Nickel-Cobalt Chalcogenide Catalysts for Reducing the Overpotential of Oxygen Evolution Reaction: A Mini Review

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The production of green hydrogen can be sustainably achieved through electrolysis using energy from renewable sources. However, the high overpotential of the oxygen evolution reaction (OER) necessitates the development of a cost-effective, active, and stable electrocatalyst. This review explores the synthesis of nickel-cobalt chalcogenide catalysts and their performance as an accessible, inexpensive catalyst for OER, along with possible applications and modifications to improve its electrocatalytic activity. Transition metal chalcogenides have the property to exist in different oxidation states, which provides the basis for its electrocatalytic activity. The catalyst morphology, structure, composition, and interface are discussed to shed light on the observed electrocatalytic activity toward OER. The review also explores the advantages of various carbon-based substrates and their impact on the electrical conductivity and stability of nickel-cobalt chalcogenides. The practical applications in the energy industry can be realized by addressing various challenges, more importantly, the catalyst degradation and high overpotential in the anode.

1. Introduction

Renewable energy sources contribute a fraction of the total energy consumption primarily due to the decarbonization of the power sector. However, the lack of storage capacity and intermittency of solar and wind-based energy pushes for more versatile storage technologies in vital sectors such as industry and transportation. Hydrogen fuel is a renewable energy source that produces only water as a by-product of its combustion and can be used to create carbon-neutral synthetic fuels for the transportation sector or newer generation fuel cell vehicles. Electrolysis of pure water into its oxygen and hydrogen components is one of the processes used to produce renewable hydrogen for electricity generation. However, a common problem with these systems is the slow kinetics of the oxygen evolution reaction (OER), owing to the complexity of the reactions involving multiple intermediates and adsorbed species. As a result, the charge transfer between the electrolyte and anode is associated with high overpotential and resistance.

Electrocatalysts can reduce the high OER overpotential by lowering the resistance to charge transfer, allowing faster reaction rates. Noble metal catalysts such as ruthenium and iridium oxides are among the preferred catalysts for the oxygen evolution reaction due to their ideal Gibbs potential for water oxidation and are considered state-of-the-art electrocatalysts. However, these are expensive and not readily available. Using more cost-effective transition metal chalcogenides (TMCs) such as nickel-cobalt (Ni-Co) selenide catalysts, which exhibit strong electrocatalytic abilities, has been explored in various studies. Therefore, it is essential to review the properties and potential of Ni-Co chalcogenide-based electrocatalysts in reducing the overpotential of the OER to determine its use for hydrogen production.

This review aims to provide insights and direction for future research on improving nickel-cobalt chalcogenide electrocatalysts. The literature reviewed in this study was amalgamated and differentiated to formulate the review method, which is explained further in each section, contributing to a novel approach to understanding and improving OER catalysts. Selected works, especially those that were recently published, were chosen as a basis for the review method because of their relevance to current research. The succeeding section provides a

brief overview of electrocatalysts, including their role and types, and offers an analysis of existing elemental and compound catalysts used in the OER.

2. Electrocatalysts in OER

The high overpotential associated with the OER is attributed to its sluggish reaction kinetics. To improve OER rates, catalysts are added to the system. Various catalysts such as Pt, Ni, IrO_x, and RuO_2 have been examined to enhance charge-transfer kinetics, improve stability, and explore the mechanistic behavior of the intermediates and adsorbed species. Pt-based electrocatalysts tend to suffer from unwanted deactivation in OER, resulting in low electrocatalytic activity and inefficient reaction rates (Reier et al., 2012). In contrast, IrO_x compounds have been reported as superior and active electrocatalysts for OER due to their performance and stability (Geiger et al., 2016). In particular, IrO_2, in its hydrous and amorphous form, exhibits high OER activity compared to its crystalline and thermally prepared counterpart (Zhao et al., 2021). However, due to its scarcity, it is impractical and expensive to use IrO_x for industrial-scale applications and scenarios.

