

Progress on the Preparation of Cobalt-Iron Based Electrocatalysts and Their Catalytic Performance in HER and OER

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Abstract: This review provides an in-depth discussion on the recent advances of iron-cobalt-based electrocatalysts in the field of hydrogen production by water electrolysis, focusing on their catalytic roles in hydrogen-extraction reaction (HER) and oxygen-extraction reaction (OER). In the face of resource scarcity and cost issues of conventional noble metal catalysts, this paper emphasizes the urgency of developing efficient and economical OER catalysts and outlines the impact of preparation methods on catalyst structure and activity. The review also discusses strategies to enhance the performance and stability of the catalysts and assesses the advantages and challenges of these catalysts for practical energy applications. The paper concludes with an outlook on the development of iron-cobalt-based electrocatalysts in the clean energy sector, suggesting possible directions and challenges for future research, with the aim of providing guidance and inspiration for research on electrocatalysts for hydrogen production via water electrolysis.

Keywords: Iron-Cobalt-Based Electrocatalysts; Water Electrolysis; Hydrogen Production; Clean Energy; Catalytic Performance

1. Introduction

Nowadays, the global demand for sustainable energy is on the rise and hydrolysis is a key technology due to its clean, renewable and efficient nature in hydrogen production. Hydrogen is destined to occupy a central position in the future energy mix due to its high energy density and its natural advantages of being zero-emission and renewable. The core of this technology lies in the catalysts on the electrodes, which facilitate the hydrogen development reaction (HER) and the oxygen development reaction (OER).

This paper provides an insight into iron-cobalt

based catalysts, which have received a lot of attention due to their potential to replace traditional and more costly precious metal catalysts. These transition metal catalysts are favored for their abundance, low cost, and chemical stability, making them strong candidates for large-scale hydrogen production via hydrolysis.

The introduction of this article sets the stage for a comprehensive review of recent advances in the synthesis and characterization of iron and cobalt-based catalysts. The article emphasizes the urgency of developing efficient and cost-effective OER catalysts to overcome the limitations of conventional catalysts due to the complexity and high cost of the OER mechanism.[9]

The review will take a long look at the field of electrocatalysis, reviewing synthetic methods based on iron oxides, cobalt hydroxides and evaluating their performance in HER and OER. It will also explore the effect of different preparation methods on the structure and activity of the catalysts and discuss strategies aimed at optimizing catalytic performance and stability.

By combing the current state of research and future prospects of these catalysts, this paper aims to provide a valuable reference for the scientific community and to promote further development in the field of electrocatalysis. Through this review, we hope to contribute to the promotion of clean energy technologies and facilitate the transition to a sustainable energy future.

2. Common Preparation Methods

2.1 Template Method

2.1.1 Brief introduction

The template method offers a simple synthesis process, and the synthesized catalyst can precisely control the micro shape and size, nature and structure of the catalyst to meet the required performance requirements. Wei et al.

used spinel-type iron cobalt oxides and nitrogen-doped mesoporous carbon materials to prepare a kind of electrocatalysts for metal-air batteries, and the template SBA-15 was prepared by using TEOS and hydrochloric acid, and then 1,10 phenanthroline hydrate, ferric acetate, cobalt acetate tetrahydrate and ethanol were added into the dispersion of SBA-15, and then 1,10 phenanthroline hydrate, ferric acetate, cobalt acetate tetrahydrate and ethanol were added to the dispersion of SBA-15. The template SBA-15 was prepared by using ethyl orthosilicate (TEOS) and hydrochloric acid, and then the dispersion of SBA-15 was mixed with 1,10 phenanthroline hydrate, ferric acetate, cobalt acetate tetrahydrate and ethanol by ultrasonic mixing, and the solution was evaporated and put into a high-purity argon environment at a constant temperature of 900 °C for 3 h, and then washed with NaOH solution, the Fe_xCo/NOMC catalyst material was obtained. Electrochemical tests illustrate the high level of activity of obtained catalysts, with the half-wave potential of the ORR reaction is 0.89 V at 10 mA/cm² and overpotential of the OER is 0.31 V. The Zinc air batteries assembled possessed a high specific energy of 820 Wh/kg at 100 mA/cm², a power density of 153 mW/cm² at 1.0 V, and a stability of up to 144 h in air. In the course of this study, a series of electrocatalysts featuring varying molar ratios of iron to cobalt were synthesized employing the template technique. This process allowed for the fine-tuning of the catalysts' surface chemistry and electronic makeup, thereby enhancing their overall electrocatalytic capabilities. Consequently, the rechargeable zinc-air batteries, integrated with these optimized catalysts, demonstrated both impressive power density and sustained operational stability.[1,4]

