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Original scientific paper

Transition metal oxide-modified carbon electrodes: A comprehensive review on surface area modulation and Randles-Ševčik electrochemical insights

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Abstract

The exceptional catalytic properties of transition metal electrocatalysts, including high activity, stability, electronic properties, and selectivity in various electrochemical processes, make them essential in electrochemistry. The combination of these electrocatalysts with carbon-based interfaces improves their performance by providing increased electrical conductivity, greater surface areas, and robust structural support. Expanding the electrode area by offering more active sites for the processes and boosting efficiency increases the catalytic activity. This review emphasizes the importance of maximizing electrode surface area and the variations in transition metal oxides (TMOs) with carbon interfaces to achieve excellent performance in various electrochemical applications. Special focus is given to the Randles-Ševčik equation as a fundamental tool for evaluating the electroactive surface area and charge transfer properties of TMO-modified electrodes. Furthermore, the review discusses the influence of structural modifications on catalytic performance. By summarizing recent advancements and challenges, this study aims to provide a comprehensive understanding of the potential of transition metal oxides as efficient electrocatalysts for sustainable energy and sensing applications.

Keywords

Transition metals, electrode modifiers; electrocatalysts; cyclic voltammetry, surface area assessment

Introduction

A series of transition metals exhibits rich catalytic activity owing to their anomalous electronic structure and their ability to readily attain multiple oxidation states [1]. This unusual property of transition metals arises from the unique nature of their d-electrons [2]. A good way to enhance

electrocatalytic performance is to introduce transition-metal catalysts into carbon-based materials. Such characteristics promote effective electron transfer and improved metal catalyst dispersion, leading to better catalytic performance [3]. Carbon materials, such as graphene and carbon nanotubes [4], are well known for their large surface area, superior electronic conductivity [5], and efficient structural support. Changes in the electrical characteristics of transition metals at carbon interfaces may enhance their reactivity and stability in an electrochemical environment. Apart from these, the electrocatalytic efficiency is highly influenced by the electrode surface area. Since the number of potential reaction sites increases with the electrode area, catalytic efficiency will improve. This is very important in practical applications because an increased active electrode surface can produce higher reaction rates and lower overpotentials. Hydrothermal/solvothermal routes frequently yield high-surface-area nanostructures (nanorods, nanoflakes) and therefore a large electroactive area [6]. Sol-gel and co-precipitation can give fine control of composition and doping, but require careful calcination to avoid pore collapse [7]. The electrodeposition technique produces well-adhered thin films with good electrical contact and lower charge-transfer resistance, but often with a smaller geometric surface area [8]. Template-assisted (hard/soft) syntheses reliably produce hierarchical porosity, but at the expense of more complex processing.

Transition metals form oxides that are more stable than those of other metals, owing to higher lattice energy and the involvement of d orbitals in bonding. The oxides are generally a perfect choice for electrode modifiers, because of the combination of high catalytic efficiency, customized characteristics, stability, and economic feasibility [9]. Transition metal oxides (TMOs) display a broad range of electrical, magnetic, and structural properties. Partly, the ability of transition metals to adopt several oxidation states and form varied crystal structures accounts for their diversity. TMOs are the most diverse group of compounds with substantial utility in electrochemistry, chemical science, catalytic activity, and many more [10]. Low conductivity of TMOs contributes to their overall electrochemical properties. Their fast electron and ion mobilities accelerate reaction kinetics, making them suitable for electrodes of fuel cells, supercapacitors, and batteries [11]. Due to their extensive surface area, TMOs could accommodate many active sites for electrochemical reactions, thereby making such electrochemical processes more efficient. Furthermore, doping or compositional modifications can tune the electrical properties of TMOs, enabling optimization for specific applications. In addition, most TMOs are very stable at high temperatures and in chemical environments, which guarantees their robustness in reliable devices that can survive the most hostile environments. Cost-effectiveness is also a major advantage, with TMOs often employing more accessible and less expensive materials than their precious metal counterparts [12]. They are also benign environmentally, very easy to produce, and are known to be available in more than one form, which expands the applications of TMOs in a variety of electrochemical systems [13].

Owing to their unique physicochemical properties, TMOs are excellent candidates for electrode modification across a wide range of electrochemical systems. To characterize and quantify their surface activity, redox behaviour, and charge-transfer kinetics, techniques like cyclic voltammetry (CV), chronoamperometry, and electrochemical impedance spectroscopy (EIS) are routinely used. CV cycling is the most popular among these, as it is a sensible tool for electrode surface activation, stability evaluation, and confirmation of reproducibility through repeated scans [14-16].

This research was chosen for the explicit reporting of literature data on electroactive surface area, Randles-Ševčik analysis, and extractable cyclic voltammetry data, which permit estimation of diffusion coefficients and surface-area modulation. The highest preference was given to reports in which

surface morphology and electrochemical parameters were correlated directly with catalytic performance. For consistency, publications on TMO-based carbon electrodes were prioritized, while systems lacking quantifiable surface-area information were removed. In this review, priority was given to five members of the first-series transition metal oxides, namely iron, nickel, copper, cobalt, and zinc oxides, due to the general accessibility of quantitative electrochemical data for Randles-Ševčik analysis. The choice was deliberately restricted to reports where the extractable electroactive surface area, diffusion coefficients, or linear Randles-Ševčik correlations are specifically documented. The number of available reports on other oxides like MnO_2, TiO_2, or RuO_2 was relatively less under this framework and almost concentrated to application in photocatalysis, energy storage, or supercapacitors rather than comprehensive electrochemical surface-area assessments. These systems were thus not examined extensively here. Their intrinsic catalytic and redox properties, however, make the extension of Randles-Ševčík-based surface-area assessments to these materials a potentially valuable avenue of future research.

Stability of transition metal oxide electrode components

A complex system of interrelated elements affects the stability of electrode-modified transition metals in electrochemical processes, as in Figure 1. Several important aspects influence the electrode stability in electrochemical applications, including mechanical, chemical, and electrochemical stability [17,18].