TMCs, including iron, nickel, and cobalt, are low-cost alternatives to precious metal catalysts such as Pt, IrO_2, and RuO_2. However, the initial performance of these TMCs and their oxides is inferior to that of the noble metal electrocatalysts. To improve the activity, mixed metal chalcogenides have been investigated and are theorized to possess enhanced activity due to synergetic effects between the mixed metals (Deng et al., 2017). For instance, in its oxide form, nickel is an inferior OER catalyst and is often doped with Fe, resulting in $NiFeO_x$ compounds (Ali Akbari et al., 2020). Cobalt is also combined with other structures to minimize corrosion, allowing it to sustain electrocatalysis for longer periods (Millet et al., 2014). Combining these transition metals with nonmetals and metalloids further enhances the catalytic activity of these transition metals (Zhou et al., 2021). Chalcogenides, combined with metallic catalysts, have been found to promote OER catalysis at active sites (Anantharaj and Noda, 2020). However, among chalcogenides, selenides have been found to exhibit superior performance compared to oxides and sulfides (Yuan et al., 2022). This is mainly attributed to higher activity levels and greater capacity for surface reconstruction, which provides better access to active sites (Anantharaj and Noda, 2020). The following section reviews the importance of configuring the microstructures of the electrocatalyst and analyzes studies geared toward catalyst doping and integration.

3. Interface engineering

Electrocatalyst performance is strongly dependent on its ability to perform electron transfer. This phenomenon is related to multiple interface structures involving the electrocatalyst. Interface engineering can enhance the performance of the electrocatalyst in water-splitting reactions by adjusting the structure of the interface (Qiao et al., 2022). An efficient strategy here to improve performance is to hybridize an electrocatalyst with another metal-based catalyst and create rich vacancies within the structure to regulate charge transfer (Yang et al., 2023).

Suo et al. (2021) synthesized a heterostructure involving cobalt-nickel sulfides and phosphides doped with vanadium to improve conductivity and increase the structure's surface area. The OER performance of the synthesized V-doped electrocatalyst has an overpotential of 210 mV, which is smaller than the 273 mV overpotential of the same electrocatalyst structure without V-doping. The study of Rajesh et al. (2021) involved the synthesis of a Ni-Co selenide catalyst through the electrodeposition of nanostructures on Ni foam. It is reported that the Ni-Co selenide electrocatalyst required a low overpotential of 244 mV with a Tafel slope of 68 $mV\text{-}dec^{-1}$. The synergistic effect between the two transition metals (Ni and Co) is a vital attribute of the performance of the bimetallic electrocatalyst, which is excellent for both water-splitting reactions.

Through these studies, the electrocatalytic performance of Ni-Co chalcogenides can be improved by interface engineering by integrating other metal-based catalysts and increasing the vacancies in the structure via doping. The next section focuses on the identities of electrocatalyst compositions, ranging from binary to multinary combinations in varying ratios. Existing research concerning the synthesis of different compounds is reviewed and analyzed further to compare the performance of each catalyst.

4. Compositional control

Modifying and controlling TMC structures and compositions are essential and promising for electrocatalyst performance. Most TMCs encompass the selenides and tellurides of Ni, Co, Fe, and Cu. With optimal synthesis and comprehensive testing and characterization, TMCs will display abundant active sites, electron-rich transition metal centers, and a highly covalent network, owing to their charge transfer ability.