2.1.2 Use iron-cobalt-based carbon aerogels as effective oxygen catalysts

(1) Preparation method:

In this research, we crafted carbon aerogels impregnated with iron-cobalt dual-metallic centers (NCAG/Fe-Co), utilizing a dual-metallic hydrogel matrix as a foundation. These aerogels were designed to possess dual-functional electrocatalytic attributes, catering to both the oxygen reduction reaction (ORR) and the oxygen evolution reaction (OER). In the study, carbon aerogels containing iron and

cobalt bimetallic centers (NCAG/Fe-Co) were successfully prepared as efficient bifunctional electrocatalysts through a series of finely tuned experimental steps. First, hydrogels containing Fe and Co precursors were prepared by mixing 1,10-dimethylphenol (PM) with FeCl₂·4H₂O and Co(Ac)₂·4H₂O to form a solution of Fe(PM)₂²⁺ and Co(PM)₂²⁺, which was then blended with gelatin, silica (SiO₂), and MilliQ water. Subsequently, these hydrogels were freeze-dried and calcined at high temperatures at 900°C in a 97% Ar + 3% H₂ atmosphere to promote the evaporation of Zn atoms and the formation of atomic spaces to trap Fe or Co atoms. After calcination, SiO₂ nanoparticles were removed by HF acid etching to obtain the final NCAG/Fe-Co product.

(2) Performance Application:

The fabricated NCAG/Fe-Co demonstrated superior activities for both the oxygen reduction reaction (ORR) and the oxygen evolution reaction (OER) during electrochemical evaluations. Notably, the NCAG/Fe-Co presented a minimized potential difference of 0.64 V for the ORR/OER at a current density of 10 milliamperes per square centimeter, outperforming commercial platinum/carbon (Pt/C) and ruthenium dioxide (RuO₂) catalysts by achieving 60 millivolts less overpotential, as they typically show 0.70 V. Furthermore, the integration of NCAG/Fe-Co into the construction of pliable zinc-air batteries resulted in an elevated open-circuit voltage of 1.47 V, along with a peak power density reaching 117 mW cm⁻², coupled with commendable charging capacity and adaptability. These findings indicate that embedding atomically dispersed bimetallic sites within carbon aerogels is an effective strategy to markedly improve the capabilities of carbon-based electrocatalysts for energy storage and conversion applications.[2]

2.1.3 Efficient spinel iron oxide-cobalt-doped nitrogen-ordered mesoporous carbon catalysts

(1) Preparation method:

A spinel-type iron cobalt oxide/nitrogen-doped ordered mesoporous carbon (Fe_xCo/NOMC) catalyst was developed in the study for oxygen reduction reaction (ORR) and oxygen precipitation reaction (OER) in rechargeable zinc-air batteries. The catalysts were synthesized using a one-step method to embed iron and cobalt precursors into

nitrogen-doped ordered mesoporous carbon (NOMC). The specific synthesis process started by dissolving 1,10-diphenylhydrazine monohydrate (Phen) and iron(II) acetate with cobalt(II) acetate tetrahydrate in a mixture of ethanol and deionized water, followed by the addition of silica template SBA-15, evaporation of the solvent after ultrasonic stirring, followed by calcination at 900°C for 3 h in an argon atmosphere, and finally washing away the SBA-15 by NaOH solution template to obtain the Fe_xCo/NOMC catalyst.

(2) Performance Applications:

Electrochemical tests showed that the obtained Fe_{0.5}Co/NOMC catalyst exhibited excellent electrocatalytic activity when the molar ratio of iron to cobalt was 0.5, with an ORR half-wave potential of 0.89 V (vs. reversible hydrogen electrode RHE), and an overpotential for OER at 10 mA/cm² of only 0.31 V. The catalyst was applied to the assembly of rechargeable zinc-air batteries, and the batteries demonstrated a high energy density of 820 Wh/kg and a high power density of 153 mW/cm², as well as superior cycling stability in ambient air for up to 432 cycles (144 hours).

