

Review

Design and Synthesis of Self-Supported Water-Splitting Transition Metal-Based Electrocatalysts via Electrospinning

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Abstract: Recent advances in transition metal-based electrocatalysts have significantly enhanced the hydrogen evolution reaction (HER) and oxygen evolution reaction (OER) in water electrocatalysis. Self-supported electrodes, where active sites are directly integrated with substrates, offer superior kinetics and stability compared to traditional powder-based electrocatalysts. The electrospinning technique is particularly effective for fabricating self-supported electrocatalysts with high surface areas, porosity, and uniform distribution of active sites, leading to improved catalytic performance. Despite extensive research on self-supported electrocatalysts, a comprehensive review focusing on those developed via electrospinning remains scarce. This review provides a detailed overview of the electrospinning process, the fundamental principles of water electrocatalysis, and recent progress in the development of transition metal-based electrocatalysts fabricated through this approach.

Keywords: electrospinning; transition metal catalysts; self-supported electrodes; electrocatalysis; water splitting



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1. Introduction

The global shift toward sustainable energy solutions has highlighted the importance of hydrogen as a clean and efficient energy carrier [1–4]. Hydrogen, with its abundant feedstock [4], high energy density [5], and environmental benefits [6], holds significant potential for addressing the growing demand for renewable energy [7]. Among the various methods for hydrogen production, water electrolysis [8] stands out as a sustainable approach using water as a feedstock to generate hydrogen and oxygen [9,10]. This process involves two key reactions: the cathodic hydrogen evolution reaction (HER) and the anodic oxygen evolution reaction (OER). However, both reactions are highly energy-intensive, requiring operating voltages of 1.8–2.0 V, which is significantly higher than the theoretical 1.23 V [3]. To enhance the efficiency of water electrolysis, highly active electrocatalysts are required to improve the sluggish reaction kinetics and reduce overpotential [11]. While noble metal-based catalysts demonstrate outstanding performance, their high cost and limited availability restrict widespread use [12]. As a result, there is a growing need to develop low-cost, highly efficient catalysts for water electrolysis.

The current research on water-splitting electrocatalysts has focused largely on transition metal compounds, including sulfides [13], selenides [14,15], phosphides [16,17], oxides, nitrides [8,18], carbides [19], etc. However, the performance of these noble metal-free catalysts in terms of activity and durability remains below expectations. The effectiveness of electrocatalysts depends on several key factors: the intrinsic properties of active sites [20], the number of active sites [21], the mass transfer efficiency [22], and the electron transfer

ability [23]. Various strategies have been proposed to address these factors. Among them, the design of nanostructures appears as one of the most widely explored approaches [24]. However, nanostructured electrocatalysts have some limitations for practical applications. For instance, nanoparticles are typically cast on a substrate using polymer binders, which increase electrical resistance, block active sites, and hamper mass transport. Additionally, the low conductivity of many electrocatalysts necessitates the use of conductive additives, which not only add complexity to the preparation process but also reduce the loading of active components. These drawbacks increase production costs and can result in poor mechanical stability due to the potential detachment of catalyst particles.

In light of these challenges, self-supported electrodes, where catalytically active phases are grown directly on conductive substrates, offer a promising alternative [25]. These electrodes offer several key advantages over conventional powder-based catalysts: (1) simplification of the electrode preparation process [26], (2) increased loading of active sites [27], (3) greater exposure of active sites by eliminating the involvement of binders [28], (4) improved charge transfer through uniform distribution of the catalyst on the substrate, (5) the ability to engineer hydrophilic or hydrophobic surfaces by adjusting the morphology and microstructure [29], and (6) enhanced stability through the immobilization of catalysts on the substrate, leading to superior electrocatalytic performance.

The methods for preparing self-supported electrocatalytic materials generally fall into two approaches: top-down and bottom-up. The top-down strategy involves dispersing active substances in solvents through mechanical or chemical processes, followed by deposition on the substrate to form thin membrane materials [30–32]. In contrast, bottom-up methods focus on building membrane materials through techniques like chemical vapor deposition, filtration, or electrostatic spinning [33]. Among these, electrostatic spinning has gained attention as a versatile and efficient technique to construct adjustable chemical components and well-defined micro/nanostructures [34].

Despite significant advances in the development of self-supported electrocatalysts, there is still a lack of comprehensive reviews focusing on those created through electrospinning. Therefore, we exclusively focus on electrospun self-supported electrocatalysts, a specialized domain that has hitherto received scant comprehensive examination. In this paper, we initiate our exploration by delving into the foundational principles of electrospinning, followed by furnishing a comprehensive elucidation of the principles governing electrolytic water splitting. Subsequently, we undertake a meticulous review of the recent advancements in the structural design and electrocatalytic properties of transition metal-based self-supported electrocatalysts fabricated via electrospinning. Finally, we delineate prospective trajectories for enhancing the efficiency and performance of these materials, proffering incisive insights for the design and development of next-generation electrocatalysts (Figure 1). This thorough investigation endeavors to bridge extant knowledge gaps and galvanize further innovation within the realm of sustainable energy electrocatalysis.

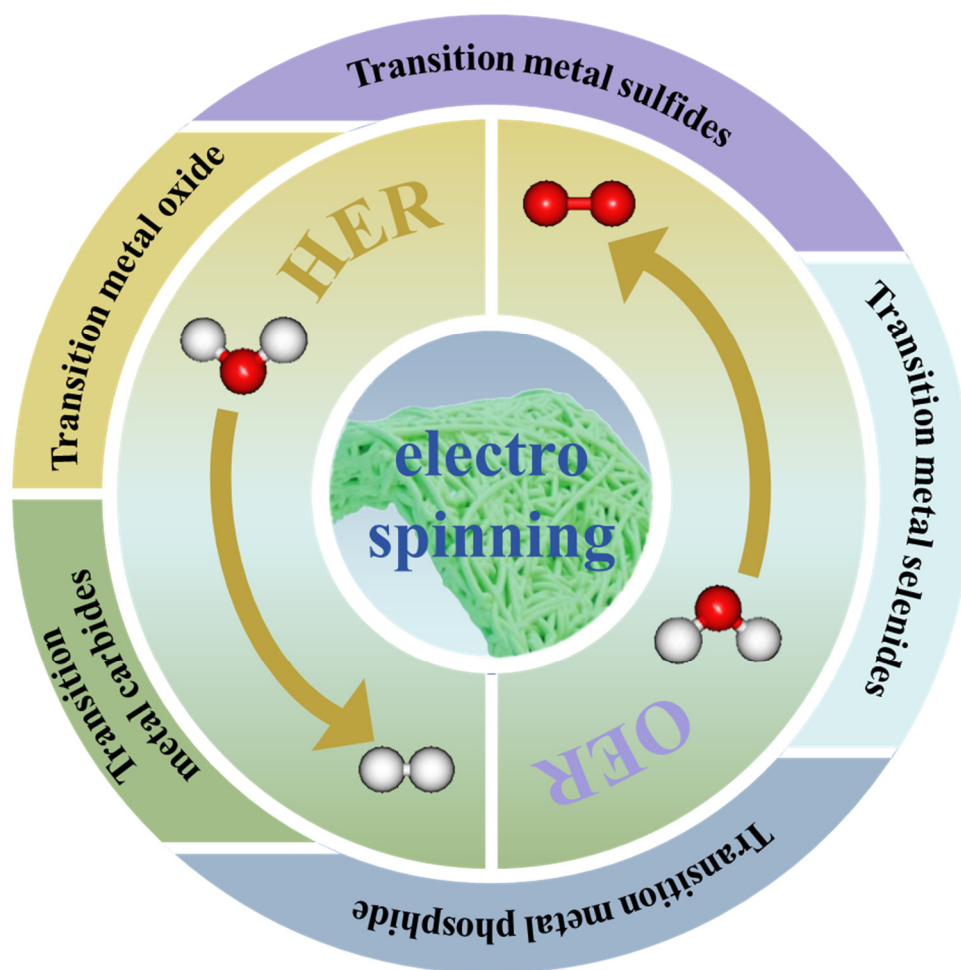


Figure 1. Schematic diagram showing the classification of transition metal-based water-splitting electrocatalysts with the employment of electrospinning technique.

2. Basics of Electrostatic Spinning

Electrospinning is derived from the electrospray technique, where high-voltage electrostatic forces are applied to propel liquid jets [35]. The equipment used for electrospinning consists of three main components: a high-voltage source, a nozzle, and a collector. The high-voltage source, which can be either Direct Current or Alternating Current, generates tens of kilovolts between the spinneret and the collector [36]. This creates a strong electrostatic field that counteracts the surface tension of the polymer solution at the nozzle, forming the Taylor cone [37]. When the electrostatic force surpasses the threshold, a jet with charge is propelled from the tip of the Taylor cone [38]. As the solution jet travels, the solvent evaporates, leaving behind charged polymer fibers that are stretched and thinned due to the movement of the jet. These fibers accumulate on a grounded collector as randomly oriented microfibers or nanofiber webs [39,40]. The final morphology of the fibers is influenced by several factors, including the properties of the polymer solution (such as molecular weight, viscosity, surface tension, and conductivity) and the processing parameters (such as voltage, distance between the nozzle and collector, and flow rate) [41,42].