The study of Nath et al. (2022) discussed the effects, performance, and characteristics of binary and multinary (ternary and more complex) TMCs in the electrocatalytic water splitting context. Binary TMCs contain one transition metal and one chalcogenide, such as nickel selenides (Ni_xSe_y), cobalt tellurides (Co_xTe_y), and nickel sulfides (Ni_xS_y). Different stoichiometric and non-stoichiometric ratios of nickel selenides were observed and

investigated for OER electrocatalytic performance against each other. Nickel telluride (Ni_3Te_2) was tested for OER activity and compared with Ni_3E_2 (E = S, Se) and NiO_x counterparts. The compound was found to have an OER overpotential of 180 mV at 10 mA cm^{-2}, one of the lowest among binary TMCs. This result confirmed the claim in the study of De Silva et al. (2018) that the decrease in electronegativity of chalcogenide ions [Te (2.1), Se (2.55), O (3.44)] and their corresponding degree of covalency led to a heightened catalyst efficiency. Xu et al. (2017) demonstrated, by characterization, that Ni_3Se_2 performed best in OER activity because of the synergetic effect of intrinsic metallic states and superior anion surface reorganization in the metal matrix compared to NiO, NiSe, and Ni. Other compounds with poor conductivity were found to be exceptionally inferior to Ni_3Se_2 (Xu et al., 2017). The electronegativity effect in improving OER catalytic activity, as evidenced in Ni-based chalcogenides, also applies to Co-based compounds. $CoTe_2$ demonstrated highly improved catalytic efficiency compared to Co_xO_y and Co_xSe_y. Additionally, the hydroxyl adsorption energy, a fundamental characteristic of OER catalytic activity, was optimal and prominent in selenides and tellurides (Nath et al., 2022). Mixed-transition-metal-based electrocatalysts or multinary TMCs were developed as a result of tuning changes in the electron density around the catalytic center through d–d transitions by doping the transition metal site with another transition metal. These compounds demonstrate better electrical conductivity and structural stability, outperforming their monometallic counterparts in catalytic activity. $NiCoSe_2$ anchored on a macroporous carbon membrane (PCM) displayed exceptional catalytic activity with an overpotential of 140 mV. $Ni_{0.75}Fe_{0.25}Se_2$ anchored on nickel foam (NF) also showed fair OER activity with a 210 mV overpotential.

Combinatorial electrodeposition helped examine quaternary mixed metal selenide compositions such as Ni-Fe-Co, Fe-Cu-Co, and Co-Ni-Cu. Experiments were done in the studies of Cao et al. (2019a, 2019b, 2018), where $(Ni_{0.25}Fe_{0.68}Co_{0.07})_3Se_4$, $(Co_{0.21}Ni_{0.25}Cu_{0.54})_3Se_2$, and $(Fe_{0.48}Co_{0.38}Cu_{0.14})$ Se were synthesized in each separate and respective study, which sought to identify the optimal quaternary TMC catalyst. Those compounds had overpotentials of 230 mV, 272 mV, and 256 mV. The studies proved that transition metal doping increased the number of catalyst active sites and accelerated the rate-determining steps through hydroxyl-adsorption kinetics manipulation. Nath et al. (2022) also corroborated specific trends after analyzing the overpotentials of several binary and multinary TMCs. Decreasing overpotential can be attributed to decreasing chalcogenide electronegativity, while doping with transition metals containing fewer d-electrons to create multinary chalcogenides improves OER activity, considering aliovalent substitution. The optimization of Ni-Co chalcogenides can be further explored and experimentally observed by incorporating less electronegative chalcogenide anions with aliovalent substitution in the active site of the catalyst.

Table 1: Tabulated summary of review highlights

Highlights	Reference
Explored the performance of different TMCs and affirmed that using less electronegative chalcogenides and doping with transition metals containing fewer d-electrons enhance OER activity.	Nath et al. (2022)
Catalyst efficiency is improved when the degree of covalency around the transition metal area increases because of decreased chalcogenide electronegativity.	De Silva et al. (2018)
Ni_3Se_2 displayed superior OER catalytic activity when compared to other Ni-based compounds and less conductive materials.	Xu et al. (2017)
Quaternary mixed-metal selenide compounds of non-stoichiometric ratios were synthesized in each study and found to be the most optimal for each multi-metal complex.	Cao et al. (2019a, 2019b, 2018)

Following this section is the discussion of catalyst geometrical structure and size, comparing different particle morphologies synthesized in different studies.

