvent sonication or by applying extensive mixing/shaking durations are widely employed in most research studies, although neither method demonstrates a viable technology with practical application [200]. To actualize the achieved and predicted property advantages, colloidal MXenes must be avoided; otherwise, the industrial adoption of MXene into the electrodes of energy storage devices would run into the same problems seen with graphene. For MXene to be used in industrial settings, the current high cost of manufacture must be reduced [196].

Despite these obstacles, the outlook for pure MXenes and MXene-based materials is brightly promising in not only the energy storage and energy conversion fields but also in a wide range of other fields including catalysis, environmental protection, and biomedicine. Specifically, improved performances may be achieved by a deeper knowledge of the electrochemical charge storage mechanism of MXene-based materials, but this requires extensive effort, novel concepts, and the application of cutting-edge research and investigation techniques.

8. Nanotechnology through Electrochemistry in Water Purification

Nanotechnology has the potential to address critical challenges in developing countries, particularly the significant public health hazard posed by waterborne pathogens in underdeveloped nations, which claim millions of lives each year. To minimize the risk of waterborne diseases, it is essential to remove and/or inactivate viruses, bacteria, and protozoa through point-of-use technologies [201]. Past research has mainly focused on the electrochemical production of active chlorine species (>2.5 V; HOCl, Cl₂ 3^-) or electrochlorination, which may generate hazardous disinfection byproducts [202]. Interestingly, studies have reported the successful elimination of bacteria and viruses by microfilters based on single-walled and multi-walled carbon nanotubes, which also possess antibacterial properties. Furthermore, carbon nanotubes have shown potential as substrates for photovoltaic and fuel cell applications. Hence, CNT-based microfilters may be employed in an electrochemical manner similar to carbon anodes for pathogen inactivation [203,204].

Boron-doped diamond anodes, which do not create effective chlorine species and are believed to be efficient for bacterial inactivation, might be a solution. Electrochemical processes are versatile and provide technologies for cleaning. Desalination in most countries is based on a technique called the reverse osmosis technique, and this method depends on the osmotic property [205]. The pressure exerted on the surface of the membranes is used to overcome the natural osmotic pressure of water. So, if a semi-permeable membrane is placed between two solutions of equal concentration under equal temperature and pressure, no water passage occurs through the membrane because of reciprocal chemical effort on both sides. If we add a soluble salt, the two solutions decrease pressure, so an osmotic flow of water occurs from the less salty side to the saltier one until the chemical voltage returns to its equilibrium state. This equilibrium occurs when the pressure difference in the saltier volume liquid becomes equal to the osmotic pressure [206]. Organic, inorganic, and biological contaminants may be water contaminants. Specific contaminants are toxic and carcinogenic and harm people and ecosystems. Highly toxic water pollutants are famous for some heavy metals. Arsenic is one of the deadliest elements in history. Cadmium, chromium, mercury, lead, zinc, nickel, and copper are other heavy metals and water pollutants with significant toxicity [207].

The population increase combined with the exploitation of water resources for domestic, industrial, and agricultural purposes has resulted in a shortage of freshwater supplies in many parts of the world [208]. Seawater desalination and brackish water treatment have become alternatives to freshwater resources. Different membrane processes are used, such as nanofiber and reverse osmotic pressure membranes, to extract potable water [209]. However, the limitations associated with existing membranes (i.e., dirt, biological decomposition, physical and mechanical change of membrane properties) result in reduced membrane productivity because of high energy requirements and the cost of cleaning procedures and regular replacement membranes [210]. In contrast, ultrafine nanofilms are less expensive and require fewer operational requirements than reverse osmotic pressure films. Despite their high productivity levels in water flow, their efficiency in filtering some saline ions is lower than that of osmotic pressure membranes. Additionally, they differ in terms of diffusion and distribution of pore size. Wastewater is defined as any water contaminated with organic or bacterial pollutants or microorganisms, whether from an industrial source or not, and can be divided into wastewater and industrial wastewater. Wastewater treatment includes primary and secondary procedures and a triple technique to remove residual protein from organic and inorganic materials and pathogenic microorganisms through filtration and sterilization. To improve water treatment, nanomaterials are used, such as nano-titanium oxide [211]. Nanoparticles of TiO₂ have become a new generation of advanced materials, with a diameter of 100 nm, and their various environmental applications, including gas sensors, are of great interest.

Recent studies have confirmed the importance of activated carbon in removing water pollutants. Granular activated carbon has numerous uses, particularly in aqueous solutions, and has increased in demand for various industrial applications [212]. The use of these GACs for water treatment has shown considerable promise, sometimes outperforming commercial carbons depending on the target pollutant [212].