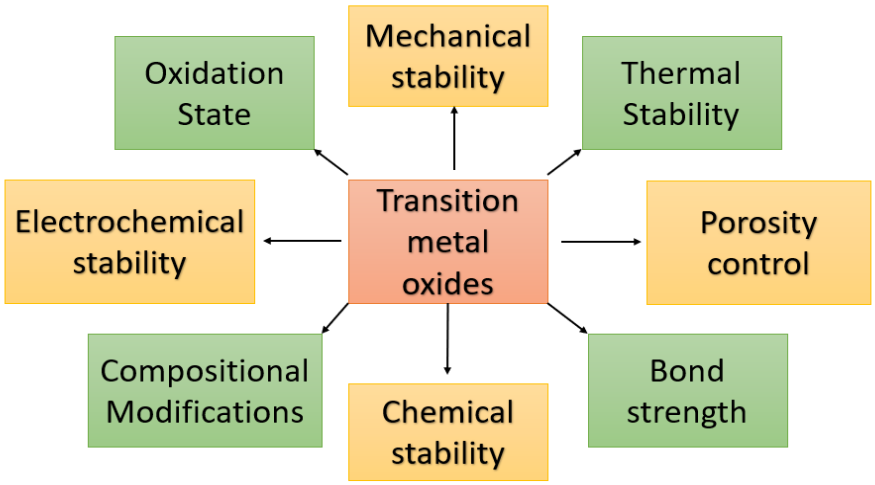

***Figure 1**. Factors influencing the stability of transition metal oxides*

To avoid phase transitions and undesirable reactions with the electrolyte, chemical stability is essential. Important considerations are the morphological control of the material (particle size and porosity), interfacial stability at the electrode-electrolyte boundary, and thermal stability [19]. By strengthening the electronic structure and resistance to degradation, compositional changes improve stability and ensure long-term durability of the electrode under demanding operating conditions [20]. Transition metals can form stable intermediates during the reaction, facilitating the overall process [21]. Employing various techniques, such as CV to track redox behaviour, EIS to monitor changes in resistance, and X-ray diffraction (XRD) to detect structural changes, researchers examine and aim to enhance the stability of TMO materials [22]. This understanding has led to the development of various strategies to stabilize oxides, *i.e.* to stabilize their crystal structure by doping with other metal ions, optimizing their conductivity and dispersive properties by incorporating carbon-based materials, and finally, designing core-shell structures that will protect the active oxide from being attacked by the electrolyte. It follows that doped TMOs and hybrid materials such as

TMO-carbon composites exhibit better cycling stability and are more resistant to degradation; they may be more suitable for energy storage, conversion, and electrocatalysis [23]. Though progress has been made in the area of TMO electrodes, these electrodes are still under significant research to further optimize them for practical-scale use in energy and environmental technologies [24].

Carbon electrode materials and carbon interfaces

Properties and advantages of carbon materials in electrocatalysis

Carbon interfaces play an essential role in amplifying electrocatalytic activity by significantly enhancing several vital aspects of the process [25]. The high surface area of carbon materials offers many electrochemically active sites [26,27]. Electrocatalysis is a process that uses a catalyst to accelerate electrochemical reactions at the electrode surface, thereby enhancing electrochemical processes at the electrode-electrolyte interface. The carbon supports stabilize the metal oxides by preventing sintering and leaching. The enhanced electron conductivity is an important factor in maintaining a high electrocatalytic reaction rate. Moreover, carbon materials serve as low-cost substitutes or supports, thereby lowering the loading of precious metals [28]. Carbon electrodes can be easily modified and functionalized to enhance their interaction with different transition metal oxides [29].

Interaction of carbon materials with transition metals

Different techniques can be used to deposit transition metals onto the carbon electrode. Some of these techniques, such as electro-deposition. In the electrodeposition process, metal ions move toward the electrode under an electric field and undergo electron-transfer-induced reduction to form a metallic layer or nanoparticles on the carbon surface [30]. Another technique that adopts chemical vapour deposition (CVD), is applied to deposit metal films on the surface of the carbon substrates. Physical vapor deposition (PVD) encompasses several techniques, such as sputtering, to deposit metal coatings on the surface [31].

Carbon materials coupled with other transition metal oxides in electrocatalysis provide a broad array of benefits driven by changes in electronic structure, synergetic effects, and the establishment of more robust metal-carbon bonds. Carbon electrodes are an ideal substrate for metal-based catalysts, as they offer very high conductivity and durability. The main reason for a significant increase in the use of transition metals as catalysts is their effectiveness in improving reaction efficiency. They allow an electron transfer through them and also faster kinetics [32]. These interactions between carbon materials and transition metals enhance the active sites and thus enhance catalytic properties. Such composites, comprising carbon support materials and transition metal oxides with high activity and increased durability at reasonable cost, are highly effective and attractive for various electrocatalytic applications.

Impact of electrode area on its electrocatalytic performance

Overview of the Randles-Ševčik equation

John Randles (English scientist) and Anton Ševčik (Czech engineer) formulated, in 1948 [33,34], a theoretical model to forecast the dependence of peak current in redox processes on the scan rate of the applied voltage by varying variables such as concentration, surface area, and number of electrons. This equation helps to predict how scan rate affects peak current observed during cyclic voltammetry experiments. The Randles-Ševčik equation can give a fairly good estimation of electroactive surface area under conditions where redox reaction is diffusion-controlled and experimental conditions (temperature, potential scanning rate range) are properly managed.

The relationship between peak current and mass transport in cyclic voltammetry was first derived independently by Randles and Ševčík. Considering the diffusion-controlled flux of electroactive species toward a planar electrode under linear potential sweep conditions, J. Randles derived an expression for the peak current given by Equation (1) [33]. In this derivation, the peak current (i_p) can be expressed in terms of bulk concentration of the analyte (C), radius of electrode (r), number of electrons transferred (n), charge transfer coefficient (α), diffusion coefficient (D), and Faraday constant (F), This formulation emphasizes that the present response is strongly dependent on the physical geometry of the electrode, the kinetics of the electron-transfer process (through n and α), and the mobility of electroactive species in solution through the diffusion coefficient (through $D^{1/2}$).

Randles expression for the peak current i_p is given by Equation (1):

$$i_p = \frac{1.24\pi F C}{0.0118^{1/2}\,10^3} r^2\ n^{2/3}\alpha^{1/2}D^{1/2} \quad (1)$$

where F = 96485.3 C mol^{-1}, C / mol mL^{-1}) and D / cm^2 s^{-1}. Other numerical factors arose from unit conversion and thermal constants incorporated during the derivation of the peak current expression at 298 K, ensuring dimensional consistency when concentration is expressed in mM.

In contrast, Ševčik has derived the equivalent Equation (2), for the height of an oscillographic polarogram due to current response of the mercury drop electrode under diffusion control, written with the notation of the formula [34]. The presence of the term $v^{1/2}$ demonstrates the underlying diffusion-law nature of the current, since an increase in scan rate reduces the time available for mass transport, therefore leading to larger peak currents.

$$i_p = \frac{0.361}{(RT)^{1/2}} F^{3/2} n^{3/2} A v^{1/2} D^{1/2} \quad (2)$$

where A / cm^2 is the surface area of the mercury drop during the time of the drop at the moment peak current occurs, v / V s^{-1} is the scan rate, R / J mol^{-1} K^{-1} is the gas constant and T / K is the temperature.