With the increasing demand for one-dimensional nanomaterials, electrospinning has emerged as one of the fastest-growing and most versatile methods for nanofiber production. A wide range of nanofibers can be synthesized through this technique, including carbon-based fibers, metal oxides, polymers, and hybrid materials [43].

A variety of polymers are readily soluble in the corresponding organic solvents and can be spun into micro-nanofibers via electrospinning. Polymer precursors commonly

involved in this process include the following: 1. natural components (i.e., hyaluronic acid (HA), chitosan, alginate, gelatin, lignin, collagen, cellulose, and chitin [44]); and 2. synthetic polymers [45–49] (i.e., polyacrylonitrile (PAN), polymethylmethacrylate (PMMA), polyethylene oxide (PEO), polyvinyl alcohol (PVA), polystyrene (PS), polyvinylidene fluoride (PVDF), polyvinylpyrrolidone (PVP), poly(lactic acid) (PLA), polyvinyl chloride (PVC), and polycaprolactone (PCL)). In addition, inorganic nanofibers, including various metallic compounds, carbon, and their composites, are often produced by electrospinning with subsequent post-processing techniques such as air annealing or oxygen-free carbonization at elevated temperatures. These processes enable the creation of high-performance nanofibers with enhanced structural integrity. Nanofibers can serve not only as functional electrocatalysts but also as support matrices for other active substances [50]. In many cases, the performance of these materials can be further enhanced by combining electrospinning with various post-processing techniques, such as carbonization, sulfuration, selenization, phosphorization, self-assembly, chemical vapor deposition, chemical etching, and solvothermal growth [51]. These combined methods are crucial for creating high-performance composites with optimized electrocatalytic properties.

In summary, electrospinning is a versatile method for producing one-dimensional nanofibers from various materials, offering precise control over fiber morphology for applications like electrocatalysis. The technique allows the creation of high-performance fibers, especially when combined with post-processing methods such as carbonization. However, it requires the careful optimization of parameters like solution properties and voltage, which can be complex and time-consuming. Challenges include achieving uniform fiber morphology and reproducibility, particularly with hybrid materials. Despite these issues, ongoing advancements in process control and material design promise scalable production of high-performance nanofibers, with potential for innovations in energy storage and conversion technologies.

3. Principle of Electrocatalytic Water Splitting and Evaluation Criteria

3.1. HER Mechanism

HER is the cathodic half-reaction of water electrocatalysis. It is a multi-step electrochemical process that occurs at the electrode/electrolyte interface following two main reaction pathways. The first step is the Volmer reaction, which is then followed by either the Tafel or Heyrovsky process [52]. The mechanism of hydrogen production differs depending on the pH of the medium (Figure 2). In acidic media, hydrogen is primarily generated by the reduction of protons (H^+), while in alkaline media, hydrogen is produced through the reduction of water (H_2O) [53]. In addition, the HER process in neutral solutions closely resembles the mechanism in alkaline media. At each step, an electrocatalyst is essential to lower the energy barrier, accelerating the reaction and enhancing the efficiency of hydrogen generation [54].

(1) Acidic media

In acidic media, solvated protons serve as the main reactants in HER. The process consists of three fundamental steps [55,56].

Volmer



Tafel



Heyrovsky



The first step of the reaction is the Volmer reaction (Equation (1)), an electrochemical adsorption process where hydrated protons (H^+) combine with electrons at the surface of active sites, resulting in the adsorption of hydrogen atoms (H_{ad}). Next, depending on the concentration of these adsorbed atoms, hydrogen is either released through the Tafel pathway (Equation (2)) or the Heyrovsky pathway (Equation (3)) [57]. If the coverage of H_{ad} is high, neighboring hydrogen atoms directly couple to form hydrogen gas (H_2) in a process known as chemical desorption (Tafel reaction). Conversely, when the coverage of H_{ad} is low, the adsorbed hydrogen atoms combine with additional H^+ and e^- , producing H_2 through an electrochemical desorption process (Heyrovsky reaction) [57].

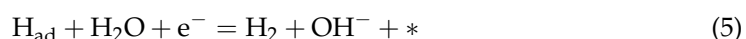
(2) Alkaline media

Compared to acidic media, the HER process in alkaline solutions is kinetically slower. This is due to the additional energy required to break the covalent bonds in H_2O for adsorption, a process more challenging than proton adsorption in acidic environments [54]. In alkaline media, the HER follows two key steps [3,54]:

Volmer



Heyrovsky



In the first Volmer reaction, water molecules undergo adsorption and dissociation at the surface of the active sites (Equation (4)), resulting in the formation of H_{ad} and hydroxide ions (OH^-). This is considered the rate-determining step (RDS) in alkaline HER. In the Heyrovsky step, H_{ad} reacts with water, gaining an electron to produce H_2 and additional OH^- (Equation (5)). While the overall HER process in alkaline solutions is similar to that in acidic media, the need to break H_2O bonds leads to slower kinetics, which makes the reaction more difficult. Despite this, alkaline conditions are less corrosive, which can enhance the long-term stability of the electrocatalysts compared to their performance in acidic environments [58].

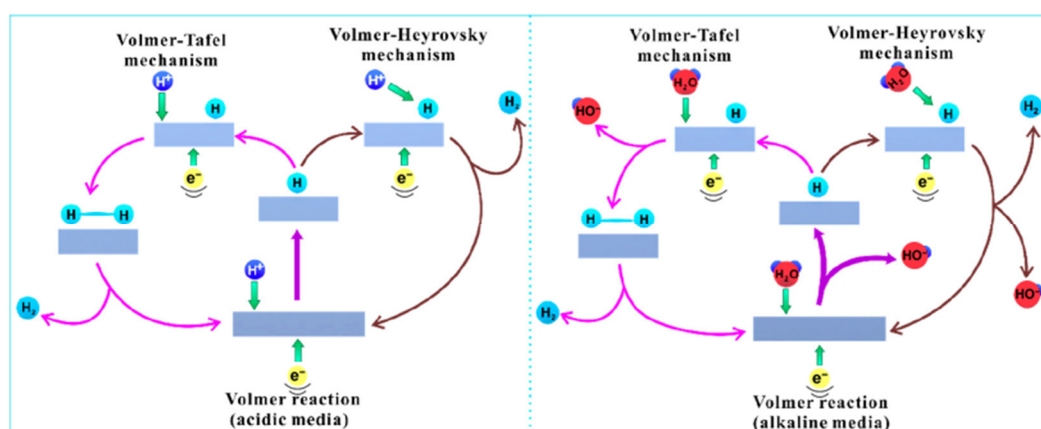


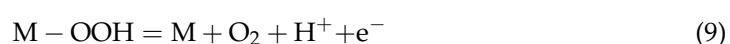
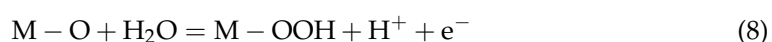
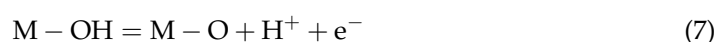
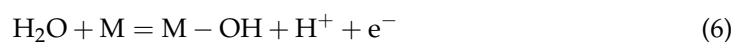
Figure 2. Mechanism of hydrogen evolution on the surface of an electrode in acidic (left) and alkaline (right) solutions [59]. Reproduced with permission. Copyright 2019 American Chemical Society.

3.2. OER Mechanism

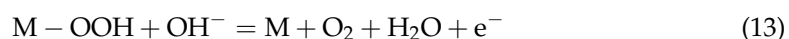
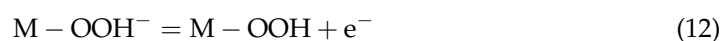
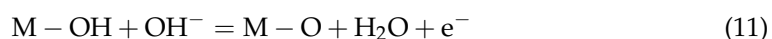
Compared to HER at the cathode, anodic OER is more complex with multiple intermediates involving a four-electron transfer process [60]. The slower reaction kinetics and intricate mechanisms make OER more challenging than HER [61]. Similarly to the HER process, the OER mechanism depends heavily on the surface structure of the electrocatalyst and the pH of the electrolyte. Throughout the OER process, several intermediates can form,

such as absorbed hydroxyl groups ($M-OH$), adsorbed oxygen ($M-O$), and adsorbed perovskite species ($M-OOH$). Oxygen (O_2) is eventually produced either by the direct combination of two $M-OH$ intermediates or through the decomposition of $M-OOH$ in acidic or alkaline media [62]. The OER mechanism in acidic and alkaline media is described as follows [3,55]:

(1) Acidic media:



(2) Alkaline media:



Due to the involvement of multiple intermediates, it can be difficult to pinpoint the rate-determining step for the OER and fully describe its kinetics. Understanding the interactions between the electrocatalyst surface and these intermediates is crucial for optimizing the overall OER performance [53,63]. In this context, density functional theory (DFT) calculations are often used to analyze the adsorption energies of intermediates and to model the reaction mechanism of OER electrocatalysts. To date, plenty of work has been performed in modeling the thermodynamics of electrochemical reactions [64]. Among electrocatalysts, Ru/Ir-based oxides have proven to be the most effective in acidic media. However, most non-precious metal catalysts are not stable in acidic environments, particularly at high current densities, due to their susceptibility to dissolution. As a result, transition metal-based OER electrocatalysts are more favorable for use in alkaline electrolytes, where they exhibit better stability and performance [65].