The electrochemical method has been used to produce titanium dioxide nanoparticles. Inorganic mediums such as tetrahydrofuran and acetonitrile in a 4:1 ratio and a stabilizing agent increase the current density. Tetra-propyl bromide salt is a commonly used organic medium. Solvent polarity, electrode distance, and stabilizer concentration are used to control nanoparticle size. Electrochemical methods are applied to synthesize titanium dioxide nanoparticles for small-sized metal nanoparticles [213]. The overall process involves oxidizing the bulk metal on the anode, migrating the metal cations to the cathode, and decreasing them in a null-oxidation state by forming a metal or oxide. An ammonium stabilizer prevents agglomeration by creating unwanted metal powder [213]. The electrochemical deposition approach is therefore discovered to be innovative and very effective in suppressing microorganisms. Shashikala et al. [214] discovered that this approach delivers silver immediately in metallic form, unlike conventional ways of preparing silver catalysts. It is particularly cheap in recovering hydrogen by eliminating its usage in the process of catalyst manufacturing. Hydrogen is the most recent energy supplier, with several social, economic, and environmental benefits, including its use in chemical industries, which accounts for up to 30% of its usage. This implies the value of H_2 and its great demand in the world today and in the future [215].

Nanoparticles can be emitted into the environment from point sources, such as production plants and wastewater treatment plants, or non-point sources, such as washing machines, garbage, or other nanoparticle materials [216]. Due to their high specific surface, most nanoparticles accumulate and absorb immediately after water contact, losing their nanoscale characteristics. The surface configuration of polymers influences the agglomeration of nanoparticles in aqueous systems, such as polyethylene, providing interaction with different proteins [217]. Besides polymer nanoparticles, different proteins like fetal bovine serum can also interact.

Nanoparticles can be placed in natural water (lakes or rivers) and industrial storage systems for a long time as agglomerates or adsorbed with macro-molecules. The nanoparticles are bound to be "inactive" but not harmless because a changing environment, such as an increase in temperature or a decrease in pH, may cause nanoparticle dissolution. Different studies have calculated environmental concentrations of nanomaterials to estimate their impact on the environment. Gottschalk et al. [218] concluded that there is a risk associated with nanosilver, nano-TiO₂, and nano-ZnO when used for waste purposes in the USA, Europe, and Switzerland. The risk quotient is defined as the predicted concentration of the environment and the predicted no-effect concentration. Nanoparticles

can be degraded and sedimented through biological treatment by interacting with the microbiological community [219]. Due to their high specific surface, nanoparticles can form large particles or absorb larger particles, aiding in their separation. If the particles do not aggregate naturally, adequate flocculation surfactants can be added to cause larger clusters to form. For example, this process can easily remove colloidal C60 aggregates.

9. Green Nanoscale and Electrochemical Methods in High-Precision Economical Products

Recently, there has been growing interest in the natural synthesis of nanoparticles and the use of electrochemical methods to produce them [220]. This trend aligns with the concept of green nanotechnology, which aims to create safe, environmentally friendly, and energy-efficient products and processes while minimizing waste, greenhouse gas emissions, and reliance on non-renewable resources [221]. By integrating nanotechnology with other fields such as materials science, environmental science, electronics, and computing, green applications are emerging that leverage various technological advancements. For example, a study has shown that green cars with energy storage and release systems benefit from nanotechnology-enabled batteries, which improve their efficiency and use a natural mineral called lithium ferrophosphate (LiFePO₄) as an electrode component [222]. However, while nanotechnology plays a small role in the final product, green nanotechnology has significant potential to enhance sustainability and economic benefits while reducing risks and costs. Nevertheless, there are still concerns about the environmental and health implications of some green nanotechnology applications [223,224].

Nanostructures can be categorized according to their size and structure, and they can be synthesized using different atoms such as copper, calcium, magnesium, protein, and DNA [225–227]. Each of these formulas serves a specific purpose: Copper nanoflowers are used for efficient drug delivery, cell imaging, biosensing, and various medical applications [105]. Calcium nanoflowers improve the function of proteins in the environment, while nano-magnesium acts as a catalyst for better drug delivery and high-resolution imaging of cells. Researchers have also developed AuPt bimetallic nanoflowers using a one-step electrochemical reduction method [207,208]. To accomplish electrochemical reduction at low temperatures, this approach makes use of a unique mechanism involving the addition of 10% water to eutectic solvents. Glassy carbon electrodes (GCEs) modified with nanomaterials (AuPt NFs/GCEs) were made available for the first time by the researchers to be used as the anode in the electrical oxidation of XT to XO [228,229]. XO was synthesized by electrical oxidation of XT with a high yield of XO (Figure 11). The researchers achieved a high yield of XO through the electrical oxidation of XT with significantly lower applied potentials (0.80 V versus Ag/AgCl) in the modified reaction and electrode system, making the system safer than other oxidation potentials. In addition to oxidizing XT to XO, the system also had a low ability to cause side reactions.