By combining equations (1) and (2), the generalized form of the Randles-Ševčík equation was derived as Equation (3):

$$i_p = 0.4463\left(\frac{F^3}{RT}\right)^{1/2} n^{2/3} A D^{1/2} C v^{1/2} \quad (3)$$

Equation (3) represents the generalized form of the Randles-Ševčík equation, which combines the derivations of Randles and Ševčík for expressing the relationship between peak current and the square root of the scan rate. Besides the scan rate, equation (3) includes other key electrochemical parameters, *i.e.* the number of electrons transferred, diffusion coefficient, analyte concentration, and electrode surface area. The coefficient 0.4463 arises from the mathematical treatment of the diffusion-layer growth during a potential sweep. The unified equation (3) ultimately demonstrates that peak current for a diffusion-controlled reversible process will be proportional to $v^{1/2}$, A and C.

At 25 °C, Equation (3) becomes equal to Equation (4):

$$i_p = 2.6910^5 n^{3/2} A D^{1/2} C v^{1/2} \quad (4)$$

Changes in the Randles-Ševčik equation's parameters can directly affect the results of surface area determination of an electrode. Because the equation relates peak current to surface area, changes in the diffusion coefficient, the number of electrons transported, or the amount of electroactive material can affect the predicted surface area. The electrode surface area often results in better electrochemical performance, such as by providing more active sites; larger surface areas generally lead to improved electrocatalytic performance because of the increased number of active sites available for reactions. If the electrode surface area is incorrectly estimated, it may affect the assessment of its

catalytic properties and electrochemical process performance. As a result, when using the Randles-Ševčik equation for surface area analysis, precise control of parameters is required to ensure reliable results. Accurate surface area measurements and reliable performance evaluation in electrochemical research are ensured by precise parameter calculations.

Review of recent studies comparing the performance of transition metal electrocatalysts with different electrode areas

Sections A to E are presented to summarize the principal electrochemical parameters like peak current, electroactive surface area, diffusion characteristics and detection limits, which have been reported for electrodes modified with iron oxide, zinc oxide, cobalt oxide, copper oxide and nickel oxide. The specific parameters presented in sections A to E are necessary because they directly control and reflect the interfacial electrochemical properties of TMO-modified electrodes. The selected supporting electrolyte and redox probe define the surface chemistry and charge-transfer environment to which scan rate studies reveal the kinetic regime of the process. Diffusion coefficients enable reliable use of the Randles-Ševčík equation to estimate the electroactive surface area, which is one of the most important descriptors of TMO performance. Given that the primary role of TMOs is played through the efficiency of electron transfer and the number of accessible active sites, the given parameters fully comply with the task of checking and comparing their electrochemical efficiency. All the entries include TMO-modified carbon electrodes to be consistent with the review's emphasis on surface-area modulation and electrocatalytic activity.

Such a comparison of the above-mentioned parameters across different studies will help in the identification of the oxide modifier exhibiting superior electrocatalytic performance and provide insights into how electrode composition and surface properties influence sensitivity, diffusion control, and overall analytical efficiency.

A. Iron oxide-based electrocatalysts

Iron is the most commonly employed transition metal because of its great theoretical specific capacity, low cost, natural abundance, and environmental friendliness. Iron oxides have the most promising potential among transition-metal oxides as electrode materials, owing to their exceptionally large theoretical-specific capacity [35,36]. The catalytic activity was enhanced by a higher surface area, which led to a higher rate of electron transfer [37,38]. Iron oxide has special properties that make it useful in a variety of applications. These properties include biocompatibility, low toxicity, super-magnetic behaviour and stability in physiological conditions [39,40]. These substantial surface areas indicate a strong potential for electrochemical applications that benefit from a high density of active sites. In contrast, Nor *et al.* [41] prepared a screen-printed carbon electrode (SPCE) modified by iron oxide nanoparticles functionalized by carboxylic acid (CA-IONPs), glucose oxidase enzyme (Gox) and Nafion film. Nafion/graphene oxide (GO)/CA-IONPs/SPCE was used in glucose sensing, which possess a high surface area of 0.80 cm^2. The diffusion coefficient reflects the ease with which electroactive species can move through the electrode's interface, thereby influencing the kinetics of electron-transfer processes. Higher diffusion coefficients tend to be often associated with more efficient electron transfer, which is vital for high-performance electrodes.

For example, the TRGO+α-Fe_2O_3 electrode displays a relatively high diffusion coefficient of electroactive species (7.6×10^{-6} cm^2 s^{-1}), indicating efficient electron transfer processes [42]. Conversely, electrodes such as the m-Fe_2O_3 ($K_3[Fe(CN)_6]$) show an extremely low diffusion coefficient of 1.63×10^{-15} cm^2 s^{-1} [43]. For example, TRGO + α-Fe_2O_3 composites have a very low electroactive area (0.016 cm^2) but relatively high diffusion coefficient (7.6×10^{-6} cm^2 s^{-1}) and decent sensing response,