5. Particle morphology

The shape and size of particles are vital in improving electrocatalytic performance. As electrocatalysis is a surface phenomenon, controlling the morphology of an electrocatalytic material is essential to ensure the proper distribution of active sites. The study of Peuckert et al. (1986) found that activity in Pt catalysts increased up until a certain point, while the study of Bett et al. (1973) found that the particle size had no significant effect on its activity. The contradictory observations could be attributed to differences in measurement, particularly in the magnitude of observed effects and in its experimental conditions. In more recent studies, an increase in nanoparticle size correlates to a higher catalytic rate per surface site in Au particles and vice versa (Besenbacher et al., 1998). Overall, larger particles within a small range generally result in better activity, although the ideal particle size may vary depending on the particle shape and surface morphology of the catalyst.

Regarding particle shape, hollow spherical structures are highly interested in the synthesis of electrocatalysts due to their high surface area, low density, and notable permeability (Chattopadhyay et al., 2016). This factor is further shown in the study of Lan et al. (2019). Spherical $CoSe_2$ particles have been found to reduce the overpotential of the OER the most compared to wire and rod-shaped particles of the same compound (Table 2).

Table 2: Overpotential and Tafel slope comparison of different morphologies.

Material	Morphology	Overpotential at 10 mA cm^{-2} (mV)	Tafel Slope (mV-dec^{-1})	Reference
$CoSe_2$	Spheres	325	80	Lan et al. (2019)
	Wires	340	85	
	Rods	366	88	
IrO_2	n/a	310	69	
Ni_xCo_yO	Spheres	336	36	Deng et al. (2017)
CoNiSe/NC-3	Spheres	270	66.5	Jiao et al. (2019)
$Ni_{0.25}Co_{0.75}Se$	Ni foam	269	74	Liu et al. (2019)
RuO_2	n/a	310	94	

It was also found that Ni, with a similar structure, was found to have a Tafel slope comparable to that of the structured $CoSe_2$ electrocatalysts in Table 2. This correlates to high electrocatalytic performance in reducing the overpotential of the OER. Some electrocatalysts also benefit from different mechanisms that form highly-faceted particles with edges that contain a significant number of active sites (Wang et al., 2015).

However, such structures have yet to be explored extensively for Ni-Co chalcogenides. Deng et al. (2017) explored the use of a near-spherical mesoporous $Ni_xCo_yO_z$ electrocatalyst, which resulted in a low overpotential of 336 mV comparable to that of noble metal electrocatalysts (Table 2). Ni foam-supported $Ni_{0.25}Co_{0.75}Se$ catalysts at 10 mA×cm^{-2} was also found to be comparable with the previously discussed spherical electrocatalysts (Liu et al., 2019). CoNiSe/NC-3 spheres containing nanosheets were observed by Jiao et al. (2019) to produce a low overpotential of 270 mV. Another study by Poorahong et al. (2021) made use of nanosheets, which obtained an OER potential of only 1.37 V. With comparable performance to noble metal electrocatalysts, these studies leave much potential for particle structure improvements in Ni-Co chalcogenides. In the penultimate section of this paper, the studies introducing support structures that influence and modify the architectural design of the catalyst and their application mechanisms are reviewed.

6. Architectural design

Surface area also plays a key role in determining the number of active sites for evolution reaction. A catalyst is desired to have a porous structure with multiple edges to enhance the number of active sites. In addition, porous support disperses aggregated catalytic particles and facilitates easier charge transfer during electrochemical reactions (Chu et al., 2019). This introduces the capability of catalytic supports that provide improved morphology, leading to overpotential reduction.

Despite presenting similar electrocatalytic activity as noble metal catalysts, TMCs suffer from structural defects that hinder effectivity. This includes low stability, the tendency for aggregation, and minimal conductivity. In particular, the tendency to aggregate decreases the surface area of the active sites and renders parts of the TMC inert (Stelmachowski et al., 2021). Electrocatalytic support structures that can expand the number of active sites to improve OER kinetics may be explored (Panganoron et al., 2020).