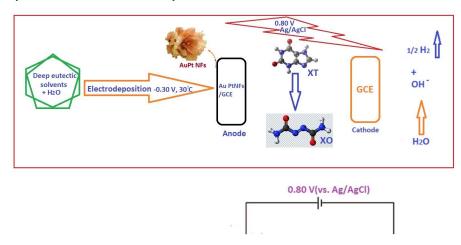


Figure 11. A glassy carbon electrode (GCE) modified with nanomaterials (AuPt NFs/GCE) as the anode for the electrical oxidation of XT to XO. Adapted from [228].

Bimetallic nanoparticles possess unique properties and have shown promise as catalysts with various applications [225,226]. The electrostatic deposition reduction method is a green, controllable, and efficient approach to synthesizing alloy nanoparticles compared to conventional methods [230]. Deep eutectic solvents, a new generation of green solvents, exhibit remarkable physical and chemical properties, making them useful in nanoparticle synthesis, electrochemistry, and biochemistry, besides ionic fluids. These natural products have significant biological and pharmaceutical properties, such as antibacterial, anti-cancer, and anti-viral properties [231].

In electrochemical oxidation reactions, potential control is critical to achieving safe electrical oxidation or avoiding unwanted side reactions [232]. Gold nanowires have been found to enhance the performance of cobalt oxide microelectrodes in electrochemical biosensors, which are widely used in clinical, environmental, industrial, and agricultural applications [233]. The catalytic activity of glucose oxidase (GOx) is sensitive to environmental conditions, such as temperature, humidity, pH, and toxic chemicals. The controlled use of nanostructured metal oxides can overcome the limitations of costly enzymes like GOx, thereby improving the sensitivity and stability of glucose detection [234].

The correlation between biosensing performance and the electrochemical and structural properties of electrodes has led to significant efforts to optimize charging and electronic transmission processes in advanced materials, thereby reducing primary resistance in bioanalysis [235]. Metal oxides with nanostructures offer promising applications as non-enzymatic catalysts in miniature glucose sensors due to their low cost, high biocompatibility, electrical catalytic activity, and enhanced electron transfer and adsorption capabilities [236]. Researchers have focused on simpler and more manageable methods to fine-tune pore size and np-Au ligaments for various applications. Electrochemical methods provide proper control over pore and ligament sizes, making np-Au electrodes an efficient and easy-to-handle option for electrochemical biosensing [237].

The surface morphology, pore size, and ligament size of np-Au can be determined using scanning electron microscopy (SEM) [238]. For higher resolutions, transmission electron microscopy (TEM) can be used to study thin np-Au films. X-ray energy dispersive spectroscopy can offer information on mineral composition, aiding in the validation of the exclusion of metals from np-Au.

Electrochemical techniques are significant for assessing the surface area and kinetics at the np-Au interface. The method of choice for determining the np-Au electrode surface area is cyclic voltammetry (CV). In CV, a particular scan rate is used to generate a triangular waveform and calculate the resulting current [238]. In the reverse scan, the Au-electrode immersed in diluted H₂SO₄ is treated with an anodic scan to form a gold oxide layer. The charge beneath the cathodic peak is measured, and the surface area of np-Au is calculated based on the required charge to extract gold oxide from a square centimeter of the gold surface. However, due to the double-layer charging, the contribution from other faradaic processes, and variation in the metal's crystal face, there is always the possibility that this value will be lost. The geometric surface area is divided from tens to thousands, depending on the preparation methods, the number of materials, and the size of the pores, and it is possible to determine the roughness factor of np-Au against a planar surface [239].

Silver nanoparticles have attracted researchers' attention due to their appropriate applications in various domains, including electronic science, physical sciences, and medicine. Nanotechnology occupies a prominent place among innovative approaches to developing agricultural processes and food production. The wide variety of methods for preparing nanoparticles in materials science, energy, medicine, and life sciences research has contributed to the use of nanotechnology and the expansion of its fields [240].