To sum up, HER and OER are essential components of electrocatalytic water splitting, with each reaction mechanism varying based on the pH of the electrolyte. In acidic media, HER proceeds via the Volmer, Tafel, and Heyrovsky steps, primarily involving proton reduction, while in alkaline media, water reduction predominates, leading to slower kinetics due to the added energy required to break water molecules. The OER mechanism, involving a more complex four-electron transfer process, also depends on the pH and electrocatalyst surface structure, with multiple intermediates such as $M-OH$, $M-O$, and $M-OOH$ formed during the process. OER in acidic media is typically dominated by Ru/Ir-based catalysts, which exhibit superior activity but suffer from instability under high current densities. In contrast, transition metal-based catalysts perform better in alkaline media, offering improved stability but requiring further optimization to enhance performance. Understanding the interactions between catalysts and reaction intermediates, often analyzed through DFT, is key to advancing the efficiency and stability of both HER and OER electrocatalysts for scalable water-splitting applications.

4. Synthesis and Morphologies of Water-Splitting Electrocatalysts

To date, various techniques have been employed to develop efficient water-splitting electrocatalysts, including electrospinning, hydrothermal, and sol–gel procedures. Electrospinning empowers the fabrication of materials with distinct one-dimensional nanofiber architectures. Through the precise modulation of process parameters, it facilitates the controlled growth of crystals, resulting in highly crystalline and well-ordered structures. Concomitantly, it endows the materials with favorable porosity characteristics and extensive surface areas, both of which are cardinal for augmenting catalytic efficiency. In the case of hydrothermal methods, by precisely manipulating the temperature and pressure within a confined reaction environment, materials with specific crystalline phases and defect structures can be synthesized, thereby exerting a deterministic influence on their physico-chemical properties. The sol–gel process, meanwhile, offers the advantage of fine-grained control over chemical composition and homogeneity, ensuring the uniformity and integrity of the final product, all of which cumulatively contribute to the overall performance.

With respect to morphology, diverse nanostructures assume indispensable roles. Nanofibers, owing to their elongated and continuous morphology, provide an optimized conduit for charge transfer, effectively minimizing electrical resistance and expediting electron mobility. Nanoparticles, characterized by their diminutive size and concomitantly high surface–volume ratio, expose a copious number of active sites, thereby catalyzing efficient catalytic reactions. Nanorods, with their anisotropic geometric configuration, can be strategically oriented to enhance the interaction with reactants, facilitating enhanced mass transport phenomena. Collectively, the judiciously designed synthesis methods and meticulously optimized morphologies operate in concert, synergistically bolstering the electrochemical performance of water-splitting electrocatalysts, thereby laying a solid foundation for the pursuit of more efficient and sustainable energy conversion pathways.

5. Water-Splitting Electrocatalysts via Electrostatic Spinning

Noble metal catalysts remain the most effective for water electrolysis, but their high cost and limited availability hinder widespread application. As a result, research in recent years has focused on developing catalysts made from inexpensive and abundant elements, particularly for renewable energy technologies [66]. Transition metals emerge as intriguing catalysts due to their unique electronic configurations. They can either provide d-orbital lone-pair electrons, acting as nucleophiles, or utilize empty d-orbitals as electrophilic sites during chemical reactions. The diverse chemisorption bonds formed on the d-orbitals of these transition metals enable molecular activation, reducing the activation energy of complex reactions [67]. Furthermore, different transition metal catalysts can alter the free energy of adsorption within the same reaction system, thereby influencing the reaction rate.

Among all the construction strategies, electrostatic spinning proves to be effective in generating high-performance self-supported transition metal electrocatalysts for water splitting. 1. Compared to other electrocatalytic reactions, water splitting involves two half-reactions, HER and OER, each with distinct kinetic barriers and mechanistic requirements. These dual demands necessitate bifunctional catalysts with complementary properties. The hierarchical porous structures and tunable compositions offered by electrospinning are particularly advantageous in addressing these dual challenges. 2. The interconnected porous networks reduce diffusion resistance for reactants and intermediates, thus enhancing mass and charge transport, which is particularly crucial for the OER. 3. Self-supported architectures minimize the detachment of active materials, a common issue in the harsh oxidative environment of water splitting, achieving significantly improved long-term stability.

The water splitting performances of electrospun transition metal electrocatalysts are summarized in Tables 1 and 2 as well as Figure 3.

Table 1. HER performance of reported electrospun transition metal-based electrocatalysts.

Catalysts	Preparation Methodology	Morphology	Electrolyte	Overpotential at 10 mA cm ^{−2} (mV)	Tafel Slope (mV dec ^{−1})	References
CNF/Co ₃ S ₄ /MoS ₂	Electrospinning and high-temperature carbonization followed by hydrothermal method	Nanofiber sandwich structure of “substrate/accelerator/catalyst”	1 mol KOH	80	99.2	[68]
NiCoP	Electrospinning with subsequent heat treatment of the precursor prepared	NiCoP polycrystalline fibers	1 mol KOH	141	66	[69]
HEMP CNFs	Electrospinning and in situ phosphating	Three-dimensional networks of self-supporting HEMP nanofibers	1 mol KOH	26	50.9	[70]
Fe ₇ Se ₈ /CNT/C	Electrospinning combined with in situ selenide process.	Uniformly distributed Fe ₇ Se ₈ nanoparticles on fibrous network	1 mol KOH	138	114	[71]
CM/Mo _x C@NC	Electrospinning method followed by specified calcination	Nanofiber interlinked network	0.5 mol H ₂ SO ₄	149	86	[72]
Sn/Mo ₂ C-CNFs	Electrospinning and carbonization process	Sn/Mo ₂ C nanoparticles encapsulated in carbon nanofibers of a 2D structure	1 mol KOH	144	49.8	[73]
CoN/MoC/NMCNFs	Electrospinning and carbonization process	1D N-doped multi-channel carbon nanofibers embedded with MoC/CoN nanoparticles	1 mol KOH	92.5	107.9	[74]
Mott–Schottky Ni/CeO ₂ @N-CNFs	Electrospinning method followed by specified calcination	Ni/CeO ₂ hetero-nanoparticles immobilized onto N-doped carbon nanofibers	1 mol KOH	100	85.7	[75]
Ni-MNCNF	Combination of coaxial electrospinning and pyrolysis	Nitrogen-doped carbon nanofibers with multi-channels loaded with nanoscale Ni nanoparticles	1 mol KOH	65	130.3	[76]
4%-SPCNF	Extraction of regenerated silk fibroin, followed by the electrospinning and carbonization	Bundled nanofibers	1 mol KOH	401.3	138.43	[77]
Fe ₃ C-Mo ₂ C/NC-0.15	Electrospinning and carbonization process	Nanoparticles within a porous carbon nanofiber matrix	0.5 mol H ₂ SO ₄	116	43	[78]
NiCoP/CNF900	Electrospinning and carbonization process	NiCoP encapsulated with the carbon nanofibers	1 mol KOH	130	83	[79]
Co/CoP@NC	Electrospinning followed by carbonization and phosphorization	Co/CoP nanoparticles encapsulated in carbon nanofibers	1 mol KOH	180	60.3	[80]
Mo _x C-NPC-1.0-800	Electrospinning and carbonization process	Porous carbon fibers with wrapped Mo ₂ C/MoC nanoparticles	1 mol KOH	71	114.1	[81]

Table 1. Cont.

Catalysts	Preparation Methodology	Morphology	Electrolyte	Overpotential at 10 mA cm ⁻² (mV)	Tafel Slope (mV dec ⁻¹)	References
C/Ni/NiP _{red}	Electrospinning followed by carbonization and H ₂ reduction	Incorporation of nanoparticles in the carbon matrix	0.5 mol H ₂ SO ₄	262	45.4	[82]
MoS ₂ /CoS ₂ -2@HCNFs	Electrospinning and high-temperature carbonization followed by hydrothermal method	Hollow nanofibers with dispersed nanosheets	1 mol KOH	124	88.7	[83]
CoS-2@CNF	Extraction of regenerated silk fibroin, followed by the electrospinning, carbonization, and hydrothermal method	Homogeneous dispersion of small CoS nanoparticles onto the rough surface of carbon nanofibers	1 mol KOH	168	265.01	[84]
CoMoS@CNF	Electrospinning and high-temperature carbonization followed by hydrothermal method	Carbon nanofibers with nanosheet	1 mol KOH	105.2	152.8	[85]
Fe-CoP/PCNF	Electrospinning followed by carbonization and phosphorization process	Porous nanofibers with dispersed nanoparticles	0.5 mol H ₂ SO ₄	151	53.9	[86]
FeNiP/NPC800	Electrospinning and carbonization process	Nanofibers with rough surfaces and dispersed nanoparticles	0.5 mol H ₂ SO ₄	277	95	[87]

Table 2. OER performance of reported electrospun transition metal-based electrocatalysts.

Catalysts	Preparation Methodology	Morphology	Electrolyte	Overpotential at 10 mA cm ⁻² (mV)	Tafel Slope (mV dec ⁻¹)	References
Fe(III)/NiS	Electrospinning technique followed by alkaline hydrothermal method	One-dimensional hollow structure with vertical nanosheets on the surface	1 mol KOH	264	77.6	[88]
Co-C/Ni ₁ Fe ₁ S	Electrospinning–carbonization–electrodeposition strategy	Hierarchical heterostructure with Co-C nanofibers core and amorphous NiFeS nanosheets sheath	1 mol KOH	233	53.1	[89]
NiCoP	Electrospinning with subsequent heat treatment of the precursor prepared	NiCoP polycrystalline fibers	1 mol KOH	540	70	[69]
Cobalt phosphate fibrous network	Simple precipitation method and electrospinning technique	Hydrated 1D cobalt phosphate fibrous network	1 mol KOH	245	229	[90]
Se-CoSe ₂ /CNFs	Electrospinning combined with in situ growth of ZIF-67 strategy, followed by carbonization and a facile selenization process	One-dimensional CNFs with ZIF-67 as a template	0.1 mol KOH	325	60.89	[91]

Table 2. Cont.

