



Fe doping modifying electronic structure of NiSe₂ for boosting electrocatalytic oxygen evolution reaction

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Abstract

The commercialization of hydrogen production by water electrolysis is severely hampered by the sluggish kinetics of anodic oxygen evolution reaction (OER), mainly arising from the four-electron transfer process. Electronic structure modification has been shown to be favorable for accelerating the reaction kinetics of OER by not only improving the intrinsic activity of inherent active site but also largely enhancing the electrical conductivity. Herein, we have reported a metal–organic framework (MOF)-derived strategy for the synthesis of Fe-NiSe₂ nanocatalysts. Upon combination of experimental data and theoretical analysis, it is reported that Fe doping can greatly modify the electronic structure of NiSe₂ and thus substantially promote their electrocatalytic OER performance. More importantly, it is also reported that Fe-dopant is identified as active site, meanwhile stimulating the adjacent Ni atoms as active site for OER. As a result, an overpotential of merely 277 mV is demanded for the optimal Fe-NiSe₂ nanocatalyst to achieve 10 mA cm⁻² and long-term electrochemical stability for more than 35 h, which surpasses the benchmarked RuO₂ catalyst.

Keywords Doping · Electronic structure · NiSe₂ · Electrocatalytic oxygen evolution reaction

Introduction

Electrochemical water splitting is widely recognized as a promising technology for the storage of renewable energy and production of green and high-density hydrogen [1–3]. Although in great promise, the practical application of electrochemical water technology is greatly hampered by the intrinsically sluggish oxygen evolution reaction involved in a four-proton-coupled electron transfer process [4–7]. To date, benefitting from their low cost, environmental friendliness, high abundance, and potentially high OER activity, transition metal chalcogenides have thus been widely investigated and attracted increasing interests [8–10]. In addition, it is also reported that chalcogenides possess higher conductivity, more varied valence states and phases, and abundant

redox chemistry than conventional transition metal oxides. For instance, NiSe (2630 S cm⁻¹) exhibits a few orders of magnitude higher conductivity than NiO (0.01–0.32 S cm⁻¹) [11, 12]. All of these favorable terms enabled the chalcogenides to be promising OER electrocatalyst. Among various chalcogenides, Ni-selenides have been confirmed to be potential OER catalyst due to the suitable adsorption energy of various intermediates and adjustable electronic structure [13, 14]. For an update, the best Ni-selenides reported so far can deliver a current density of 10 mA cm⁻² with the overpotential lower than 250 mV [15, 16]. However, most of the Ni-selenides delivers an overpotential of at least 300 mV [17], and furthermore, a current stability was maintained for only dozens of hours, letting alone a high current density for thousands of hours. One of the major obstacles that limit their catalytic activity is the non-optimized electronic structure for electron transfer and the adsorption/desorption of OER intermediates [18, 19]. Therefore, tuning or creating an optimal electronic structure of Ni-selenides will benefit for the substantial improvement in electrocatalytic OER performance [20, 21].

In recent years, many effective strategies have been proposed for tuning the electronic structure [22, 23]. Among

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them, heteroatom doping is reported to be a highly effective method of realizing the electronic structure regulation [24, 25]. According to the *d*-band center theory, the adsorption energy of oxygen intermediates could be flexibly tuned by heteroatom doping [26, 27]. For example, Ta doping can induce the lattice expansion of layered double hydroxide (LDH) and modify electronic structure through electron transfer from Fe to Ta [28]. Also, the modified electronic structure among Ni, Fe, and Ta and the modified e_g orbital of Ta induced by charge transfer also benefit for the adsorption of OH species on Ta site in Ta-doped NiFe LDH and increase the intrinsic metallic property of NiFe LDH. In addition, it is also demonstrated that heteroatom doping can also activate the adjacent metal atoms to further improve the intrinsically OER activity [29]. More importantly, excepting for optimizing the binding strengths with oxygen intermediates, it is demonstrated that heteroatom doping can also substantially suppress the dissolution of the electrocatalyst during long-term electrochemical operation [30].

By taking considerations of the intrinsically high activity of Ni-selenides and positive influences of heteroatom doping on the optimization of electronic structure, we herein reported the synthesis of Fe-doped NiSe₂ nanocatalysts via a simple MOF-derived strategy. Importantly, the optimal Fe-NiSe₂-6 catalyst can achieve a current density of 10 mA cm⁻² with the overpotential of 277 mV. Inspiringly, the water-alkali electrolyze of Fe-NiSe₂-6 || Pt/C exhibited a low cell voltage of 1.57 V to reach 10 mA cm⁻² and excellent durability for at least 30 h. By combining the experimental data and theoretical study, it is demonstrated that the electronic structure optimization of Ni after Fe doping predominantly contributes to the substantial improvement in electrocatalytic OER activity

and therefore provides a better understanding on the key importance of electronic structure engineering for the promotion of OER activity.