Moreover, from its applications in genetically changed crops and agricultural chemical production techniques, silver nanoparticles show potential antimicrobial effects against infectious organisms such as Escherichia coli, *Bacillus subtilis, Vibrio cholera, Pseudomonas aeruginosa, Syphilis typhus,* and *Staphylococcus aureus* [240]. More recently, Ag-NPs have

been used in a wide range of applications in fields such as mechanics, optics, biomedical sciences, chemical industries, and electronics.

Different electrochemical techniques, thermo-destructiveness, laser ablation, and microwaves have been used in manufacturing silver nanoparticles [241]. To reduce the electrochemical silver, a galvanostatic method (electrolysis) was used in this proposal. A simple system of three silver anodes ($50 \times 10 \times 0.2 \text{ mm}^3$) and two silver wires (0.8 mm diameter) as cathodes was placed at the half-distance between the anodes. Anodes were positioned perpendicular to the direction in which the electrodes were placed. The distance between neighboring electrodes was 15 mm [242]. The electrolysis was performed at 323 K with a current frequency of 0.5 mA cm⁻². A Phillips CM20 TWIN microscope operating at 200 kV was used to investigate the size, morphology, and composition of the obtained nanoparticles using transmission electron microscopy (TEM) (Figure 12A), energy dispersive X-ray analysis (EDX) (Figure 12B), and electron diffraction. Silver oxidation is noticeable above 0.4 V in the previous work on rising current densities in the anode curve. This silver anode dissolution process proceeds on a surface with a one-electron reaction. The nanoparticles form a polarization of silver in an ethanol solution, either potentiostatically or galvanostatically. In the presence of ethanol, the proposed process mechanism assumes anodic silver oxidation and dissolution. Aldehyde was obtained simultaneously with the anode during ethanol oxidation to reduce Ag_2O in the last step. The presented method is simple and ecological, does not require costly tools, and there will be no need for surfactants or other reduction agents.

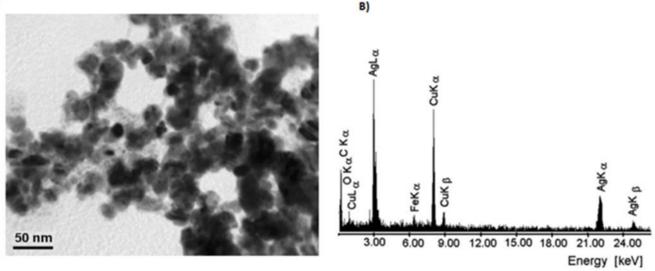


Figure 12. (**A**) TEM assessment was conducted on the particles obtained by the galvanostatic method. (**B**) The EDX spectrums of particles obtained through the galvanostatic method indicate the characteristic X-ray lines of the observed elements. Ref. [242], permission from Elsevier, 28 April 2023. license number 5533131415449.

Another study described an efficient electric approach for colloidal AgNP synthesis using bulk silver, bidistilled water, trisodium citrate, and an applied direct current (DC) voltage source [243]. A substantial majority of colloidal AgNPs can be derived in one process with a high antibacterial effect, as synthesized AgNPs could be used for different biomedicinal uses. This way, the magnet stirrer was connected as an electrode in the 500 mL glass beaker to two silver bars parallel to the direct voltage source. The two electrodes were 6.5 cm apart, and bidistilled water was filled in with silver electrodes up to 7 cm. The magnetic mixing was performed for 0.016 percent (wt%) of trisodium citrate. For a further 2 h under magnetic revolution and room temperature, it supplied a 12 V DC voltage to the silver bars. The stirring was still in operation for half an hour to prevent the scattering

A)

of the residue produced during synthesis. The solution beaker was kept in the dark for 24 h to complete the AgNP formation. At 2000 RPM, the AgNP solution was decanted and sediment-free before use and measurements at 2000 RPM for 10 min. The colloidal AgNP solution for Gram-positive and -negative bacteria achieved high antibacterial effectiveness. The advantages of the above method show the simplicity of preparation, high efficiency, uniformity, and purity of colloidal ANPs. A recent study illustrated evaluating photo and electrocatalytic activities in Iso-AgNPs using isoimperatorine, a furanocoumarin-natural, as a significant decrease in the synthesis of synthesizing isoimperatorin mediated silver nanoparticles (Iso-AgNPs) [244].

Iso-AgNPs, which were spherical particles ranging from 79–200 nm in size, exhibited catalytic activity for sunlight-induced degradation (increased yield). This suggests that Iso-AgNPs hold potential as effective catalysts for environmental remediation applications that require sunlight-induced degradation. Compounds contained 4-chloro-fluorescein, methylene blue, erythrosine B, and new fuchsine. Degradation rates for MB, NF, ER, and 4-CP with iso-AgNPs were 96.5%, 96.0%, 92.0%, and 95.0%, respectively. Iso-AgNPs showed an excellent electrocatalytic activity towards H_2O_2 in many biological systems without additional binder and conductive additives. There is a wide range of catalytic applications for synthesized nanoparticles. A dropping method has been developed as Iso-AgNPs can change glassy carbon electrodes (GCEs).