Catalysts	Preparation Methodology	Morphology	Electrolyte	Overpotential at 10 mA cm ⁻² (mV)	Tafel Slope (mV dec ⁻¹)	References
LFNOSe-III NFs	Electrospinning and subsequent heating treatment	Porous perovskite nanofibers	1 mol KOH	287	87	[92]
Co-ZIF _{1.5} /10CNF ₂	Electrospinning of CNF and Co-ZIF and calcination as a bifunctional catalyst	Co nanoparticles uniformly dispersed and encapsulated inside the CNFs	0.1 mol KOH	390	122	[93]
Mott–Schottky Ni/CeO ₂ @N-CNfS	Electrospinning method followed by specified calcination	Ni/CeO ₂ hetero-nanoparticles immobilized onto N-doped carbon nanofibers	1 mol KOH	230	54.2	[75]
S/LMO-E	Electrospinning and sulfurization treatment	Nanorods and tiny nanoparticles	1 mol KOH	314	47	[94]
SNCF-NF-Co ₃ O ₄ @N ₂	Electrospinning followed by co-precipitation method and sintering under N ₂	Heterostructure with oxygen vacancies	0.1 mol KOH	343	71.3	[95]
CoNC/NCNTs@CNF	Electrospinning method followed by pyrolysis process	3D callistemon-like structure with 1D nanofiber matrix	1 mol KOH	390	82	[96]
NiFe@NCNFs	Electrospinning process and subsequent heat treatment	One-dimensional mesoporous structures with metal components encapsulated in the N-doped carbon nanofibers	1 mol KOH	294	52	[97]
CoP/NC	Electrospinning followed by MOF fabrication	Ultra-small CoP nanoparticles incorporated into nitrogen-doped carbon nanofibers derived from ZIF-67	1 mol KOH	290	62	[98]

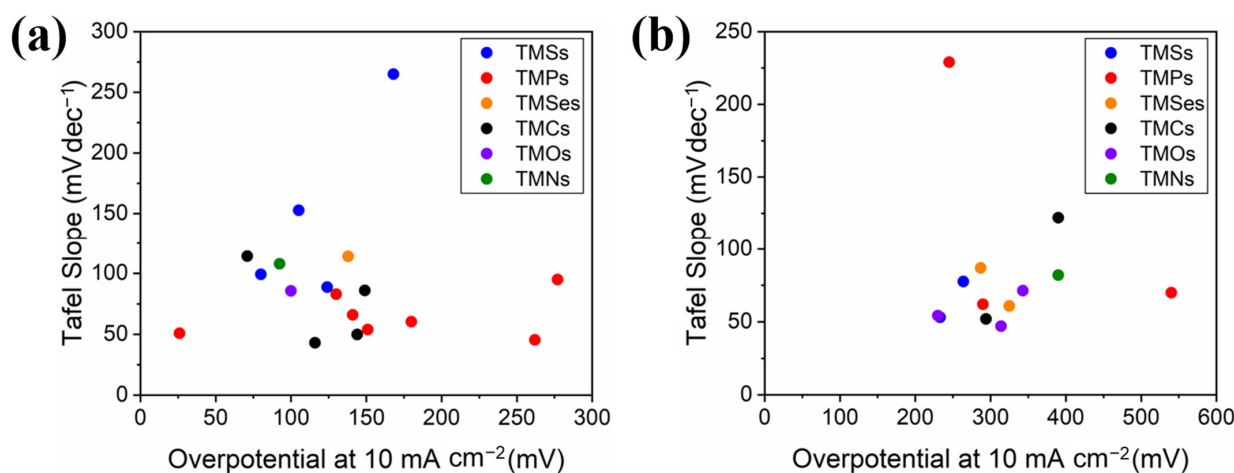


Figure 3. Summary of the water splitting performance of the reported electrospun transition metal-based electrocatalysts with the relationship of overpotential at 10 mA cm⁻² and Tafel slope. (a) HER performance; (b) OER performance.

5.1. Transition Metal Sulfides (TMSs)

TMSs have garnered significant attention due to their exceptional electrical conductivity, thermal and mechanical stability, and redox capabilities. These properties make them highly attractive for applications in catalysis, energy conversion, storage, and even optoelectronic devices [99–101]. When combined with electrospun nanofibers, these metal sulfides can form innovative nanostructures, leading to hybrid materials with unique interfaces such as core–shell, Janus, and hierarchical structures. These hybrid materials exhibit enhanced electron transfer and abundant active sites, essential for catalysis and other energy-related applications [102]. The strong interaction between the different components in these materials often leads to electronic modulation and the reconstruction of active sites, improving the overall catalytic performance.

Electrospun nanofibers not only provide structural stability but also serve as an excellent support matrix for metal sulfides, often leading to synergistic effects that enhance the long-term stability of these hybrid catalysts. For example, Zhong et al. synthesized [88] novel Fe(III)/NiS electrocatalysts via electrospinning combined with an interfacial adsorption method (Figure 4a–c). The resulting tubular morphology and highly active Ni(Fe)OOH during oxygen evolution led to a low overpotential of 264 mV at 10 mA·cm^{−2} (η_{10}), outperforming conventional RuO₂ and other metal sulfide-based electrocatalysts. Li et al. developed [89] a catalyst (Co-C/NiFeS nanofibers) composed of carbon nanofiber-supported amorphous NiFeS nanosheets embedded with cobalt nanoparticles using a combination of electrospinning, carbonization, and electrodeposition. The optimized catalyst demonstrated impressive OER performance in 1 mol·L^{−1} KOH, with a low η_{10} of 233 mV and a Tafel slope of 53.1 mV·dec^{−1}, along with excellent HER activity.

Among TMSs, molybdenum disulfide (MoS₂) has attracted considerable interest due to its layered structure and favorable electron arrangement [103]. The Gibbs free energies of hydrogen adsorption (ΔG_{H^*}) at the Mo and S atomic edges of MoS₂ are near zero, making these sites highly active for HER catalysis. However, MoS₂ suffers from low current-carrying density and poor electrical conductivity, which limit its catalytic performance [104]. To address these limitations, researchers have focused on synthesizing MoS₂ nanomaterials with exposed edges on high-conductivity carbon nanofibers (CNFs) to improve HER efficiency. Guan et al. demonstrated [68] this by growing MoS₂ in situ on nitrogen-doped nanofibers with Co₃S₄ nanoparticles via electrospinning, carbonization, and hydrothermal processes (Figure 4d). The resulting CNF/Co₃S₄/MoS₂ sandwich structure allowed molybdenum disulfide to act as the primary HER catalyst, while Co₃S₄ nanoparticles accelerated electron transfer from the substrate to the catalyst, promoting water molecule decomposition. The CNF/Co₃S₄/MoS₂ composites exhibited a η_{10} of only 80 mV and a Tafel slope of 99.2 mV·dec^{−1} (Figure 4e,f). Due to the in situ growth, Co₃S₄ particles remained stably embedded in the CNF, facilitating prolonged and efficient HER activity (Figure 4h,i).

As a conclusion, TMSs are renowned for their exceptional electrical conductivity, stability, and redox properties, making them attractive for catalysis and energy conversion applications. When combined with electrospun nanofibers, TMSs form hybrid nanostructures with enhanced catalytic performance due to improved electron transfer and abundant active sites. These hybrid materials benefit from synergistic effects that enhance stability and overall efficiency, addressing some of the limitations of TMSs such as poor conductivity and low current-carrying capacity while improving long-term catalytic performance.