Experimental section

Preparation of NiFe-MOF-74

NiFe-MOF-74 were prepared by a facile solvothermal based on previous report [31]. Typically, 90 mg 2,5-dihydroxyterephthalic acid, 157 mg nickel nitrate hexahydrate (Ni(NO₃)₂·6H₂O), and 15.7 mg ferrous acetate (Fe·(C₂H₃O₂)₂) were poured into the mixed solution of 30 mL DMF, 1.8 mL ethanol, and 1.8 mL DI water. After 30 min of sonication, the mixture was transferred into a 100-mL Teflon-lined stainless autoclave for keeping reaction at 120 °C for 24 h. After cooling to room temperature, the NiFe-MOF-74(6:1) were obtained by centrifugation and washed with methanol and ethanol for several. For comparison, the Ni-MOF, NiFe-MOF-74(3:1) and NiFe-MOF-74(9:1) were also prepared by only tuning the content of Fe·(C₂H₃O₂)₂ to 0 mg, 31.4 mg, and 10.4 mg, respectively.

Preparation of Fe-NiSe₂

Ten milligrams of NiFe-MOF-74(6:1) was dispersed into 5 mL DI water, 1 mL aqueous solution with 12 mg SeO₂ was also added into the above solution, and subsequently, 0.1 mL N₂H₄·H₂O was dropped into the mixture. After stirring for 10 min, the mixture was poured into a 30-mL Teflon-lined stainless autoclave to maintain for 8 h at 160 °C. After cooling to room temperature, the product Fe-NiSe₂-6 was collected through centrifugation and washed with DI water.