The working electrode was polished with alumina powder to achieve a mirror-like surface. A 5 μ L volume of an aqueous solution of Iso-AgNPs was directly applied to a cleaned area of the glassy carbon electrode (GCE) and allowed to dry at room temperature, resulting in the formation of Iso-AgNPs/GCE. This process ensures the uniform and controlled application of Iso-AgNPs to the GCE surface, which is critical for accurate and reliable electrochemical measurements.

The electrocatalytic activity, stability of Iso-AgNPs, and the decrease in H_2O_2 in 0.1 M PBS was assessed using cyclic voltammetry and differential pulse voltammetry. The PBS buffer was purged with ultra-pure nitrogen for 10 min to remove dissolved oxygen before measuring.

10. A Fusion of Nanostructures with the Electrochemical System in Applications of Economic Importance

Nanoscale working techniques have become essential in electronics, and nanoscale materials are emerging in consumer products, although it may take decades to develop small usable devices [245]. For example, millions of nano-lines are molecularly applied to natural and synthetic fibers to impart resistance to clothing and other fabrics at a scale of around 10 nanometers. Zinc oxide nanocrystals are used to create invisible sunscreens that block UV rays, and silver nanocrystals are added to dressings for killing and preventing bacterial infections [246]. The future scope for nanotechnology is vast. It is possible to manufacture lightweight, robust, and programmable materials that require less energy to build than traditional materials, which produce less waste and increase fuel efficiency in land, ship, and aircraft transport compared to conventional manufacturing. Additionally, electronic, magnetic, and mechanical devices and data processing systems can be manufactured for human and environmental conservation, as well as chemical, optical, and biological sensors. Innovative photovoltaic materials will make efficient solar panels possible, and molecular hybrid semiconductors will drive the next revolution in the information age. There is enormous potential for improvement in health, safety, quality of life, and conservation, and we must address these opportunities to reap the benefits of nanotechnology [247].

Hierarchical assemblies of nanoscale materials, or their incorporation into devices, provide a basis for the radical design of new materials and machines, even those intended for large structures. This advance in structural materials benefits the automotive industry because reduced weight leads to improved fuel efficiency [248]. Additional improvements can improve safety or reduce the environmental impact of manufacturing and recycling.

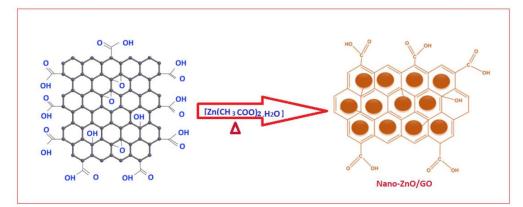
Further advances, such as active materials showing impending failure or even being capable of self-repairing faults, may be possible.

For almost every modern control system, sensors are critical. Multiple sensors, for example, are used in automobiles for various tasks, such as motor control, emission control, safety, convenience, vehicle monitoring, and diagnostics [249]. Nanosensors exhibit unexpected speed and sensitivity because of their small size and sometimes extend to the detection of individual particles. Nanowires made of carbon nanotubes, silicon, and other semiconductor materials, for example, have an extraordinary sensitivity to chemical species or biological substances. The charging-induced changes in current can detect different species on the nanowire surfaces coated with sensor molecules, which selectively bind species. Many classes of sensor systems have adopted the same strategy [250]. There will be several applications for new kinds of highly sensitive and specific sensors. When composed of a few cells, sensors that can detect tumors are a significant development. Various nanostructure fabrication technologies are becoming significant in sensors. Nanostructured materials have sparked great interest in many sensor applications because of their superior physical, chemical, and plasmonic properties [251]. Although there are many applications for these nanostructures in sensor applications, certain limitations can restrict their use. Gas sensors made of carbon nanotubes, for example, are nonselective. For example, there was a lack of selectivity for gas sensors based on carbon nanotubes [252]. This lack of selectivity is an essential factor that can prevent the further use of these CNT-based devices, which can be reduced by the combination of CNT with other materials. There are also technical problems in the manufacturing of nanostructures and serious concerns concerning toxicity of nanostructures that could vary in the quality of each new type of particle's physical characteristics [252]. Many sensors, such as electrochemical sensors, enzyme-based biosensors, genetic sensors, immune sensors, and cellular sensors, are available. Relevant electrochemical sensor developments open new avenues and strategies for research into the future [253]. The high specificity of enzymes is combined with the sensitivity of electrochemicals in the electrochemical enzyme-based sensor. Enzyme electrodes are electrochemical samples on the surface of the working electrode with a thin layer of immobilized enzyme. Because it provides selectivity for the sensor and catalyzes the formation of the electroactive substance for detection, the enzyme is the most significant part of the enzyme electrode. The production of various materials, techniques, and applications of electrochemical enzyme-based biosensors has been the subject of several studies in the last two years [254]. Nanoforms of minerals such as silver, titanium, and zinc and their oxides are used in several kinds of toothpaste, cosmetics, sun protection, paint, clothes, and food. Carbon nanotubes (graphene) also have exceptional properties. They are stronger than steel, conduct electricity better than copper, and have a higher thermal conductivity than diamond [255]. In a recent study, nanosynthesis, characterization, and implementation of zinc oxide-graphene oxide (ZnO/GO) in the electrochemical system have been carried out. Because of its exceptional properties, for instance, large area size proportions and low production costs, graphene oxide has been given considerable attention in science and engineering. It has emerged as a new carbon-based nanomaterial that provides an alternative route to graphene. Graphene oxide (GO) is a feedstock for graphene, and it has unique properties from its analog.