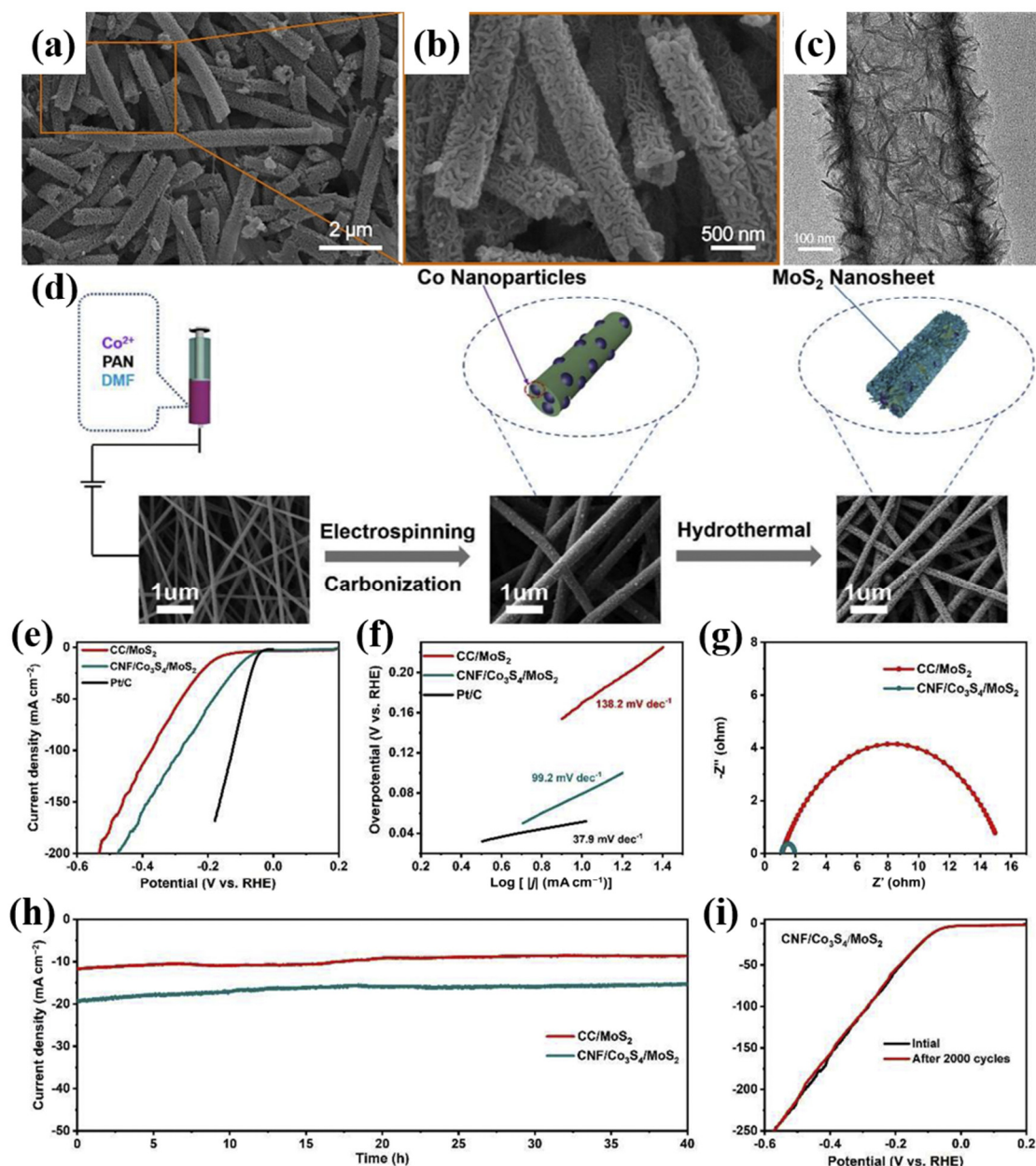


Figure 4. (a,b) SEM and (c) TEM images of Fe(III)/NiS-18 NTs. Reproduced with permission. Copyright 2023, Elsevier. (d) The schematic illustration of the CNF/Co₃S₄/MoS₂ synthesis. For CNF/Co₃S₄/MoS₂, CC/MoS₂, and Pt/C, the LSV (e), the corresponding Tafel slope curves (f), and the EIS (g) curves in 1 M KOH. (h) The time–current density curves of CNF/Co₃S₄/MoS₂ and CC/MoS₂ under a constant voltage. (i) Comparison of the LSV curves before and after 2000 cycles of CV. Reproduced with permission. Copyright 2022, Elsevier.

5.2. Transition Metal Phosphides (TMPs)

TMPs have garnered considerable attention due to their unique HER and OER catalytic properties [105]. The negatively charged phosphorus (P) sites in TMPs are known to weaken the metal-hydride (M-H) binding strength, facilitating hydrogen desorption and enhancing HER activity [106,107]. Simultaneously, the positively charged metal sites in TMPs can form metal-oxygen/hydroxyl radicals, which serve as active species during OER, significantly improving the electrocatalytic efficiency [108].

A key strategy to further enhance the performance of TMPs is their combination with conductive carbon-based materials such as carbon nanotubes, carbon nanofibers, and graphene. These conductive matrices provide an excellent platform to boost electrical

conductivity and facilitate charge/mass transport. Electrospun nanofibers, with their high specific surface area and rapid charge transfer capabilities, are especially well suited for this purpose [109,110]. For instance, Yang et al. synthesized [69] NiCoP electrocatalysts using a needle-free electrospinning technique. These electrocatalysts exhibited impressive HER activity, with low overpotentials of 141 mV in alkaline media and 146 mV in acidic environments at a current density of $10 \text{ mA} \cdot \text{cm}^{-2}$. The corresponding Tafel slopes were 53.0 and $97.8 \text{ mV} \cdot \text{dec}^{-1}$, respectively, indicating its fast reaction kinetics. In addition, the bimetallic phosphides demonstrated excellent OER performance in 1 M KOH and sustained durability during long-term stability tests. The bimetallic phosphides also show excellent oxygen precipitation reactivity in the alkaline medium 1 M KOH and satisfactory durability in long-term stability tests. The innovative fibrous structure of these electrocatalysts plays a crucial role in modulating their electronic properties, enhancing the availability of electrochemically active sites, and improving their overall catalytic performance in water-splitting reactions. Furthermore, the fibrous architecture improves the conductivity, utilization, and mechanical stability of the electrodes while also promoting better permeability and access to reactive sites.

Sankar et al. demonstrated [90] another approach by mixing synthesized cobalt phosphate powder with polymers and solvents to form fibers through electrospinning (Figure 5a). They successfully produced a network of hydrated, one-dimensional cobalt phosphate fibers (Figure 5b–d) which exhibited promising electrocatalytic activity. This material achieved a relatively low η_{10} of 245 mV in 1 M KOH (Figure 5e), along with a small Tafel slope of $229 \text{ mV} \cdot \text{dec}^{-1}$. In addition, the wrapping of TMPs by nanofibers not only enhanced the electrocatalytic performance but also protected the TMPs from corrosion in alkaline environments, significantly improving the stability of the composite catalyst (Figure 5f). Wang et al. took a more advanced approach by preparing [70] three-dimensional (3D) networks of self-supported high-entropy metal phosphide (HEMP) nanofibers via electrospinning and in situ phosphorylation. These nanofibers showed an outstanding HER performance with a η_{10} as low as 26 mV and a Tafel slope of $50.9 \text{ mV} \cdot \text{dec}^{-1}$. This exceptional catalytic activity was attributed to the synergistic effects of multiple metals and the formation of metal-P bonds. The introduction of electronegative P atoms modulated the electronic structure of the catalyst, inducing lattice strain and lowering the free energy of hydrogen adsorption. This enhanced the overall electrocatalytic performance, making the HEMP nanofibers highly efficient for hydrogen evolution.

To sum up, TMPs have gained attention for their unique catalytic properties in both HER and OER. The phosphorus sites in TMPs help weaken metal-hydride bonds, promoting efficient hydrogen desorption, while metal sites facilitate oxygen species formation, enhancing OER efficiency. Combining TMPs with conductive carbon-based materials, such as electrospun nanofibers, improves their electrical conductivity, charge transport, and catalytic performance. The fibrous structures further enhance the active site availability, stability, and overall efficiency of TMP-based electrocatalysts for water-splitting applications.

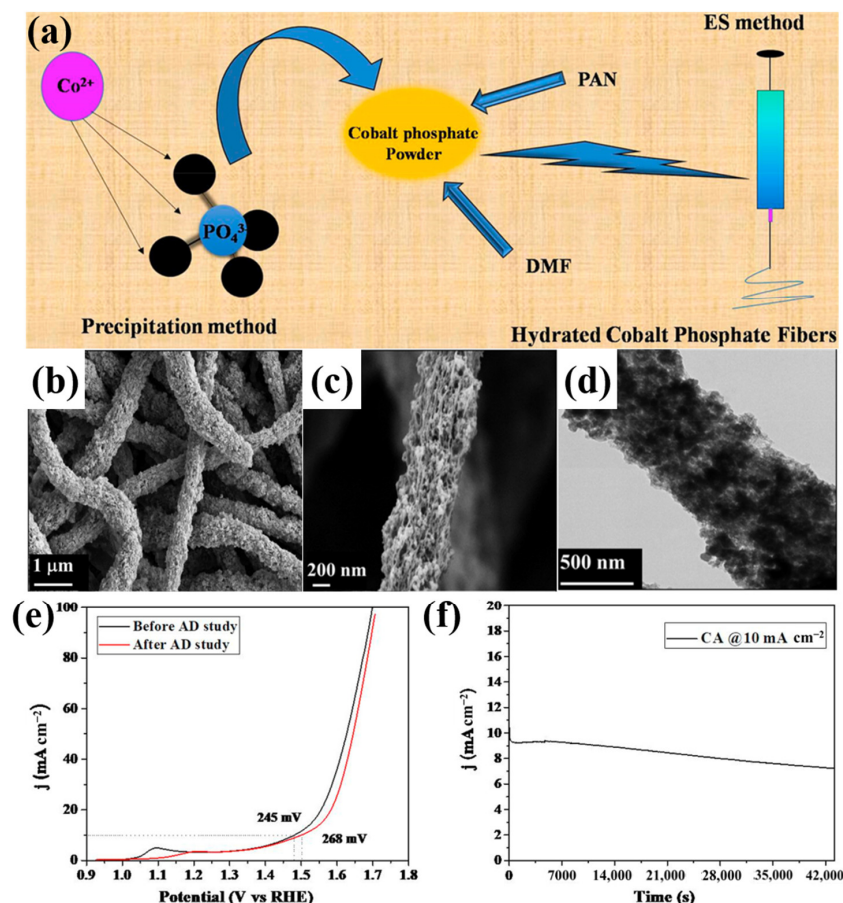


Figure 5. (a) Schematic illustration of the synthetic procedure for making hydrated cobalt phosphate fibers. (b,c) The FE-SEM images and (d) HR-TEM images of the hydrated cobalt phosphate fibers. (e) OER LSV curve and (f) stability study of the cobalt phosphate fibrous network. Reproduced with permission. Copyright 2021, Elsevier.