Catalyst supports are merged with electrocatalysts through various application mechanisms, including solvothermal synthesis and electrospinning. These allow better distribution of catalysts across the surface alongside more active sites for oxygen evolution reactions. It also produces an interface between the catalyst, water, and oxygen that improves electrocatalytic activity. SnO_2 and TiO_2-based compounds are extensively used alongside porous nickel supports that have resulted in a lower OER system overpotential (Wang et al., 2019). These supports can be expensive or difficult to synthesize. Hence, a need for more cost-effective alternatives. Carbon-based supports are promising and well-used in improving oxygen evolution reactions. Carbon nanotubes and composites, in particular, exhibit high conductivity, porosity, and flexible properties that improve the electrocatalytic properties of OER at a lower cost than traditional supports (Zoller et al., 2021).

In nickel cobalt selenide catalysts, electrocatalytic supports have been utilized to improve their architectural design and stability for greater activity. Liu et al. (2019) synthesized $Co_{0.75}Ni_{0.25}Se$ electrocatalysts on nickel foam through solvothermal selenisation. This process resulted in improved charge transfer from the interconnected nanorods. Its addition also resulted in a surface area 6.2 times greater than that of Co-based/NF, which may lead to an increase in active sites. Compared with other commercial electrocatalysts, the $Co_{0.75}Ni_{0.25}Se$/NF combination exhibited the lowest overpotential of 269 mV even against a more traditional RuO_2/NF (310 mV) and a Tafel slope of 74 mV-dec^{-1}. A possible cause of the low overpotential of the former

compound could be the superior compatibility between the Ni and Co ions in the structure. $NiCoSe_2$ has also been developed on a porous carbon membrane through solvothermal selenisation. This synthesis resulted in a sample with many pores capable of hosting OER. Compared with $NiSe_2$ and $CoSe_2$ catalysts synthesized with PCM, the $NiCoSe_2$/PCM electrocatalyst exhibited the best OER and HER potential that facilitated fast mass and charge transfer (Poorahong et al., 2021). The ternary compound containing included Ni atoms helped decrease the free energy of hydrogen adsorption in Se sites for improved kinetics (Jiao et al., 2019). The method in which the TMC is combined with electrocatalytic support may determine the level of activity and efficiency for OER. Jiao et al. (2019) also found that the mere electrodeposition of a TMC catalyst on the surface of a carbon skeleton may lead to aggregation that affects its effectivity. The encapsulation of $NiCoSe_2$ within the carbon skeleton through selenisation resulted in less aggregation and a lower overpotential at 270 mV.

7. Conclusions

Water electrolysis is a vital process considered in the production of pure hydrogen as a fuel source. However, this reaction is limited by the slow kinetics of the OER, which is associated with its high overpotential. Electrocatalysts can reduce this overpotential, although the noble and precious metals found to be highly effective are expensive and scarce. Low-cost substitutes with similar electrocatalytic performance are of high interest in cost-efficient water electrolysis. Transition metals have been found to exhibit comparable albeit inferior electrocatalytic performance to noble metal electrocatalysts. Transition metals are mixed with other compounds to enhance electrocatalytic activity.

Such mixtures include Ni-Co chalcogenides, which are low-cost transition metal electrocatalysts possessing characteristics that facilitate the effective reduction of OER overpotential. Their activity is further improved by modifying the structure and surface morphology of the electrocatalyst. Cheaper electrocatalytic structures derived from carbon structures have been found to improve the number of active sites, conductivity, and mechanical properties of TMCs. However, many of these factors are yet to be explored specifically for Ni-Co chalcogenides. Future studies may consider exploring improvements in the interface, composition, particle morphology, and structure of Ni-Co chalcogenides to enhance its electrocatalytic performance and to improve the understanding of the mechanisms that allow it to catalyze the OER.

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