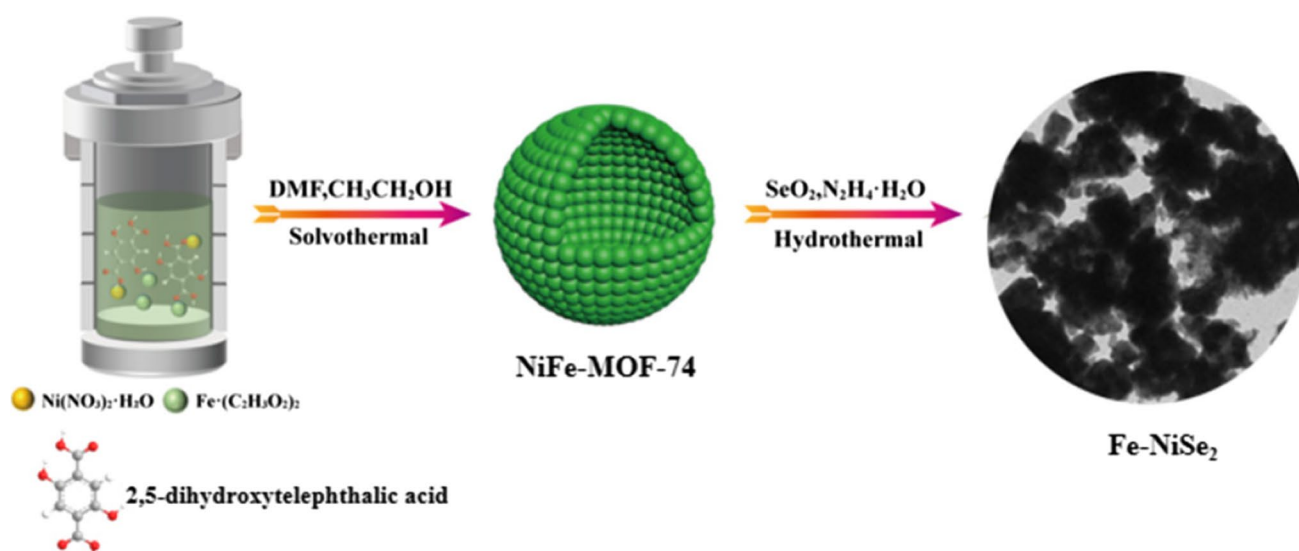


Fig. 1 The scheme for the synthesis of Fe-NiSe₂

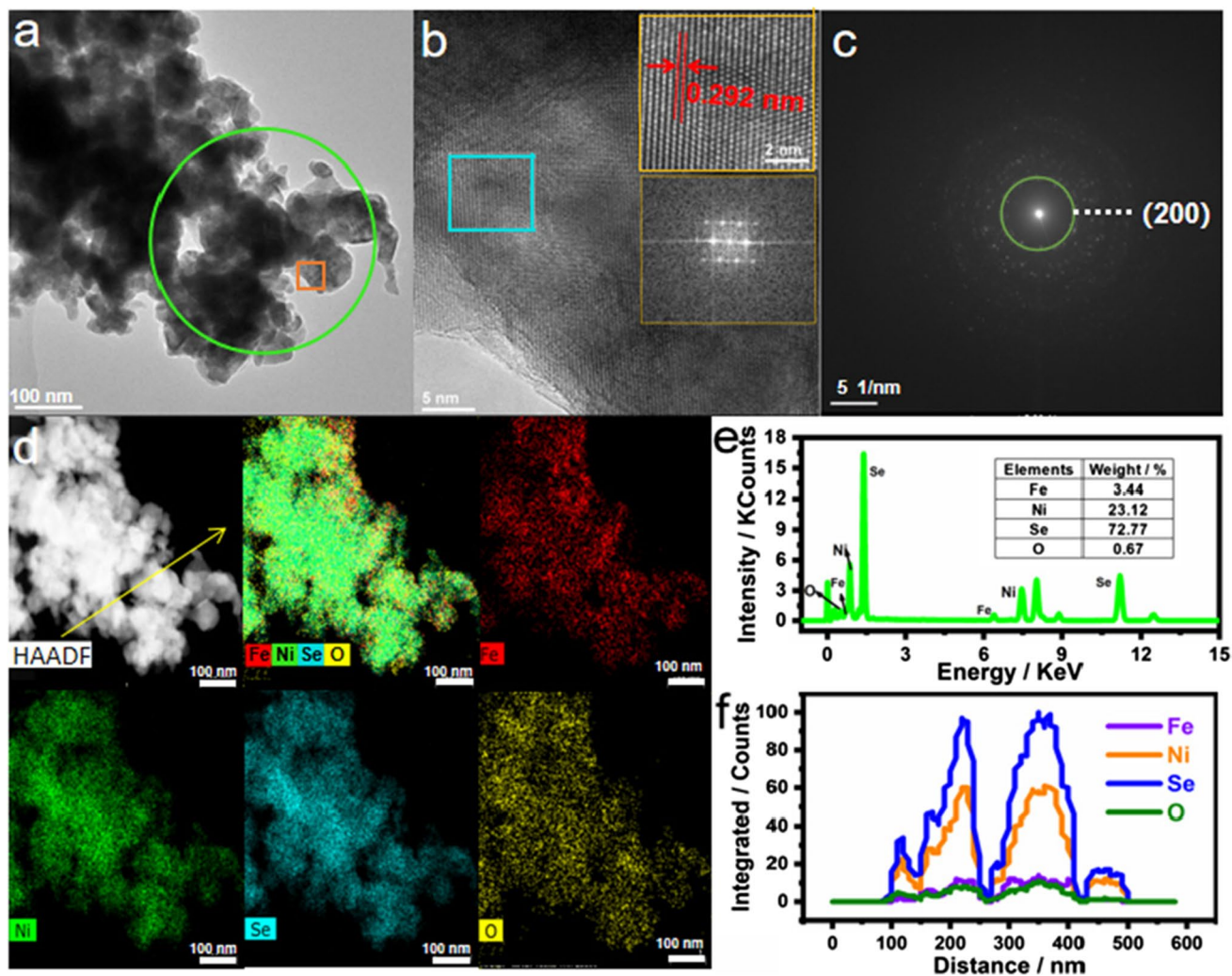


Fig. 2 a TEM image of Fe-NiSe₂; b HRTEM images (orange area in a), inset images represents the corresponding HRTEM of area and FFT patterns of selected areas of fresh green box; c SAED pattern (corresponding the fresh green area in a); d STEM image and

the corresponding elemental mapping images of Fe, Ni, Se, and O in Fe-NiSe₂-6; e EDS spectrum of Fe-NiSe₂-6; f the compositional line profile highlighted in yellow line

Characterizations

The phase and structure of all the samples were confirmed by powder X-ray diffraction (XRD, Rigaku UIV, Japan). The morphology of the samples was observed on scanning electron microscopy (TEM, JEOL-2100, Japan). The valence states and composition of surface elements in the samples were explored by X-ray photoelectron spectroscopy (XPS, JEOL JPS-9010 MC, Japan).