5.3. Transition Metal Selenides (TMSeS)

In the structure of TMSeSs, the Se-H bonds in TMSeSs are weaker than those found in TMSs and TMPs, resulting in improved hydrogen dissociation efficiency and excellent HER activity [111]. In the context of OER, TMSeSs facilitate more efficient molecular oxygen transport and accelerate OER kinetics. This is largely due to the cumulative 3d-2p repulsion between negatively charged selenium sites and the transition metals, which efficiently release oxygen from the active sites, contributing to superior OER performance. These characteristics make TMSeSs highly valuable in the field of overall water-splitting electrocatalysis [112].

The integration of electrospinning techniques with TMSeSs further enhances their catalytic potential. Electrospun nanofibers help increase the electrochemically active surface area, providing more active sites for catalysis, reducing contact resistance, and facilitating faster charge transfer [113,114]. For example, Cui et al. synthesized [91] selenium-doped CoSe_2 embedded in CNFs ($\text{Se-CoSe}_2/\text{CNFs}$) using electrospinning coupled with the ZIF-67 in situ growth strategy, followed by carbonization and a selenization process. The selenium doping not only modulated the electronic structure of CoSe_2 but also improved the reaction kinetics and optimized the adsorption of intermediates. The one-dimensional (1D) CNFs provided enhanced conductivity and structural stability, while the ZIF-67 template prevented the agglomeration of the materials. As a result, the $\text{Se-CoSe}_2/\text{CNF}$ catalysts demonstrated excellent OER performance, requiring only 325 mV overpotential at 10 mA cm^{-2} .

Li et al. prepared [92] porous $\text{LaFe}_{1-x}\text{Ni}_x\text{O}_3$ (LFNO) chalcogenide nanofibers through electrospinning and subsequent heat treatment with selenium doping (Figure 6a–d). The selenium-doped LFNO nanofibers required a low η_{10} of just 287 mV with a Tafel slope of 87 mV and excellent stability in alkaline medium (Figure 6e–g). The high specific surface area provided by the porous nanofibers improved the mass-specific activity of the catalysts. Additionally, selenium doping enhanced the intrinsic conductivity of $\text{LaFe}_{0.25}\text{Ni}_{0.75}\text{O}_3$, improving charge transfer kinetics and mass transfer efficiency (Figure 6h–m).

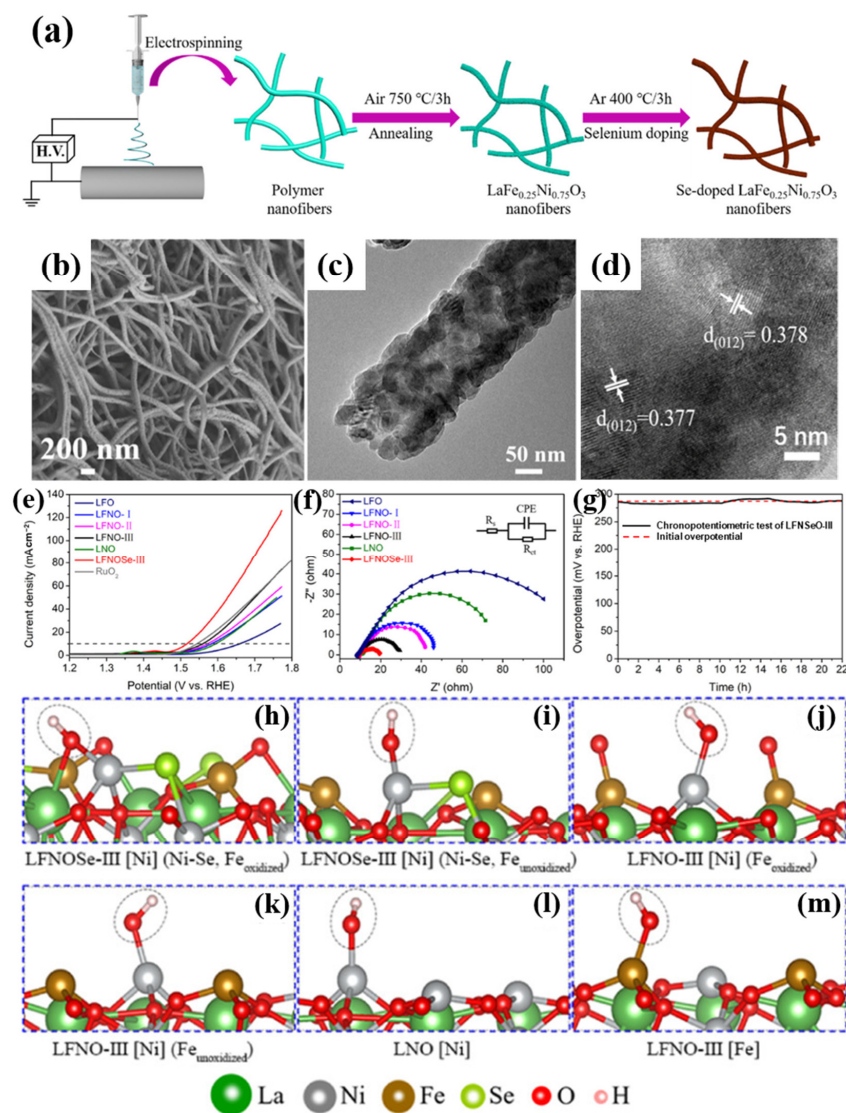


Figure 6. (a) Schematic illustration of the processing of Se-doped $\text{LaFe}_{0.25}\text{Ni}_{0.75}\text{O}_3$ nanofibers. (b) The SEM image, (c) TEM image, and (d) HRTEM image of LFNOSe-III. (e) The polarization curves and (f) Nyquist plots of LFO, LFNO-I, LFNO-II, LFNO-III, LNO, and LFNOSe-III. (g) The chronopotentiometric measurements of LFNOSe-III at 10 mA cm^{-2} . (h–m) Corresponding adsorption geometries of *O and/or *OH intermediates on catalysts. Reproduced with permission. Copyright 2020, American Chemistry Society.

As a brief summary, the integration of electrospinning with TMSes significantly enhances their electrocatalytic properties. By increasing surface area and facilitating faster charge transfer, electrospun nanofibers improve both hydrogen evolution and oxygen evolution reactions. Selenium doping optimizes reaction kinetics while enhancing structural stability and conductivity for better performance in water splitting.

5.4. Transition Metal Carbides (TMCs)

The growing interest in various TMCs, such as Mo_2C , W_2C , and Fe_3C , has led to extensive research into their water-splitting capabilities [115] due to their metal-like electronic structures, excellent hydrogen adsorption properties, high stability, and low cost [116–118]. The carbon-based framework in TMCs can effectively adjust the distribution of metal ions, resulting in a more uniform distribution of active sites [93,117,119]. For instance, He et al. synthesized [93] a cowpea-like Co-ZIF/CNF bifunctional catalyst using a straightforward electrospinning and calcination method.

Among different categories of TMCs, molybdenum carbide stands out as one of the highest-performing electrocatalysts, attributed to its d-band electronic structure, which closely resembles that of platinum [72]. However, the carbonization of Mo-based compounds at high temperatures often results in the aggregation and overgrowth of MoC [120]. To mitigate this issue, electrostatic spinning technology has been employed to produce metal-embedded carbon nanofibers. The limited mobility of metal particles on the carbon surface helps maintain a high dispersion of nanoparticles, effectively suppressing aggregation [121]. Zhang et al. developed [73] a bimetallic carbon-based electrocatalyst consisting of Sn/ Mo_2C composite nanoparticles encapsulated in carbon nanofibers. This unique structure prevents the binding and aggregation of $\beta\text{-Mo}_2\text{C}$ with the metal nanoparticles, ensuring the structural stability of the electrocatalyst and reducing the energy barrier during the water-splitting process. The catalyst demonstrated impressive HER performance in alkaline media, requiring a low η_{10} of 144 mV overpotential and exhibiting a Tafel slope of 49.8 mV-dec^{-1} over 60 h of stable operation. Wang et al. further advanced this field by preparing [74] one-dimensional nitrogen-doped multichannel carbon nanofibers encapsulating MoC/CoN nanoparticles (Figure 7a–c). This design helped avoid nanoparticle aggregation while increasing the specific surface area of the catalysts, resulting in the better accessibility of active site (Figure 7d–g). The resulting CoN/MoC/NMCNFs exhibited enhanced HER performance in both alkaline and acidic electrolytes, achieving a η_{10} of only 92.5 mV and 199.2 mV, respectively.

To sum up, TMCs, such as Mo_2C and W_2C , are gaining attention for their exceptional stability, hydrogen adsorption, and low-cost properties, making them ideal for water-splitting applications. Electrospinning aids in producing metal-embedded carbon nanofibers, which prevent nanoparticle aggregation, enhancing the catalyst's performance and structural stability.