while some Fe_2O_3-based electrodes with higher areas possess very low diffusion coefficients (*e.g*. m-Fe_2O_3 with $D \approx 1.63\times10^{-15}$ cm^2 s^{-1}) [42,43]. This low value indicates significant limitations in electron transfer, likely reducing the electrode's overall catalytic efficiency. The ability to detect Pb ions in aqueous solution was investigated using the prepared electrode. The smaller size of the Fe_2O_3 nanoparticles (NPs) ensured a higher surface area, promoting a faster electron transfer rate between the Pb ions and the electrode. Furthermore, the greater surface area enabled more active sites to be available, thereby increasing Pb ion detection. Increased surface area enhanced catalytic activity by facilitating a higher rate of electron transfer. Lekshmi *et al.* [44] selective electrochemical detection of catechol at carbon paste electrodes (CPEs) modified in the presence of NiO with α-Fe_2O_3 nanocrystals. Therefore, the expansion of the nanoelectrodes' surface area may be attributed to an increase in current intensity. Ranku *et al.* [45] designed a SPCE modified by sulphonated polyether ether ketone-iron (III) oxide composite, SPCE/Fe_3O_4/SPEEK, for dopamine detection. The increase in the intensity of the current could therefore be related to a rise in the nanoelectrode's surface area. The (SPCE/Fe_3O_4/SPEEK) modified nanocomposite electrode had a surface area of 2.79 cm^2, which was greater than the bare electrode's geometry. Sharma *et al.* [46] successfully encapsulated PB (Prussian blue) on Fe_3O_4 NPs, which possess immense potential for the realization of electrochemical biosensors based on a mechanism of direct electron transfer at the interface, owing to their higher electrocatalytic activity. The enhanced electroactive surface area as well as its surface concentration, is higher and aligns with increased redox currents upon immobilization of cholesterol particles. Gross *et al.* [47] reported a multilayered iron oxide-reduced graphene oxide, ION/reduced graphene oxide (RGO), nanocomposite electrode material for voltammetric bisphenol-A (BPA) sensing. Greater surface area of the ION-RGO composite and increased electronic mobility within the enhanced electrode can accelerate the electron transfer. Liu *et al.* [48] reported electrochemical detection of hydroquinone using single-walled carbon nanotubes, SWCNTs/Fe_2O_3 nanoparticles composite material. Weng *et al.* [49] synthesized magnetite-reduced graphene oxide (Fe_3O_4/rGO) modified glassy carbon electrode (GCE) to detect uric acid. The Fe_3O_4/rGO/GCE electrode used for uric acid detection also has a low diffusion coefficient of 4.57×10^{-13} cm^2 s^{-1}, which may necessitate further optimization to improve performance [49]. The enhanced electrocatalytic performance improves the rapid electron transfer, exhibiting high sensitivity and selectivity compared to a bare electrode. Shetti *et al.* [50] worked with Fe_2O_3 NPs-bentonite clay modified carbon paste electrodes. The composite electrode's surface area was 0.124 cm^2 and the surface area of bare CPE was observed to be 0.042 cm^2. Bentonite clay mixture and the nanoparticles of γ-Fe_2O_3 on the electrode's surface caused this increased surface area. An increase in current proved the electrocatalytic action of γ-Fe_2O_3 nanoparticles. Although most studies have reported a positive relationship between electroactive surface area and electrochemical performance, several conflicting results were also observed. These findings indicate that electrochemical kinetics are not fully defined by surface area alone. Furthermore, discrepancies among reported diffusion coefficient and surface area values, *e.g*. anomalously large diffusion coefficients in cholesterol/Fe_3O_4 sensors compared to very low values in other Fe_3O_4 composites, are probably due to differences in experimental conditions. These inconsistencies highlight the requirement of standardized reporting of data, especially for diffusion coefficients and electroactive surface-area calculations, to allow more valid cross-study comparisons.

Table 1 summarizes the literature data on different iron oxide-based electrodes where electroactive surface area and diffusion coefficient of the analyte were directly reported, and the Randles-Ševčík equation was used to relate electrode morphology to electrochemical behaviour [41-51].

Table 1. *Comparison of electroactive surface area and diffusion coefficient of iron oxide modified electrodes*

Modified electrode	Electrolyte/redox probe	v / mV s^{-1}	A / cm^2	D / cm^2 s^{-1}	Application	Ref.
Nafion/GOx/ CA-IONPs/SPCE	100 mM PBS	20	0.13	1.32× 10^{-9} and 4.43× 10^{-9}	Detection of glucose	[41]
TRGO+α-Fe_2O_3	0.1 M $K_3[Fe(CN)_6]$	50 to 150	0.008	7.6 × 10^{-6}	Supercapacitor	[42]
m-Fe_2O_3	$K_3[Fe(CN)_6]$	0.1	1.96	1.63 ×10^{-15}	Lithium-ion batteries	[43]
Lac-MCPE-Fe_2O_3	$K_3[Fe(CN)_6]$	50	0.089	6.67 × 10^{-6}	Catechol biosensing	[44]
SPCE-Fe_3O_4/SPEEK	5 mM $K_3[Fe(CN)_6]$	25	2.799	-	Detection of dopamine	[45]
ChOx/PB-Fe_3O_4/ITO	100 mM PBS	50	0.31	0.97 × 10^{-8}	Cholesterol estimation	[46]
ION-RGO/ITO	1 mmol L^{-1} $K_3Fe(CN)_6$	5 to 100	0.402	5.08 × 10^{-4}	bisphenol-A sensing	[47]
Fe_2O_3/CNTs/FTO	1 mM $K_3[Fe(CN)_6]$	100	7.16		Hydroquinone sensing	[48]
Fe_3O_4/RGO/GCE	$K_3[Fe(CN)_6]$	50	0.08	4.57×10^{-13}	Uric acid detection	[49]
Fe_2O_3-bent/CPE	0.1 mM $K_3Fe(CN)_6$	50	0.124		Antiviral drug acyclovir	[50]
GCE/MWCNT/α-Fe_2O_3	5 mM $K_3Fe(CN)_6$]	10 to 200	0.137	0.76 ×10^{-5}	Dye-sensitized solar cells (DSCs)	[51]

GOx: glucose oxidase enzyme; CA: carboxylic acid; IONPs or ION: iron oxide nanoparticles; TRGO: thermally reduced graphite oxide; m-Fe_2O_3: mixed phase Fe_2O_3; Lac-MCPE: laccase enzyme modified CPE, SPEEK: sulphonated poly ether ketone; ChOx; cholesterol oxidase enzyme: PB: Prussian blue; ITO: indium tin oxide; FTO: fluoride doped tin oxide; CNTs carbon nanotubes; Bent: bentonite clay

B. Nickel oxide-based electrocatalysts

NiO nanoparticles possess remarkable electrocatalytic properties, conductivity, biocompatibility, nontoxicity, and good chemical stability [52,53]. They expedite the transport process of the electrode between redox species and the electrode surface. Because of their enhanced surface and electrical properties, nickel oxides have exceptional electrocatalytic capabilities and are strongly associated with size-dependent traits, including large surface area and small particle size [54].