The general structure of graphene oxide can be described as a hexagonal carbon lattice in which some carbon atoms exhibit sp2 hybridization and others exhibit sp3 hybridization due to the presence of oxygen functional groups. These oxygen groups can be found attached to both two- and three-hybridized carbon atoms, resulting in a highly functionalized graphene lattice. These oxidative functions give it fluctuation properties by being at the hydrophilic boundary and hydrophobic level. Zinc oxide (ZnO) is an inorganic and non-toxic semiconductor compound. Its chemical and thermal properties are stable; as an inexpensive material, this makes it a significant material for research. A unit crystal cell with constants $a = 3.2499A^0$ and $c = 5.2060A^0$ where the u-coordinate is from creates the following relationship [256]:

$$u = 1/4 + a^2/3c^2 = 3/8 = 0.375$$
 Wurtzite

The compact formation of the ZnO/GO nanocomposite was stated by microscopy of electric field scans, HRTEM, X-ray diffractive, and reduced overall reflectance spectroscopy [257]. Compact ZnO/GO nanocomposites are also confirmed (Scheme 4). The efficiency of direct electron transfer was therefore confirmed and was used for hydrogen peroxide amperometric detection. In another analysis, the GO/ZnO nanocomposite was produced with ZnO nanoparticles synthesized by decorating expanded and oxidized graphite oxide nanosheets. Photocatalysts were used to degrade basic fuchsin (BF) color. The production process was based on the deposition of a two-step sol–gel process. A twodimensional structure with 0.54Go/0.46ZnO (w/w) composition and an average ZnO particle size of 25–30 nm was developed by the GO/ZnO nanocomposite. The GO nanosheet ZnO nanoparticles have 3.25 eV bandgaps, while the bulk ZnO nanoparticles have a 3.70 eV bandgap [140]. This research could lead to the growth of structurally engineered and efficient composite catalysts by providing detailed technical details for catalyst synthesis, characterization, and testing of performance issues [258].



Scheme 4. A diagram showing the ZnO/GO nanocomposite preparation and the electrode manufacture of the sensor. Adapted from [259].

At the bench level, global efforts are still working on developing desired materials that can treat water supplies. The principal trustworthy applicant suffered from many challenges considering adsorbent and photocatalytic materials that limit its large-scale application. Magnetic activated carbon, nanotubes (CNT), graphene (G), quantum GO, carbon nanotubes and reduced graphene oxide (RGO), zeolite, silica, and clay-based nanomaterials are recently advanced water treatments. Materials are available in various countries [260]. Thus, water-treating techniques have been developed.

The adsorption and photocatalytic properties of these nanomaterials make them a promising option for the removal of heavy metals and organic dyes. Additionally, their photocatalytic properties enable the degradation of pollutants under light irradiation, further enhancing their potential for environmental remediation applications. The main features of these materials make them suitable to treat wastewater even in a low pollutant concentration in combination with higher kinetics of adsorption or decomposition [260].

11. Future and Challenges

Nanomaterials have shown great potential in electrochemical devices. However, several challenges and limitations need to be addressed before their widespread adoption can take place.

One of the key challenges is the synthesis and scalability of nanomaterials. The synthesis of nanomaterials can be challenging due to their small size and complex structures

and may require specialized equipment and expertise. Scaling up the production of nanomaterials to meet commercial demands can also be difficult, especially if the synthesis process is complex and expensive [261].