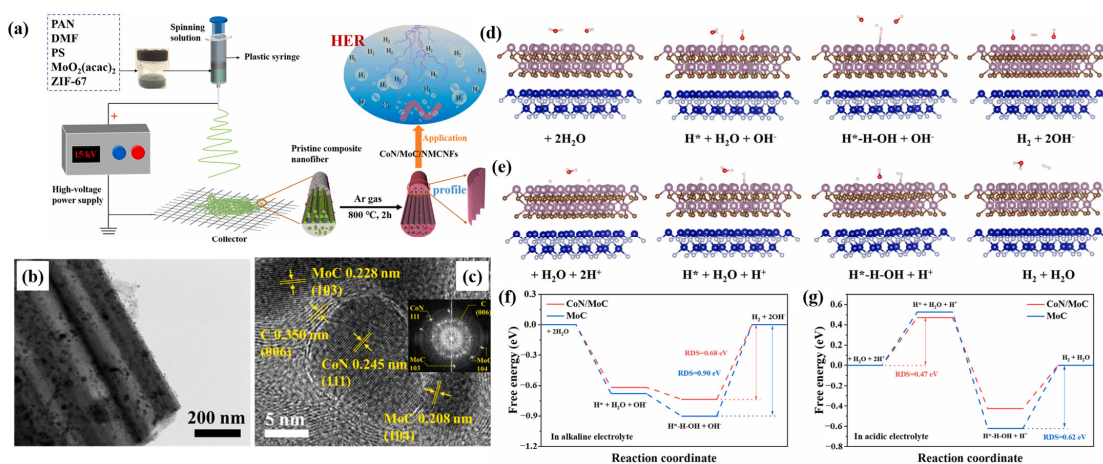


Figure 7. (a) Synthetic process of CoN/MoC/NMCNFs electrocatalysts by electrospinning. (b,c) HR-TEM images of CoN/MoC/NMCNFs. (d,e) Schematic configurations and (f,g) free energy profiles of (d,f) alkaline HER and (e,g) acidic HER through the Volmer–Heyrovsky–Rideal pathway on the CoN/MoC surface. Reproduced with permission. Copyright 2023, Elsevier.

5.5. Transition Metal Oxides (TMOs)

TMOs are widely utilized in electrocatalysis due to their availability, lower cost compared to noble metals, and their ability to provide d orbitals for oxygen binding [122–125]. For example, Li et al. developed [75] a Mott–Schottky-type electrocatalyst by immobilizing Ni/CeO₂ heterogeneous nanoparticles on nitrogen-doped carbon nanofibers (N-CNFs). This catalyst design significantly enhanced the charge transfer rate, optimized the chemisorption energy of reaction intermediates, and accelerated the reaction kinetics. As a result, the Ni/CeO₂@N-CNFs exhibited excellent catalytic activity for both HER and OER in alkaline solutions, achieving overpotentials of 100 mV for HER and 230 mV for OER at 10 mA·cm^{−2}.

Among the various TMO catalysts, perovskite-type oxides, such as monocalcicite (ABO₃) [126], dicalcicite (A₂B₂O₆) [127], Ruddlesden–Popper chalcocite (A₂BO₄) [128], and pyrochlore-type oxides (A₂B₂O₇) [129], have gained recognition as excellent electrocatalysts. These materials are notable for their low-cost transition metals, robust skeletal structures, intrinsic oxygen vacancies, versatile electronic configurations, and combinatorial flexibility [130,131]. Wang et al. explored this further by selecting [94] appropriate doping elements based on ionic radius, electronegativity, and oxygen vacancy formation energy. They introduced oxygen vacancies into high-entropy perovskite oxides (HEPOs) using a two-step strategy via electrospinning. This approach optimized the number of orbital-filling electrons, significantly increased the active surface area, boosted oxygen vacancy content, and improved conductivity. In addition, the pronounced pH dependence and tetramethylammonium cation inhibition clearly identify the involvement of lattice oxygen-mediated mechanism. The obtained catalyst exhibited a highly efficient OER catalyst with a η_{10} of 314 mV at 10 mA·cm^{−2} and a Tafel slope of 47 mV·dec^{−1}. This work highlights the potential of high-entropy catalysts for efficient water splitting, providing a promising path for the production of renewable hydrogen. Yuan et al. also contributed to this area by preparing [95] the Co-based perovskite of SrNb_{0.1}Co_{0.7}Fe_{0.2}O_{3−δ} (SNCF) nanofibers through electrospinning (Figure 8a), significantly increasing the specific surface area and providing more active sites. Co₃O₄ nanoparticles were then uniformly anchored onto the SNCF nanofibers using a co-precipitation method (Figure 8b–d), followed by sintering under a nitrogen atmosphere. The resulting SNCF-NF-Co₃O₄@N₂ heterostructures contained numerous oxygen vacancies, which contributed to the excellent OER catalytic activity and long-term stability (Figure 8e,f), with a low η_{10} of 340 mV.

As a conclusion, TMOs are widely used in electrocatalysis due to their low cost, availability, and ability to provide oxygen-binding d orbitals. In particular, perovskite-type oxides stand out for their structural stability, oxygen vacancies, and versatile electronic configurations. Electrospinning techniques enhance their performance by increasing surface area and optimizing active sites.

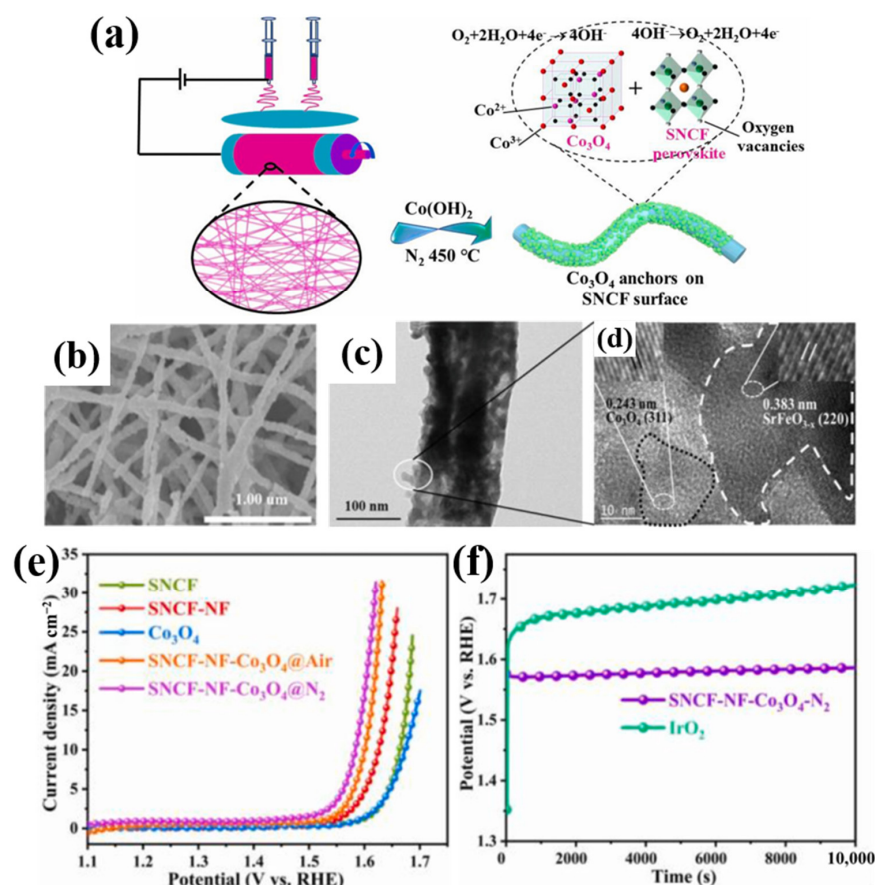


Figure 8. (a) Schematic illustration of the preparation of the SNCF-NF-Co₃O₄@N₂ sample. (b) The SEM, (c) TEM, and (d) HETEM images of SNCF-NF-Co₃O₄@N₂. (e) The OER LSV curves, and (f) OER stability test of SNCF-NF-Co₃O₄@N₂. Reproduced with permission. Copyright 2024, Elsevier.