Nickel oxide with mixed valency state (NiO_x) on the GCE was employed for the electrocatalytic oxidation of urea with an area of 0.07 cm^2 at 100 mV s^{-1} [55]. To detect nicotinamide adenine dinucleotide (NADH), the carbon paste electrode (CPE) was modified with NiO nanoparticles [56]. NiONPs/modified carbon paste electrode (MCPE) used for NADH sensing has a surface area of 0.155 cm^2, which is a larger surface area that may contribute to a more efficient catalytic process, which is necessary for the sensitive detection of NADH. In contrast, the NADH sensor exhibits a significantly higher diffusion coefficient of 6.67×10^{-6} cm^2 s^{-1}, reflecting more rapid electron transfer kinetics for the redox system [56]. The excellent electrocatalytic activity of the modified CPE was demonstrated in the experimental analysis of uric acid and NADH, with a surface area of 0.01 cm^2, exhibiting a diffusion coefficient of 6.43×10^{-5} cm^2 s^{-1}, indicating higher stability, sensitivity, and selectivity. For paracetamol detection, the CPE was modified with NiO nanoparticles and an ionic liquid crystal (ILC), forming NiONPs/ILC/MCPE with a surface area of 0.0706 cm^2 (Atta *et al.* [57]). This relatively small surface area is indicative of an electrode optimized for specific, targeted detection, in which the number of electroactive sites is balanced to prevent excessive background noise while still allowing effective analyte interaction. Murugadoss *et al.* [58] explored GCE/NiO (0.106 cm^2), which has a larger surface area than bare GCE (0.06 cm^2), and the modified electrode's electroactive surface area has increased, which shows NiO to be a useful modifier as it offers a substantial surface that enables the efficient facilitation of electron transfer reaction. These findings clearly show the electrocatalytic potential of NiO in the oxidation process of paracetamol. In the detection of dopamine, in contrast, the tyrosinase/PB-NiO/ITO shows a larger surface area of 0.125 cm^2 and has a diffusion coefficient of 1.33×10^{-8} cm^2 s^{-1} [59]. The increasing surface area here is likely beneficial for neurotransmitter detection, where higher sensitivity and the ability to capture more analyte molecules are crucial. The lower diffusion coefficient implies slower but more regulated electron transfer, which is important for the precision required in neurotransmitter detection.

Danial *et al.* [60] demonstrated the electrocatalytic oxidation of glucose using a glassy carbon electrode, the presence of the Ni(II)/Ni(III) redox pair, and NiO_x as the key catalyst. Hence, tiny NiO_x on the GCE leads to a notable improvement of glucose electrocatalytic oxidation with an area 0.07 cm^2 at 100 mV s^{-1} [60]. ITO electrode modified by xantine oxidase (XOx) enzyme on nanostructured NiO, *i.e.* XOx/n-NiO/ITO modified electrode showed electroactive active surface area of 0.25 cm^2 at 0.05 V s^{-1} [61]. Khairy *et al*. [62] used a NiO-modified screen-printed electrode with a higher diffusion coefficient of 7.2×10^{-6} cm^2 s^{-1} and concluded that greater surface area and multi-directional accessibility, along with richer active sites, will be provided by multi-diffusional spaces/cavities, thereby improving electrocatalytic performance. The results clearly show that NiO modification improves electroactive surface area and electron-transfer rates across different biosensing platforms.

A comparison of different NiO-modified electrodes with regard to electrolyte systems, electro-active surface area, scan rates, diffusion coefficients, and applications is listed in Table 2 [55-63].

***Table 2**: Comparison of electroactive surface area and diffusion coefficient of nickel oxide modified electrodes*

Modified electrode	Electrolyte/redox probe used	A / cm^2	v / mV s^{-1}	D / cm^2 s^{-1}	Application	Ref.
GCE/NiO_x	0.5 M NaOH	0.07	100	1.6×10^{-6}	Urea oxidation	[55]
NiONPs/MCPE	Fe $(CN)_6$	0.155	10	6.67×10^{-6}	b-nicotinamide adenine dinucleotide (NADH) sensing	[56]
(NiONps/ILCMCPE)	1 mM acetaminophen (N-acetyl-p-aminophenol)	0.0706	100		Detection of paracetamol	[57]
GCE/NiO	1 mMPA in 0.1 M PBS	0.106	5		Determination of 4-acetaminophen in paracetamol	[58]
Tyrosinase/NiO/ITO	PBS (50 mM, pH 6.5, 0.9 % NaCl)	0.125	100	5.79×10^{-12}	Detection of dopamine	[59]
nano-NiO_x/GCE	0.3 M KOH	0.07	100	6.7×10^{-6}	Electrocatalytic oxidation of glucose	[60]
XOx/n-NiO/ITO	50mM PBS	0.25	50	1.33×10^{-8}	Freshness biosensor	[61]
NiO-SPE	$[Fe(CN)_6]^{4-/3-}$	0.0962	100	7.2×10^{-6}	Parathion pesticide sensing	[62]
GCE/MWCNT	0.1 M PBS	1.88	25		Determination of serotonin in urine samples	[63]

NiO NP/MCPE: Nickel oxide nanoparticle modified carbon paste electrode; ILCMCPE; ionic liquid crystal modified composite electrode; XOx: xanthine oxidase enzyme; ITO: indium tin oxide; SPE: screen printed electrode; MWCNT: multiwalled carbon nanotubes

C. Copper oxide-based electrocatalysts

Large surface areas and very small size CuO nanostructures result in better chemical and physical characteristics and they are environmentally friendly, non-toxic, low-cost, and possess high catalytic properties [64-67], having unique optical and electronic properties [68,69]. CuO nanoparticles having large surface area and numerous reaction sites in CuO/GCE exhibit excellent electrocatalytic activities [70-72]. Patil *et al.* [73] fabricated a modified carbon paste electrode using nanoparticles of graphene oxide and copper oxide, CuO-GO/CPE, which was applied to demonstrate electrochemical studies of theophylline (THP), one of the most important drugs in today's scenario. THP-containing drug and urine samples can be quantified by using the modified electrode. It has been demonstrated that CuO nanoparticle deposition on a glassy carbon electrode exhibits good sensitivity. CuO emerged as a potential material for accurate and efficient non-enzymatic detection of H_2O_2, primarily because of outstanding sensitivity, response speed, and linearity- a characteristic largely caused by massive surface areas and effective electron transport in related processes [74]. Mihailova *et al.* [75] prepared nanostructured copper oxide on copper wires to detect hydrogen peroxide. Petal-like CuO nanostructures give an enormously higher surface area of 6.5 cm^2 along with a greater number of active bonds and high-speed pathways due to the highly porous nature of the surface of analyte molecule transfer, featuring electron transfer and efficient mass diffusion processes, which are more efficient than the less developed film. Balaji *et al.* [76] investigated the electrochemical activity of CuO nanoflakes-reinforced GCEs for the sensing of caffeine. Due to their elevated aggregation and stacking levels, the