Electrochemical measurements

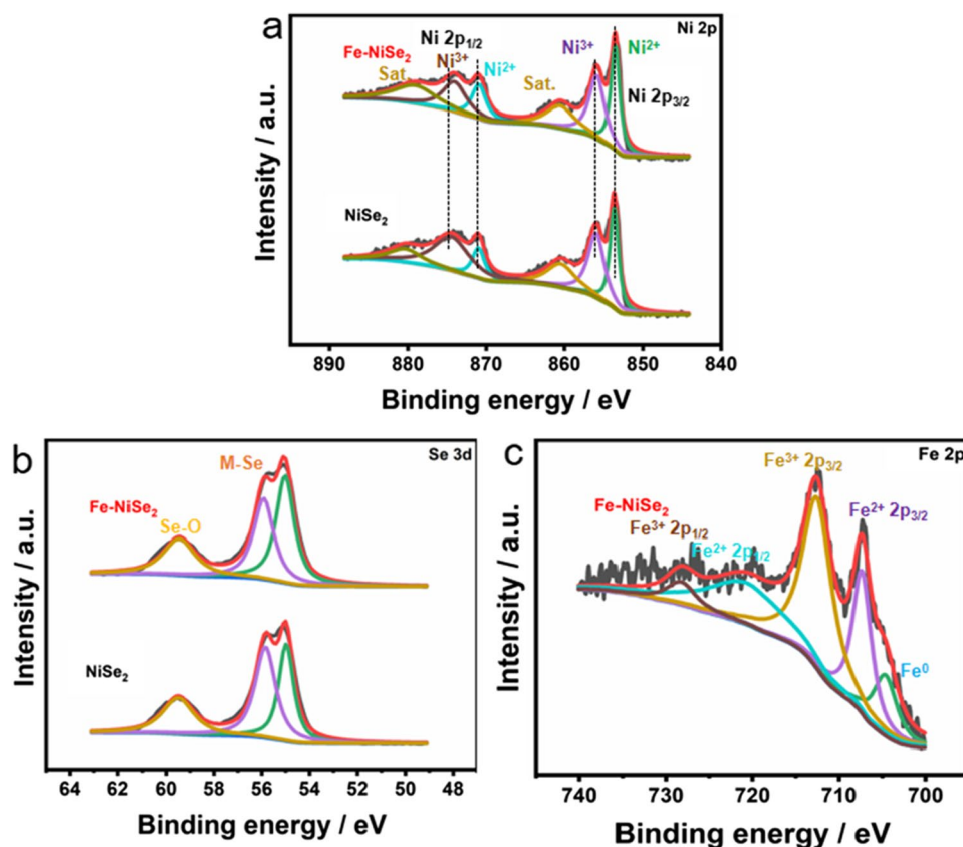
The standard three-electrode system, which equipped with modified glass carbon electrode, Ag/AgCl electrode, and carbon rod as working electrode, reference electrode, and counter electrode, respectively, was applied to evaluate their electrocatalytic

performance with a CHI-760E electrochemical workstation. Linear sweep voltammetry (LSV) was obtained at the scan rate of 5 mV·s⁻¹. Capacitance (*C_{dl}*) was calculated with assistance of cyclic voltammetry (CV) in a non-faradaic region at 10, 20, 50, 100, and 200 mV·s⁻¹, respectively. The electrochemical impedance spectroscopy (EIS) was performed from 1 Hz to 1000 kHz with a sinusoidal perturbation of 0.5 mA. The chronopotentiometry (CP) test was continuously carried out at a constant current density of 10 mA cm⁻².

Results and discussion

As illustrated in Fig. 1, the synthesis of Fe-NiSe₂ involved two steps, where the NiFe-MOF-74 can be synthesized via a simple solvothermal method by employing Ni(NO₃)₂·6H₂O