5.6. Transition Metal Nitrides (TMNs)

TMNs are intriguing compounds formed by the incorporation of nitrogen into the transition metal lattice. This insertion significantly alters the material's properties, blending the characteristics typical of covalent compounds, ionic crystals, and transition metals [132,133]. Nanostructured TMNs are typically synthesized via the temperature-programmed nitridation of oxidized precursors, a process known as a “local structural reaction” [134,135]. During nitridation, nitrogen atoms expand the metal lattice, weaken interatomic forces, and redistribute d-band electrons, resulting in a high-density state close to the Fermi level [136], resulting in enhanced electrocatalytic performance [137]. However, TMNs usually suffer from high overpotentials, particle agglomeration, and suboptimal catalytic performance. Addressing these limitations has been the focus of recent research, with strategies aimed at modifying the composition, structure, morphology, and surface properties of TMNs to enhance their catalytic activity. For example, Yao et al. designed and synthesized [96] 1D CNFs using electrospinning, forming a unique callistemon-like nanostructure. This structure featured the in situ formation of N-doped carbon nanotubes (NCNTs) that integrated into a 3D hybrid nanostructure. The presence of NCNTs enabled high mass transport and an increased electrochemically active region, while the Co-N-doped graphitic carbon at the tips of the structure exposed catalytic sites more effectively to the electrolyte. This configuration also facilitated fast electron and ion transport, contributing to enhanced catalytic performance. The resulting electrocatalyst exhibited a η_{10} of 390 mV for OER, with a Tafel slope of 82 mV·dec⁻¹. For HER, the catalyst achieved a η_{10} of 190 mV with a Tafel slope of 117 mV·dec⁻¹. Wei et al. constructed [97] porous nitrogen-doped carbon nanofibers (NCNFs) encapsulated with nickel–iron (NiFe) alloy nanoparticles (NiFe@NCNFs).

as OER electrocatalysts (Figure 9a). The combination of a 1D mesoporous structure and the N-doped carbon nanofibers, along with the optimized binary metal composition (Figure 9b–d), resulted in an optimal synergistic effect (Figure 9e,f). This led to enhanced OER performance, with a low overpotential of 294 mV at a current density of $10 \text{ mA} \cdot \text{cm}^{-2}$. The catalyst also demonstrated excellent durability in alkaline solution, maintaining performance for over 10 h. Moreover, the two-electrode water electrolyzer with a NiFe@NCNF anode and Pt/C cathode showed extraordinary overall water splitting performance, with an initial cell voltage of only 1.531 V at 10 mA cm^{-2} , as well as long-term stability up to 20 h (Figure 9g,h). Feng et al. utilized [98] electrospinning combined with metal–organic framework (MOF) preparation to create hybridized CoP/N-doped carbon (NC) nanofiber composites, designed as bifunctional oxygen electrocatalysts. The CoP/NC catalysts exhibited good electrical conductivity thanks to the exposure of more active sites. As a result, excellent OER performance was achieved in alkaline solution with a η_{10} of 290 mV and a Tafel slope of $62 \text{ mV} \cdot \text{dec}^{-1}$.

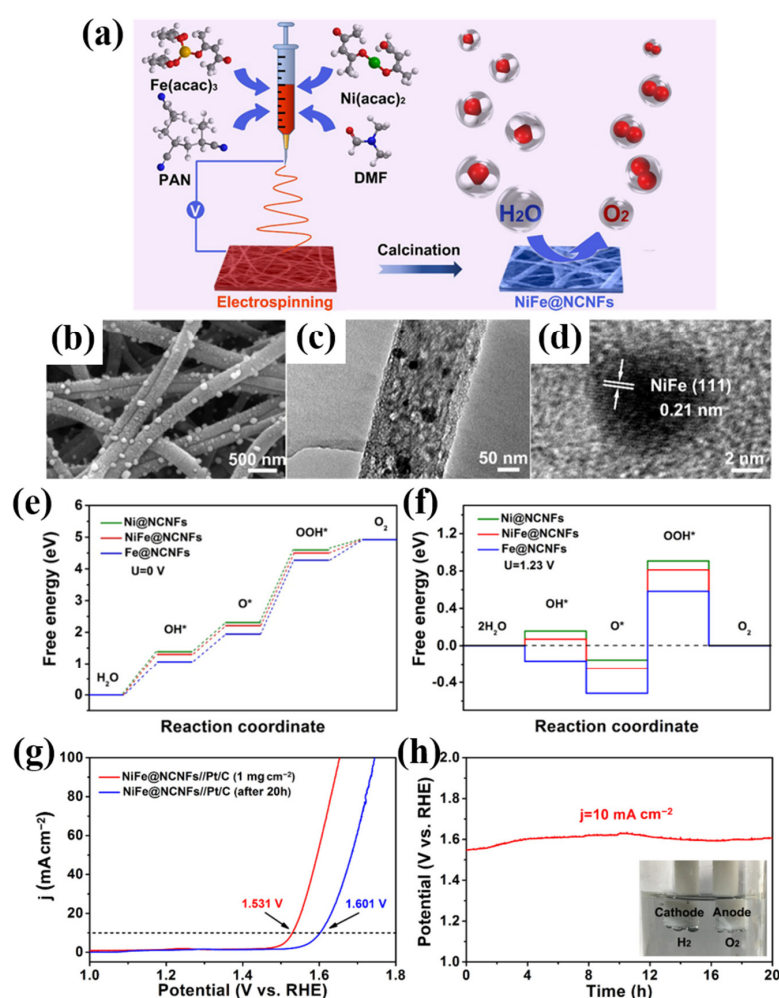


Figure 9. (a) Schematic illustration of the synthesis process of the NiFe@NCNFs. (b) The SEM and (c,d) TEM images of the NiFe@NCNFs. Free energy profiles (e) at the zero potential ($U = 0 \text{ V}$) and (f) equilibrium potential ($U = 1.23 \text{ V}$). (g) The overall water splitting LSV curves of the NiFe@NCNFs in a two-electrode device. (h) The overall water splitting chronopotentiometric curve of the NiFe@NCNFs at 10 mA cm^{-2} in 1.0 M KOH . Reproduced with permission. Copyright 2020 American Chemical Society.

To sum up, TMNs are created by incorporating nitrogen into transition metal lattices, enhancing electrocatalytic performance. However, challenges like high overpotentials and particle agglomeration remain. Recent advancements, such as using electrospun

nanostructures with nitrogen doping or metal alloy integration, address these limitations, improving catalytic activity and stability for OER and HER.

6. Conclusions

In summary, this review has furnished a comprehensive and meticulously detailed exposition of the recent extraordinary advancements in electrospun electrocatalysts for water splitting. During the past five years, a heterogeneous assemblage of transition metal sulfides, phosphides, carbides, selenides, nitrides, oxides, and affiliated compounds has emerged as pre-eminent contenders in the realm of contemporary research. Electrospinning, as a highly innovative and propitious technique within the purview of electrocatalysis, has unequivocally manifested its distinctive capabilities in endowing materials with one-dimensional architectures. These architectures not only augment electron and mass transport kinetics but also engender expansive surface areas and an exceptional aptitude for the fine-grained manipulation of chemical compositions and active sites. Through an exhaustive exploration of the fundamental principles underpinning electrospinning, a profound dissection of the characteristic traits of transition metal-based compounds, and a lucid elucidation of the merits attendant to their integration, a resilient knowledge foundation has been established, poised to galvanize further investigative pursuits.

Nevertheless, significant impediments endure that necessitate immediate redress. Firstly, from a mechanical vantage point, electrospun materials presently exhibit a relatively diminished mechanical strength when juxtaposed with other self-supporting electrodes. This inadequacy severely circumscribes their direct and unimpeded incorporation into practical energy conversion apparatuses. Consequently, future research endeavors must be steered towards devising novel stratagems and materials engineering paradigms to fortify their mechanical integrity. This could potentially encompass the exploration of composite materials, nanostructured reinforcements, or avant-garde fabrication procedures capable of conferring augmented tensile and compressive strength.

Secondly, environmental variables, with humidity being a particularly prominent factor, pose a formidable barricade to the consistent quality and reproducibility of electrospun fibers. Even within exquisitely controlled laboratory environs furnished with state-of-the-art humidity regulation apparatus, fluctuations in humidity can instigate disruptions in the spinning process, precipitating erratic fiber morphologies and, ineluctably, elevated energy consumption. To surmount this obstacle, it is of paramount urgency to integrate real-time environmental monitoring systems with automated feedback loops. Such systems would facilitate the dynamic modulation of humidity levels during electrospinning, thereby ensuring a stable and regulated milieu. Additionally, the development of closed-loop electrospinning chambers equipped with advanced dehumidifiers or humidifiers is indispensable. These chambers would not only stabilize environmental conditions but also vouch for the safe production of electrospun fibers with uniform morphology and quality. Moreover, we advocate the exploration and development of temperature-controlled spinning heads and adaptive nozzle designs. These innovative constituents would possess the capacity to modulate solution flow rates in real time in response to environmental perturbations, potentially augmented by machine learning algorithms. These algorithms could prognosticate and optimize process parameters predicated on continuous environmental inputs and historical data, thereby effecting a revolutionary transformation of the electrospinning process.

Furthermore, while electrospinning has demonstrated its efficacy in fabricating one-dimensional nanostructures, our current capabilities in effecting precise control over surface morphology, defect generation, and grain boundary manipulation remain nascent. Future work must pivot on the design and implementation of advanced synthesis methodologies

and cutting-edge surface/interface engineering tactics. This could potentially entail the utilization of templating techniques, molecular self-assembly, or precision etching to engineer nanostructures with hitherto unprecedented complexity and control. By attaining such precise control, we can unlock novel tiers of electrocatalytic performance.

Finally, from an ecological and economic perspective, the preponderant reliance on organic solvents in electrospinning represents a significant Achilles' heel. These solvents not only inflate production costs but also engender grave toxicity and safety hazards, both during the manufacturing process and in potential end-of-life disposal scenarios. Therefore, the urgent development of environmentally innocuous, green solvents that can satisfy the exacting process requirements is not merely a desirable aspiration but an ineluctable exigency. Such solvents would underpin the scalability and long-term viability of electrospinning for electrocatalyst fabrication, paving the way for a more sustainable future.

Overall, by methodically addressing these multifaceted challenges, we can unlock the heretofore unexploited full potential of electrospun electrocatalysts in water-splitting and energy conversion applications, heralding a new era of clean energy technology.

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Conflicts of Interest: The authors declare no conflicts of interest.

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