numerous CuO nanoflakes provide more reaction sites for catalytic reactions with higher specific activity. It is an appropriate form for electrocatalysis since it has a higher specific surface area. The CuO/NPs/MCPE method was effectively applied to determine the presence of bisphenol A in food samples. An electrode conductivity increases in the presence of CuO/NPs [77]. Employing a direct electrochemical approach for the estimation of bisphenol A, a very selective and sensitive chemical sensor made up of a graphene oxide-modified electrode of glassy carbon decorated with metallic copper oxides and zinc oxide was developed. The nanosheets in the GO structure increased the robustness and electrode surface area while producing additional active sites. Aparna *et al.* [78] developed a ternary nanocomposite material utilizing Au-Cu_2O/rGO proposed for the identification of uric acid and dopamine. The synergistic effect of conductive multi-walled carbon nanotubes (MWCNTs) and catalytically active $CuFe_2O_4$ improved the electrocatalytic performance of the modified carbon paste electrode MWCNTs/$CuFe_2O_4$/CPE. An electroactive surface area of 0.075 cm^2 and a high diffusion coefficient of 7.6×10^{-6} cm^2 s^{-1} indicated efficient electron transfer and mass transport. A wide scan-rate range (10 to 130 mV s^{-1}) showed good electrochemical stability. All these features allowed the sensitive determination of dacarbazine [79]. The CuO/MIPPY/GCE is a combination of copper oxide and a molecularly imprinted polymer (MIPPY) that is specifically designed for tyrosine recognition. The electroactive surface area of 0.070 cm^2 and the diffusion coefficient of 7.6×10^{-6} cm^2 s^{-1} reveal fast electron transfer rates in the presence of $K_3[Fe(CN)_6]$. High selectivity is achieved through molecular imprinting, and CuO enhances electrocatalytic performance [80]. The improvised electrode depends largely on the ternary nanocomposite material used for its electrocatalytic properties. The rGO's presence in the composite enhances the surface area that can be used for a reaction and fast electron transfer kinetics. It has been observed that the combination of Au and rGO with Cu_2O yields enhanced performance, as evidenced by Au-Cu_2O/rGO/ /GCE. It is recognized that the electrocatalyst's surface roughness often plays a significant role in catalytic reactions. Muthukutty *et al.* [81] fabricated SPCE to determine the electrocatalytic behaviour of copper oxide-tin oxide. The increased electrocatalytic activity in the detection of anti-psychotic drugs is due to an increase in specific surface area, which often impedes electron mobility at the electrolyte-electrode interface. Kumar *et al.* [82] reported modification of the carbon paste electrode by copper oxide (CuO) and GO nanocomposite (CuO/GO) for the determination of hazardous Di-hydroxybenzene isomers. Modifying the electrode increased the electrode's electroactive surface area by 0.055 cm^2 [82]. Sakthinathan *et al*. [83] described the electrocatalytic activity of the glassy carbon electrode (GCE) modified with the rGO/CuO composite for flutamide detection. This, due to its excellent electrocatalytic properties, presented a very selective and sensitive reaction [83]. Copper oxide was decorated on MWCNTs modified glassy carbon electrode for the measurement of isoniazid (INZ) [84]. There was a decline in resistance following an electrode change, indicating increased electron transport kinetics. Because of its electrochemical properties, the GCE/MWCNT/copper oxide with mixed valency state (CuO_x) electrode had a large electrode area and low charge-transfer resistance, making it a candidate for trace analysis. Daizy *et al.* [85] used ascorbic acid as a highly sensitive active recognition element on a glassy carbon-modified electrode to detect non-electroactive melamine. The synergistic effect of copper nanoflowers (CuNF) on reduced graphene oxide (rGO) films enables efficient, rapid electron transport between the GCE substrate and the modifying layer. At 0.255 cm^2, the electrochemical surface area of the enhanced electrode was larger than that of the bare electrode, indicating high electron-transfer kinetics. Karami *et al.* [86] fabricated a reduced graphene oxide-polydopamine-copper(I) oxide nanocomposite, RGO-polydopamine (PDA)-Cu_2O, applied to the GCE surface and observed an intensive rise in the active surface area compared to the GCE electrode. Cu_2O/MnO_2/GCE demonstrated a superior electrocatalytic response for the H_2O_2

oxidation [87]. Assaf *et al.* [88] fabricated a carbon paste electrode decorated with CuO nanoparticles to detect omeprazole. The comparison of results obtained for some CuO modified electrodes is presented in Table 3 [73-88].

Table 3. *Comparison of electroactive surface area and diffusion coefficient of copper oxide modified electrodes*

Electrode	Electrolyte/ redox probe used	A / cm^2	v / mV s^{-1}	D / cm^2 s^{-1}	Application	Ref.
CuO-GO/CPE	0.1 M KCl +1mM $K_3[Fe(CN)_6]$	0.096	50 to 500	-	Theophilline detection	[73]
CuO/GCE	1 mM $K_3Fe(CN)_6$, 0.1 M KCl	0.1447	100	7.6×10^{-6}	H_2O_2 detection	[74]
CuO/Cu	0.1 M NaOH	6.5	100	6.8×10^{-5}	H_2O_2 detection	[75]
CuO/GCE		0.103	-	6.7×10^{-6}	Caffeine sensing	[76]
Cu-Zn/GO/GCE	$K_4[Fe(CN)_6]$	0.282	50	6.7×10^{-6}	Determination of bisphenol A	[77]
Au-Cu_2O/rGO/GCE	K_4[]$Fe(CN)_6$	13.61	100		Dopamine and uric acid detection	[78]
MWCNTs/$CuFe_2O_4$/CPE	$K_3Fe(CN)_6$	0.075	10 to 130	7.6×10^{-6}	Determination of dacarbazine	[79]
CuO/MIPPY/GCE	$K_3Fe(CN)_6$	0.070	100	7.6×10^{-6}	Recognition of tyrosine	[80]
CuO/SnO_2 NPs/SPCE	$[Fe(CN)_6]^{3-/4-}$	0.128	50	7.6×10^{-6}	Detection of anti-psychotic drug	[81]
CuO-GO/CPE	0.3 mM $K_4[Fe(CN)_6]$	0.055	50	-	Determination of hazardous di-hydroxybenzene isomers	[82]
RGO/CuO/GCE	$K_3[Fe(CN)_6]^{3-/4-}$	0.182	50	--	Detection of flutamide drug	[83]
GCE/ MWCNTs/CuO	1 mM $K_4Fe(CN)_6$	1.14	50	6.7×10^{-6}	Determination of isoniazid drug	[84]
ErGO-CuNF/P-Arg/GCE	$[Fe(CN)_6]^{3-/4-}$	0.255	50	-	Detection of melamine	[85]
RGO-PDA-Cu_2O/GCE	$[Fe(CN)_6]^{-4/-3}$	0.011	50	0.76×10^{-5}	Detection of glucose	[86]
Cu_2O/MnO_2/GCE	$K_3Fe(CN)_6$	0.0012	50	1.65×10^{-5}	Hydrogen peroxide sensing	[87]
CuO NPs/CPE	$K_3Fe(CN)_6$	0.07	50	7.6×10^{-6}	Omeprazole determination	[88]

MIPPY: molecularly imprinted polypyrrole; ErGO: electrochemically reduced graphene oxide; P-Arg: polymerized arginine; CuNF: copper nanoflowers

D. Cobalt oxide-based electrocatalysts

The biocompatibility, higher specific area, eco-friendliness, chemical stability, high conductivity, electrocatalytic activity, cost-effectiveness, antifouling properties, and accessibility of cobalt oxide nanoparticles have attracted many researchers [89-91]. Modified electrodes with cobalt oxide nanostructures provided larger active sites for the reactions because they are porous, enabling diffusion and adsorption of electroactive compounds [92]. Electrodes modified with cobalt oxide have therefore proven useful in a variety of applications, including heterogeneous catalysis, magneto-resistive devices, sensors, fuel cells, biomaterials, supercapacitors, and Li-ion batteries. The modified carbon paste electrode sensor is versatile and can be used for electrochemical drug testing [93].