Another challenge is the stability and durability of nanomaterials. Nanomaterials can be susceptible to degradation over time, especially under harsh operating conditions. This can limit the lifetime and reliability of electrochemical devices that incorporate these materials. Developing new synthesis methods and surface modification strategies that can protect the materials from degradation will be critical to improving their stability and durability [262].

Nanomaterials also have the potential for toxicity and environmental impact. Some nanomaterials are toxic to living organisms, especially at high concentrations. In addition, the environmental impact of nanomaterials is not yet fully understood, and their potential effects on ecosystems and human health need to be carefully evaluated [263]. Rigorous testing and evaluation will be necessary to address the toxicity and environmental impact of nanomaterials used in electrochemical devices.

Integration and compatibility with existing device components are other challenges for nanomaterials. Nanomaterials may not always be compatible with certain electrode materials or electrolytes and may require specialized equipment or conditions that may not be compatible with existing device manufacturing processes. Developing new integration strategies and improving compatibility with existing device components will be critical for the successful incorporation of nanomaterials into electrochemical devices [264].

Finally, cost-effectiveness is a limitation of nanomaterials in electrochemical devices. While some nanomaterials can be synthesized at a low cost, others may require more expensive production methods. Additionally, the cost of incorporating nanomaterials into electrochemical devices can be higher than traditional materials, which can limit their commercial viability [265]. Developing new synthesis methods and improving scalability will be critical for reducing the cost of nanomaterial production. Identifying applications where the performance benefits of nanomaterials outweigh the additional cost will also be important for maximizing their commercial potential. According to some studies, nanoparticles may cause symptoms similar to those induced by asbestos fibers. The inhalation of TiO_2 particles of an elemental particle size between 2 and 5 nm has resulted in pneumonitis at a concentration of 8.8 mg per metric m³, as confirmed by other authors [266].

Carbon nanotubes (CNTs), including nanocrystals and quantum dots, have a lot in common in creating more sophisticated and powerful solar cells [267]. Today, we can obtain multiple groups of these advanced nanotubes and control the change in the shapes and sizes of their grains. In nano-energy, significant work has been conducted to develop an all-in-one system made up of multiple components that, when integrated, could produce synergistic effects and meet all the criteria for a successful solar-to-fuel conversion [268].

Silicon electrodes now stand out because they have nearly ten times the power of a graphite anode. Intelligent structural designs aim to combat instability caused by massive volumetric changes. Nanowires, porous and core–shell structures, and surface change composites produced with CNTs and graphene have been documented to have a low specific ability compared to theoretical values [269].

Scientists are adjusting their research methodologies to create renewable energy from readily available resources at a low cost. Because of its large surface area, optical characteristics, and catalytic nature, they discovered that Nanoparticles (NPs) are the optimum contender for this purpose. Therefore, NPs are used to generate energy from photoelectrochemical and electrochemical water splitting, particularly in photocatalytic applications [270].

Green nanotechnology applications can clean polluted surroundings such as air and water and prevent climate change by lowering gas emissions and hazardous waste. It is still not clear how green nanotechnology will achieve environmental sustainability in the future. The progress of green nanotechnology solutions must mitigate these risks [271].

As part of a comprehensive initiative toward green nanotechnology, research is performed to produce safe and non-toxic nanomaterials to minimize potential risks.

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The properties of nanostructures that lead to increased sensor selectivity are a significant downside of electrochemical sensors. Furthermore, the shape of nanoparticles is particularly important in identifying their applications. Recently, the tip-head ellipticity in gold nanoparticles (AuNPs) has been identified as a physical parameter that directly affects electromagnetic radiation scattering [272].

Intensive research using nanotechnology and electrochemical methods holds promise for making desalination more efficient. Much of this research focuses on improving the efficiency of the membranes used in reverse osmosis and other membrane desalination processes [273]. Nanotechnology-based membranes have been shown to achieve productivity gains of up to 20%. Using packed carbon nanotubes in reverse osmosis membranes can reduce the costs of desalination to the level of technology [274]. For water technology to be sustainable and cost-effective, we need to invest more in best practices that promote efficiency and sustainable energy. In hard water and seawater, nanoparticles appear to accumulate, and the type of organic matter or other natural particles (colloids) present in freshwater has a significant impact [275]. Dispersion affects ecotoxicity, but several abiotic factors that influence it, such as pH, salinity, and organic matter, have yet to be thoroughly investigated in ecotoxicological studies [276].