The physical and chemical properties of the catalysts were characterized in detail by X-ray photoelectron spectroscopy (XPS), Mössbauer spectroscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM) and Raman spectroscopy (Raman). The results showed that the excellent oxygen electrocatalytic performance of the catalysts was attributed to the electronic modulation effect of bimetallic oxides, the mesoporous structure of NOMC, the high electrical conductivity and the high specific surface area, and the catalytic active sites of its ORR and OER were nitrogen-activated carbon (N-C) and bimetallic oxides, respectively. These findings not only provide new ideas for the rational development and construction of efficient oxygen electrocatalysts, but also provide strong technical support for the practical application of zinc-air batteries.

2.2 Hydrothermal Method

2.2.1 Brief introduction:

The hydrothermal synthesis is conducted within a pressurized vessel, utilizing water as the solvent to establish a high-temperature, high-pressure setting conducive to the

dissolution and recrystallization of typically non-soluble substances. In the work of Song and colleagues, a biphasic water-oil hydrothermal technique was implemented for the creation of cobalt-enriched Fe₅(PO₄)₄·H₂O (Co-FPOH). This process commenced with the dissolution of a 50% aqueous phytanic acid in deionized water, constituting Solution A. Concurrently, iron(III) phytate (Fe-(C₁₇H₃₅COO)₃) was introduced into toluene, allowing for a 4-hour interaction to yield Solution B. The amalgamation of Solutions A and B within a temperature-controlled reactor set at 180 degrees Celsius for one hour precipitated the synthesis. Post-reaction, the product was subjected to multiple rinses with deionized water and ethanol, followed by a 60 degrees Celsius drying phase for 12 hours, culminating in the formation of Co-FPOH. This material showcased an OER overpotential of 290 millivolts, surpassing the 350 millivolts typically associated with RuO₂. The ORR activity catalyzed by Co-FPOH approached a four-electron transfer process, nearing the theoretical efficiency of Pt/C. When integrated into a zinc-air battery configuration, Co-FPOH endowed the system with an extended cycle life of 450 hours at a current density of 5 mA/cm², peaking at a power density of 167.8 watt-hours per square centimeter, and maintaining an open-circuit voltage of 1.42 volts. Additionally, the battery demonstrated a discharge capacity of 817 milliampere-hours per gram at a current density of 10 mA/cm². The Co-FPOH stands out as a superior bifunctional electrocatalyst, with its performance bolstered by the meticulous regulation of material diffusion at the oil-water interface, electrocatalysts with nanostructured morphology were prepared in the research, which exhibited high active bifunctional catalytic activity, and the assembled and prepared zinc-air batteries had high power density and long service life.[7]

2.2.2 Nitrogen-doped graphene-encapsulated FeCoMoS nanoparticles as advanced multifunctional catalysts for water separation devices and zinc-air batteries

(1) Preparation method:

In the study, the authors have successfully synthesized nitrogen-doped graphene-encapsulated FeCoMoS nanoparticles (FeCoMoS@NG) through well-designed experimental steps and applied them as

efficient and multifunctional catalysts for water decomposition devices and zinc-air batteries. The synthesis process began with the dissolution of iron(III) nitrate, cobalt(II) nitrate, sodium molybdate, and thioacetamide in deionized water (DI water), followed by the addition of glacial acetic acid and ultrasonication to promote sufficient mixing of solvents and precursors. Next, the mixed solution was slowly added to a graphene oxide (GO) suspension and injected with ethylenediamine to promote the interaction of metal ions with GO. Afterwards, the resulting mixture was transferred to a polytetrafluoroethylene (PTFE)-lined stainless steel autoclave for 12h of hydrothermal synthesis at 180 °C to promote the formation of nanoparticles and in situ growth of nitrogen-doped graphene. At the end of the synthesis, the FeCoMoS@NG nanocomplexes were finally obtained by repeated washing and drying with deionized water and ethanol.[10]

(2) Performance Applications:

Electrochemical tests showed that the FeCoMoS@NG nanocomplexes exhibited excellent trifunctional catalytic capabilities in alkaline environments, including OER, HER, and ORR. In the OER test, the FeCoMoS@NG catalysts required an overpotential of only 238 mV at a current density of 10 mA cm⁻², while in the HER test, the corresponding overpotential of 137 mV. In addition, in the ORR test, the FeCoMoS@NG catalyst showed a half-wave potential of 0.83 V, which exhibited comparable performance to that of commercial Pt/C catalysts. By applying the FeCoMoS@NG catalyst to zinc-air batteries, the researchers observed that the batteries showed excellent overall water decomposition performance by requiring only 1.58 V of cell voltage at a current density of 10 mA cm⁻² to achieve a current output of 10 mA cm⁻². [5,11]

2.3 In-Situ Growth Method

2.3.1 Brief introduction:

To prepare electrocatalysts by in situ growth method, a certain material is used as a substrate, and other materials are physically or chemically loaded on the substrate to design a composite material that combines the advantages of the two materials. Lei et al. firstly synthesized tetrakis(pentafluorophenyl)phthalocyanine, and dissolved pentafluorobenzaldehyde and

pyrrole in propionic acid by refluxing for 2.5 h, and then rotary evaporation and drying to obtain a dark residue, which was dissolved by CH₂Cl₂, and then washed by saturated sodium carbonate and water, subsequently, the mixture was dried using anhydrous sodium sulfate to remove any residual moisture, after which the resulting residue underwent purification through a silica gel column chromatography process. Methylene chloride (CH₂Cl₂) was employed to dissolve the leftover material. This solution was subsequently treated with a saturated solution of sodium carbonate to facilitate washing, followed by a rinse with water. After drying over anhydrous sodium sulfate to eliminate any remaining moisture, the product was further refined through silica gel column chromatography, yielding a purple compound identified as tetrakis(pentafluorophenyl)phthalide. Then the complex Co-P was obtained by refluxing cobalt acetate tetrahydrate and tetrakis(pentafluorophenyl)phthalocyanine in dimethylformamide for 4 h. Following the removal of the solvent via rotary evaporation, the resulting substance was subjected to purification using silica gel column chromatography, ultimately yielding a pure form of the cobalt-phthalocyanine complex (Co-P). The synthesis of the iron-phthalocyanine complex (Fe-P) was achieved by heating a blend of ferric dichloride and tetrakis(pentafluorophenyl)phthalocyanine in a CHCl₃/EtOH solvent mixture for one hour. The complex was then reconstituted in dichloromethane (CH₂Cl₂), a process repeated multiple times to ensure thorough dissolution. Post rotary evaporation, the Fe-P complex was redissolved in CH₂Cl₂, subjected to elution with a 2 M HCl solution, and subsequently purified through a silica gel column using a CH₂Cl₂/MeOH mobile phase, culminating in the acquisition of the purified Fe-P. Subsequently, the tetrakis(pentafluorophenyl)scarylene (Co-P) and the ferric(pentafluorophenyl)scarylene chloride (Fe-P) were deposited onto carbon nanotubes (CNTs). The resultant CNT-based (Co-P)_{0.5}(Fe-P)_{0.5}@CNT composites exhibited pronounced activity in the oxygen reduction reaction (ORR), highlighted by a half-wave potential of 0.80 volts. The electrode's potential reached 1.65 volts under a

current density of 10 milliamperes per square centimeter in a 0.1 molar solution of potassium hydroxide (KOH). When integrated into zinc-air batteries, these (Co-P)_{0.5}(Fe-P)_{0.5}@CNTs composites demonstrated a minimal voltage loss of 0.74 volts at a current density of 2 milliamperes per square centimeter. Furthermore, the assembled zinc-air battery, as reported by Wang et al. under similar conditions, featured a compact charge/discharge voltage gap of 0.74 volts, a substantial power density of 174.5 milliwatts per square centimeter, and remarkable charging stability exceeding 120 cycles.[8]

2.3.2 Electronic structure modulation of embedded multidimensional carbon-based iron-cobalt nanoparticles

(1) Preparation method:

In our investigation, we crafted Fe₁Co₂ alloy nanoparticles (Fe₁Co₂-NC) through an in situ synthesis approach, integrating them into a framework of nitrogen-doped carbon nanotubes and carbon nanosheets (CNTs/CNSs). The fabrication utilized Fe-chitosan, Co-chitosan chelate, and urea as the primary components, which were uniformly distributed via mechanical milling. Subsequently, additional chitosan was incorporated, blended, and subjected to milling before being heated in a nitrogen-rich environment. Throughout the calcination, urea underwent decomposition, leading to the progressive formation of two-dimensional g-C₃N₄ structures. These structures, along with the chitosan molecules, spontaneously assembled into porous carbon nanosheets studded with FeCo alloy nanoparticles at elevated temperatures. Concurrently, the g-C₃N₄ decomposed to produce CN_x fragments, which further catalyzed the in situ growth of nitrogen-doped carbon nanotubes (NCNTs), culminating in the formation of the Fe₁Co₂-NC composite.[12]

(2) Performance Applications:

The Fe₁Co₂-NC catalyst demonstrated remarkable dual-functional catalytic efficacy, particularly in the oxygen reduction reaction (ORR) and the oxygen evolution reaction (OER). Within the ORR domain, the Fe₁Co₂-NC presented a notable half-wave potential reaching 0.88 volts against the standard hydrogen electrode, coupled with a minimal overpotential of 0.356 volts in the OER. This resulted in an exceptionally low potential gap

of 0.706 volts, a figure that rivals the performance of the combined platinum/carbon (Pt/C) and ruthenium dioxide (RuO₂) catalysts, which typically show a gap of 0.69 volts. When integrated into the construction of zinc-air batteries (ZABs), using Fe₁Co₂-NC as the cathodic catalyst, these batteries realized a high open-circuit voltage of 1.501 volts, a peak power density of 203.4 milliwatts per square centimeter, and an energy density of 820.30 watt-hours per kilogram. After enduring a prolonged charge/discharge test spanning 209 hours, the system only experienced a minor voltage increase of 0.058 volts, underscoring the robust durability of the Fe₁Co₂-NC catalyst and highlighting its significant potential for practical applications in energy-related technologies.[3,12]

2.3.3 In situ growth of CoFeS₂ on metal-organic framework-derived Co-NC polyhedron
(1) Preparation method:

In the study, the preparation of Co-NC@CoFeS₂ composites started with the synthesis of ZIF-67 by co-precipitation followed by high temperature heat treatment under argon atmosphere to obtain Co-NC. To grow CoFeS₂ on Co-NC in situ, 15 mg of Co-NC powder was firstly added to a solvent mixture of dimethylformamide (DMF) and ethylene glycol (EG) in a ultrasonic bath for 30 min for uniform dispersion. Then, 1 mmol of CoCl₂·6H₂O, 1 mmol of FeCl₃·6H₂O and 2 mmol of thioacetamide (TAA) were added to the Co-NC dispersion and further stirred for 30 minutes. The mixed slurry was then transferred to a polytetrafluoroethylene (PTFE) lined stainless steel autoclave reactor, heated to 180 °C and maintained for 18 hours. At the end of the reaction, the precipitate was collected and washed with deionized water and finally dried at 60 °C to obtain the layered Co-NC@CoFeS₂ composite. In the comparison experiments, CoFeS₂ was synthesized without using Co-NC powder, but the same steps were carried out.

In terms of experimental conditions, the synthesis of ZIF-67 involved dissolving Co(NO₃)₂·6H₂O and 2-methylimidazole in methanol, mixing them, and then stirring at room temperature for 1 min and aging at room temperature for 6 h. The preparation of Co-NC, on the other hand, involved heating ZIF-67 at 120 °C for 2 h, followed by a further heating at 800 °C for 2 h, at a heating rate of 5 °C/min.

The Co-NC@CoFeS₂ was synthesized by mixing the solvents DMF and EG at a volume ratio of 1:1, at a reaction temperature of 180 °C, and at a reaction time of 18 h. These conditions were favorable for the uniform growth of CoFeS₂ on the surface of the Co-NC, avoiding the severe aggregation of the particles, contributing to the formation of a porous structure and enhancing the activity of the electrocatalyst. After the reaction was completed, Co-NC@CoFeS₂, an efficient bifunctional electrocatalyst for oxygen reduction reaction (ORR) and oxygen evolution reaction (OER), was obtained by cooling to room temperature, washing and drying steps.