Usai *et al.* [94] examined the electrocatalytic behaviour of a glassy carbon-based sensor for 2,4-dichlorophenol (2,4 DCP) that was composite-modified with cobalt oxide (Co_3O_4 NPs) and graphene oxide nanosheets (GONS), with a surface area of 0.13 cm^2. The higher surface area and enhanced conductivity of cobalt oxide nanoparticles in combination with graphene oxide may contribute to improved electrocatalytic behaviour [94]. The GONS/Co_3O_4 NPs-modified GCE electrode catalyses the oxidation of 2,4-dichlorophenol at a less positive potential, thus indicating that the conjugated nanomaterial used exhibits enhanced catalytic activity. This superiority results from the cooperation between graphene oxide nanosheets and cobalt oxide nanoparticles. Strong catalytic characteristics are added by the Co_3O_4 NPs, while an exceptionally large surface area is provided by the GONS. A similar application is recorded in the determination of dopamine [95]. The Co_3O_4/GO-modified screen-printed carbon electrode has a large electroactive area of 0.709 cm^2. This can be attributed to the synergistic effect that results from the combination of Co_3O_4 and GO. The electrode also exhibits favourable electron conductivity when the $[Fe(CN)_6]^{3-/4-}$ redox couple is used. This makes it ideal for the determination of dopamine and uric acid [96]. The Ni-Co nanosheets on reduced graphene oxide demonstrate strong electrocatalytic performance in an alkaline electrolyte (0.1 M NaOH). In addition, with a relatively low surface area of 0.047 cm^2, a large diffusion coefficient of 4.33×10^{-4} cm^2 s^{-1} is achieved, showing that a faster electron transfer process takes place, making this electrode more

suitable for glucose biosensing applications [97]. The electroactive surface area of the Co_3O_4 nanorod-modified glassy carbon electrode is found to be 0.031 cm^2, with stable electrochemistry, indicating efficient electron-transfer kinetics [98]. The Au/cobalt oxide with mixed valency state (CoO_x)-modified glassy carbon electrode has the advantage of combining gold nanoparticles and cobalt oxide, which increases conductivity and catalytic properties. It performs well in detecting dopamine at an electrochemical platform [99]. Atta *et al.* [100] reported the use of cobalt oxide nanoparticles/ /graphene/ionic liquid crystal-modified carbon paste for narcotic drug detection have a surface area of 0.312 cm^2. The high surface area in this case is attributed to the intrinsic properties of carbon paste, which can be easily modified to increase porosity and surface roughness, thus providing a large surface area conducive to enhanced electrochemical activity. Mafuwe *et al.* [101] observed that adding polyaniline (PANI) or Co_3O_4 NPs to the probe containing metallophthalocyanine enhances the ability to transport electrons, increasing the area of the surface and increasing the modified electrode's electroactivity. Kovethan *et al.* [102] used Co_3O_4 modified GCE for thioridazine detection and exhibited a significantly larger surface area of 0.320 cm^2. This substantial increase compared to other electrodes suggests that the Co_3O_4 modification alone, without the addition of other materials such as graphene oxide, can substantially enhance surface area, likely due to the highly porous or nanostructured coating formed on the electrode surface. Manjula *et al.* [103] reported a Zn-Co_2O_4-modified electrode for ofloxacin detection; the area was 0.1524 cm^2, slightly larger than the NrGO-Co_3O_4 GCE. This suggests that Zn-Co_2O_4 forms a highly porous structure, providing a larger surface area for electrochemical interactions, most likely because of the synergistic effects of Zn with Co_2O_4 in creating more active and accessible sites. This increase indicates that Co_3O_4 nanoparticles create additional surface roughness and active sites, enhancing the overall surface area available for electron transfer.

A cobalt oxide modified carbon paste electrode was used to determine flufenamic acid, with double the surface area of 0.07cm^2 compared to bare (0.04 cm^2) and showed an excellent sensitivity [104]. Table 4 represents the features of cobalt oxide modified carbon electrodes [94-104].

Table 4. *Comparison of electroactive surface area and diffusion coefficient of cobalt oxide modified electrodes*

Modified electrode	Electrolyte/redox probe used	A / cm^2	v / mV s^{-1}	D / cm^2 s^{-1}	Application	Ref.
GONS/Co_3O_4NPs/GCE	0.1 M KCl	0.13	100	7.6×10^{-6}	Sensing of 2,4 dichlorophenol	[94]
NrGO-Co_3O_4/GCE	0.1 M KCl	0.139	50	7.60×10^{-6}	Determination of dopamine	[95]
Co_3O_4/GO/SPCE	$[Fe(CN)_6]^{3-/4-}$	0.709	50	--	Dopamine and uric acid determination	[96]
Ni-Co NSs/RGO/GCE	0.1M NaOH	0.047	50	4.33×10^{-4}	Glucose biosensing	[97]
Co_3O_4NR-GCE	$K_3[Fe(CN)_6]$	0.031	5	6.7×10^{-6}	Hydroquinone and catechol detection	[98]
Au/CoO_x/GCE	2.5 mM $K_3Fe(CN)_6$	0.13	75	6.7×10^{-6}	Detection of dopamine	[99]
CoGILCCP-SDS	0.1 mol L^{-1} PBS	0.312	50	--	Determination of a narcotic drug	[100]
CoTCPc-PANI-Co_3O_4/GCE	$[Fe(CN)_6]^{3-/4}$	0.071	100	7.6×10^{-6}	Electrocatalytic oxidation of amitrole	[101]
$MgCo_2O_4$/GCE	$[Fe(CN)_6]^{3-/4-}$	0.320	50	--	Determination of thioridazine	[102]
Zn-Co_2O_4 /GCE	$Fe(CN)_6^{3-/4-}$	0.1524	50	--	Determination of ofloxacin in urine samples	[103]
Co_3O_4/CPE	$Fe(CN)_6^{3-/4-}$	0.07	100	-	Determination of flufenamic acid	[104]