The issue is how to implement new technology on a small scale and then scale it up to commercial use on a large scale, and there is no single new technology that can solve this problem. Performance, cost, safety, compliance with regulations, and lower environmental impacts are some of the barriers that can prevent new technology from being used on a large scale. Novel solutions in large-scale research can help identify current obstacles and develop solutions that enhance efficiency, cost, safety, environmental acceptability, and customer satisfaction [277].

Titanium dioxide has recently gained popularity as a large-scale semiconductor material used in the fields of energy conversion due to its low cost, zero contaminants, relatively high performance, and excellent chemical stability. However, it faces challenges such as reduced carbon dioxide absorption and fast electron–vacuum recombination [278]. Carbon materials, in addition to suitable metals and metal oxides, are a viable option. The electrochemical reactor system was designed and innovated to convert carbon dioxide from the solid electrochemical state to chemicals with benefits [279]. We recommend that nanoparticles be used carefully, especially for unknown toxic materials. Some authors consider the risk of nanotoxicity to be superficial in terms of concentration volume and nanoparticles.

12. Conclusions

The conclusion drawn from the extensive analysis and findings presented in this manuscript highlights the significant role of nanomaterials in shaping the future of various industries and scientific domains. The demand for new devices capable of manipulating materials at the nanoscale underscores the need for continuous advancements in the field of nanotechnology. Computer simulations of atomic and molecular structures have emerged as crucial tools in realizing the full potential of nanomaterials.

Nanomaterials have demonstrated their exceptional utility in enhancing electrochemical performance, thanks to their unique properties such as large surface areas, distinct structures, and abundant active sites. Their application extends beyond performance enhancement, with an emphasis on cost reduction in the development of nanotechnology products. This pursuit of improved efficiency and affordability opens up new avenues for widespread adoption and commercialization of nanomaterial-based solutions.

Looking ahead, the future of nanomaterials appears incredibly promising. These materials are poised to play a vital role in addressing the increasing energy needs of the future by powering next-generation devices. Additionally, they are anticipated to revolutionize biosensors and nanomedicine, with far-reaching implications for diagnostics and medical treatments. The ongoing advancements in nanomaterial research will continue to drive innovation and contribute to addressing global challenges.

In essence, the exploration and application of nanomaterials present remarkable opportunities for scientific and technological advancements. By harnessing their exceptional properties and leveraging advanced computational tools, we can unlock transformative possibilities in material design, energy technologies, and healthcare. The remarkable potential of nanotechnology ensures a future characterized by sustainability, enhanced performance, and groundbreaking innovations.

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Abbreviations

1D	One-dimensional
2D	Two-dimensional
3D	Three-dimensional
AgNPs	Silver nanoparticles
Au	Gold
AuPt	Gold-platinum
BET	Brunauer–Emmett–Teller
BF	Basic fuchsin
BTD-COF	Boronate ester-linked COF
CNTs	Carbon nanotubes
CNTs	Carbon nanotubes
Co	Cobalt
CO_3O_4	Cobalt tetraoxide
CoFe ₂ O	Cobalt ferrite
COFs	Covalent organic frameworks
Cu	Copper
CuO	Copper oxide
CV	Cyclic voltammetry
EB	Erythrosine B
EDX	Energy dispersive X-ray
ES	Electrical sensors
GCE	Glassy carbon electrode
GO	Graphene oxide
GOX	Glucose oxidase
HPLC	High-performance liquid chromatography
Iso-AgNPs	Isoimperatorin-mediated silver nanoparticles
LC/MS	Liquid chromatography-mass spectrometry
Li	Lithium
LiFePO ₄	lithium ferrophosphate
MA	Methyl-ammonium
MB	Methylene Blue
MOFs	Metal–organic frameworks (MOFs)
MoP-NC	Molybdenum phosphide nanoparticles and nitrogen-doped carbon
MS	Mechanochemical synthesis
NaOH	Sodium hydroxide
NF	New Fuchsine
NFs	Nanoflowers
Ni (OH) ₂	Nickel(II) hydroxide

Ni	Nickle
NiONPs	Nickel oxide nanoparticles
NiP	Nickel phosphide
NPS	Nanoparticles
POFs	First covalent organic frameworks
Pt	Platinum
RGO	Reduced graphene oxide
SEM	Scanning electron microscope
Si	Silicon
SiCNPs	Silicon carbide nanoparticles
SiO ₂ NPs	Silicon Dioxide Nanoparticles
TEM	Transmission electron microscope
TiO2	Titanium dioxide
ХО	Xanthone
XT	Xanthene
ZnO	Zinc oxide

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