(2) Performance Applications:

Co-NC@CoFeS₂, functioning as a dual-purpose catalyst, has proven to be highly effective for ORR and OER. Within an alkaline medium, this catalyst demonstrates reduced overpotentials and diminished Tafel slopes, indicative of its enhanced kinetics for the OER. When incorporated into a rechargeable zinc-air battery configuration, Co-NC@CoFeS₂, serving as the air electrode, has achieved notable benchmarks: a high open-circuit voltage of 1.44 volts, a substantial specific power density of 174.4 milliwatts per square centimeter, and an extended cycle life surpassing 400 cycles. Collectively, these attributes suggest that Co-NC@CoFeS₂ holds promise as an advanced electrocatalyst for zinc-air batteries, offering innovative pathways in the development and synthesis of high-performance electrocatalysts for such energy systems.[6]

3. Environmental Impact and Economic Feasibility

Cobalt-iron based electrocatalysts, as an emerging material to replace traditional precious metal catalysts, have significant advantages in terms of environmental impact and economic feasibility.

3.1 Environmental Impact

3.1.1 Resource Sustainability

Cobalt-iron based materials have richer crustal reserves compared to precious metals such as platinum and iridium, reducing dependence on rare resources and favoring sustainable development.

3.1.2 Life Cycle Assessment (LCA)

The synthesis of cobalt-iron based catalysts usually involves more environmentally friendly chemical methods such as templating and hydrothermal methods, which are less environmentally burdensome than traditional precious metal refining processes.

3.1.3 Recycling

Recycling technology for cobalt-iron based materials is relatively mature, which helps to reduce the environmental pollution caused by discarded catalysts.

3.2 Economic Feasibility Aspects

3.2.1 Cost-effectiveness

The raw material cost of cobalt-iron based electrocatalysts is much lower than that of precious metals, which directly reduces the production cost and makes large-scale production and application possible.

3.2.2 Performance to price ratio

Studies have shown that cobalt-iron based catalysts exhibit catalytic performance comparable to or even better than precious metal catalysts in applications such as water electrolysis for hydrogen production and zinc-air batteries, providing a higher cost performance ratio.

3.2.3 Market Potential

As the global demand for clean energy technologies grows, cobalt-iron based electrocatalysts have a huge market potential due to their excellent performance and cost advantages, and are expected to occupy an important position in the field of energy conversion and storage in the future.

3.3 Challenges and Outlook

Despite the obvious environmental and economic advantages of cobalt-iron-based electrocatalysts, they still face a number of challenges, such as uneven geographical distribution of cobalt resources and price fluctuations that may affect their economics. In addition, the environmental impact during large-scale production and the long-term stability of the catalysts still need to be further investigated. Future research should focus on optimizing synthesis methods, improving material properties, and developing effective recycling strategies to achieve commercialization and environmental sustainability of cobalt-iron based electrocatalysts.

4. Conclusions

This article has delivered an exhaustive summary of the progress made in the realm of cobalt-iron-based electrocatalysts, specifically designed for the hydrogen evolution reaction (HER) and the oxygen evolution reaction (OER) within the context of water electrolysis. It has highlighted the significant strides in developing these catalysts as efficient and cost-effective alternatives to traditional noble metal catalysts. The review has detailed the impact of various synthesis methods on the structural and catalytic properties of the electrocatalysts and has discussed innovative strategies to improve their performance and stability.

The article has underscored the significant capacity of cobalt-iron-based electrocatalysts to boost the performance of zinc-air batteries, highlighting their tangible utility in the realms of energy storage and transformation. Looking ahead, the review points to the importance of in-situ techniques for identifying active sites and computational methods for optimizing catalyst performance. It also underscores the need for further research in protective layer integration and surface defect engineering to bolster catalyst stability.

The collective efforts in addressing these challenges are expected to expand the applicability of these electrocatalysts, contributing to the advancement of clean and sustainable energy technologies. The review concludes with an optimistic outlook on the future of cobalt-iron based electrocatalysts and their integral role in the transition towards a green energy economy.

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