GONS: graphene oxide nanosheets; NrGO: nitrogen doped reduced graphene oxide; NSs: nanostructures; NR: nanorods; GILCCP: graphene-ionic liquid modified carbon paste electrode; SDS: sodium dodecyl sulphate; CoTCPc: cobalt phtalocyanine; PANI: polyaniline; CG: carbon graphite; Au/CoO_x: gold-supported cobalt oxide

E. Zinc oxide-based electrocatalysts

ZnO NPs are materials that possess superior electrocatalytic activity against biomolecules, higher electron transfer kinetics, less toxicity, and strong electrical conductivity [105]. ZnO NPs' strong electrochemical properties make them promising candidates for antiviral drug sensing [106]. ZnO's

broadband gap energy of 3.2 eV, chemical and thermal durability, strong exciton binding strength of 60 meV, green compatibility, strong bond strength, and varying morphological qualities render ZnO a material of interest [107]. Sawkar *et al*. [108] tested the nanocomposite electrode stability and presented results indicating excellent stability and high efficiency for determining cetirizine. Hatamie *et al.* [109] built a voltammetric sensor for amoxicillin determination using zinc oxide nanorods (NRs). High-surface-area conducting ZnO NRs currently applied exhibit better catalytic performance, thereby enhancing their activity for the detection of amoxicillin (AMX). Hanabaratti *et al.* [110] reported that the significant increase in surface area of the modifier enhanced the conductivity, thereby allowing simpler electro-catalytic oxidation of paracetamol. The modified glassy carbon electrode with the ZnO nanoparticles has an electroactive surface area of 0.021 cm^2. It is through this arrangement that caffeine can be detected. The use of ZnO nanoparticles increases the number of catalytic sites, thereby improving electron transfer [111]. The combined effect of GO and a 3D ZnO structure enhances the surface area that is electroactive (0.076 cm^2). The conductivity provided by GO facilitates transport, and the 3D structure of ZnO enhances the availability of active sites [112]. The Cu-Zn/GO-modified GCE has a relatively large electroactive surface area of 0.282 cm^2 with a favourable diffusion coefficient of 6.7×10^{-6} cm^2 s^{-1} [113]. Bukkitgar *et al.* [114] treated the electrode with barium-doped ZnO nanoparticles (BDZnONPs), which exhibited notable changes in nimesulide behaviour. Sensitivity and selectivity were greatly enhanced due to the electrode's electroactive surface area increasing significantly. Table 5 includes the relevant data for zinc oxide modified electrodes discussed in this section [108-114].

Table 5. *Comparison of electroactive surface area and diffusion coefficient of zinc oxide modified electrode*

Modified electrode	Electrolyte/redox probe used	v / mV s^{-1}	A / cm^2	D / cm^2 s^{-1}	Application	Ref.
ZnO-Gr/CPE	$K_3[Fe(CN)_6]$ and KCl	50	0.095cm^2	7.6×10^{-6}	Determination of Cetirizine	[108]
ZnO NRs/gold/glass electrode	$K_3Fe(CN)_6$ in KCl	80	0.015	7.6×10^{-6}	Determination of Amoxicillin	[109]
ZnONPs/GCE	$K_3Fe(CN)_6$	50	0.201	7.6×10^{-6}	Determination of paracetamol	[110]
ZnONPs/GCE	$K_3Fe(CN)_6$	50 to 400	0.021	--	Detection of Caffeine	[111]
GO/3D-ZnO	$[Fe(CN)_6]^{3-/4-}$	20-200	0.076	--	Antibiotic drug chloramphenicol detection	[112]
rGO-ZnO-GCE	KCl & $[Fe(CN)_6]^{3-/4-}$	50	0.0493		Detection of tetracycline	[113]
BDZONPs/GCE	KCl & $[Fe(CN)_6]^{3-/4-}$	50	0.165	7.6×10^{-6}	Electro-oxidation of nimesulide	[114]

NRs: nanorods; 3D: three dimensional; rGO: reduced graphene oxide; BDZO: barium-doped zinc oxide

Future directions

Subsequent research in electrochemical sensing could benefit from standardised calibration procedures to improve the comparability and reproducibility of data across different laboratories. Detailed composite design, the optimisation of modifier type, morphology, and loading, is essential for achieving maximum sensitivity and stability. Synthesis of experimental efforts with density functional theory simulations provides a useful understanding of electrode-analyte interactions, allowing rational design of sensing platforms. Lastly, ongoing reporting of critical parameters like electroactive surface area, diffusion coefficients, and Randles-Ševčík analysis will enhance the dependability of comparative evaluations and progress the field toward normalized evaluation tools.

Conclusion

Transition metals are essential in electrocatalysis, particularly at carbon electrodes. Their properties make transition metals excellent electrocatalysts in a wide range of electrochemical transformations. Transition metal oxides on carbon electrodes are crucial in many electrochemical

processes, including energy conversion and storage, environmental remediation, and chemical synthesis. Their properties enable improved catalytic performance, and they play an essential role in modern electrochemical technologies. Further research in this area continues to address issues of stability, efficiency, and cost, opening the way to more efficient and sustainable electrocatalytic systems. The challenge is certainly the stability of the transition metal catalysts, which could degrade or even dissolve under very harsh conditions. Their development and optimized electrode condition received a lot of attention. Active research will also continue to focus on the resolution of problems in the realm of stability, efficiency, and cost, thus bringing an electrocatalytic system that is efficient and sustainable. Combining transition-metal electrocatalysts with carbon-based interfaces and the strategic expansion of electrode surface area are important features that improve the performance of electrochemical systems. In this instance, more active sites for electrochemical processes have increased, simultaneously a significant increase in catalytic activity and efficiency. Moreover, the carbon-based material, with good electrical conductivity and structural stability, when married to transition metals for their catalytic ability, provides a stable support network that promotes electron transfer through the synergy between transition metal oxides and carbon interfaces, thus stabilizing the system during extended operation. In addition, the electrode area will be maximized to ensure greater interaction between the catalytic surface and the electrolyte, thereby improving the reaction rate and the system's efficiency. These factors - better surface area, better conductivity, and specific characteristics for transition metal oxides - culminate in more efficient, stable, and selective catalytic performance. This integrated approach needs to be the way forward for expanding the boundaries of electrochemical applications, particularly in energy storage with conversion, fuel cells, and advanced catalytic technologies. Next-generation electrocatalysts that integrate high-surface-area electrodes and hybrid catalyst structures are thus overdue.

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