Fig. 3 The high-resolution XPS spectra of a Ni 2p, b Se 3d, and c Fe 2p



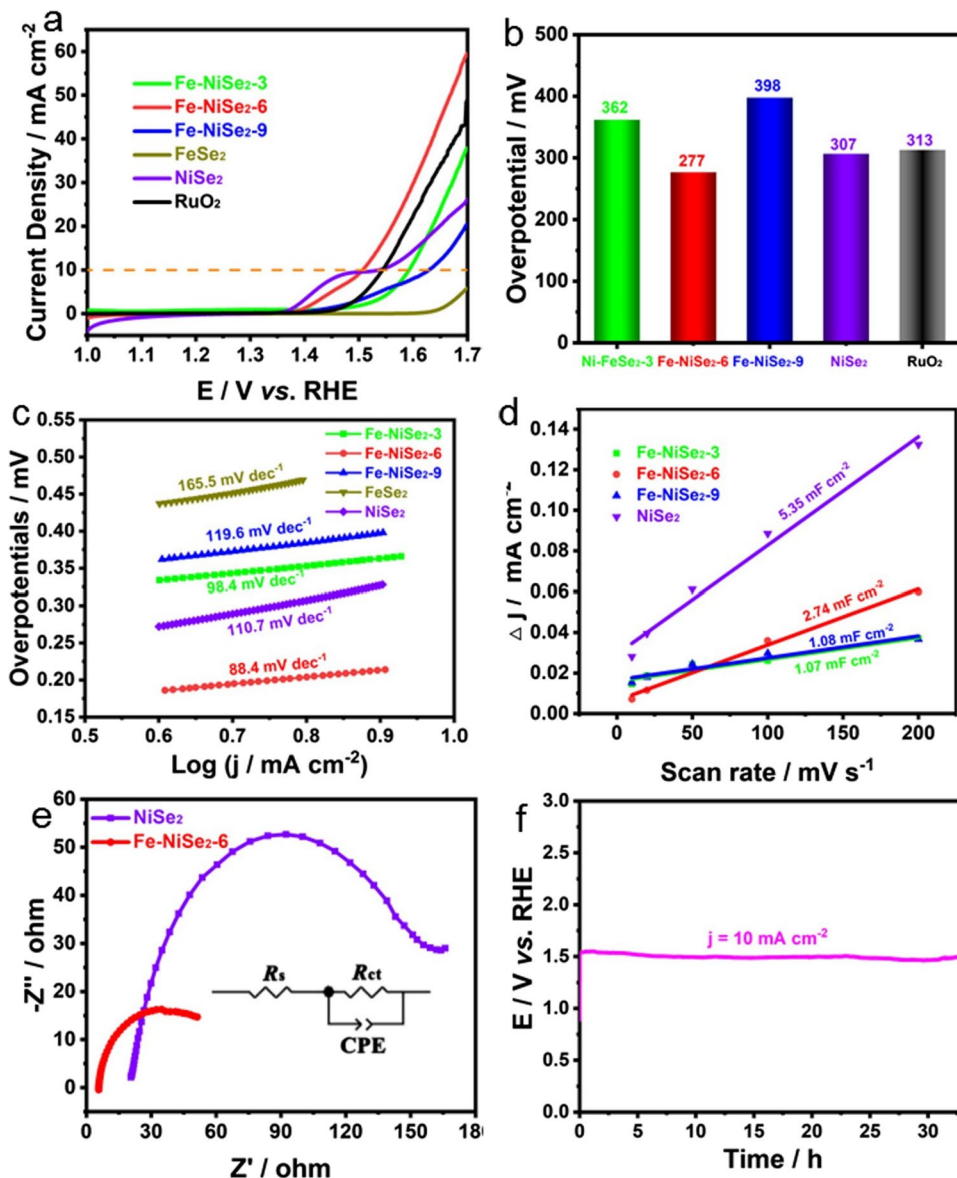
and $\text{Fe} \cdot (\text{C}_2\text{H}_3\text{O}_2)_2$ as precursors, DMF, and $\text{CH}_2\text{CH}_2\text{OH}$ as solvent. Interestingly, the morphology of the NiFe-MOF-74 is varied by changing the molar ratio of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and $\text{Fe} \cdot (\text{C}_2\text{H}_3\text{O}_2)_2$. As shown in Fig. S1a, the pristine Ni-MOF-74 is featured with typical sphere-like morphology. When varying the molar ratio of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and $\text{Fe} \cdot (\text{C}_2\text{H}_3\text{O}_2)_2$ from 3:1 to 6:1 and 9:1, the morphology is changed into spinning conical (Fig. S1b), hollow nanosphere (Fig. S1c), and nanoflower (Fig. S1d), respectively [32, 33]. Then, the Fe-NiSe_2 can be obtained via a simple hydrothermal method using NiFe-MOF-74 as precursors and SeO_2 as Se source.

To catch the morphological and structural information of the as-prepared Fe-NiSe_2 catalyst, a series of morphology and structure characterizations have also been conducted. The TEM image in the Fig. 2a implied that the as-prepared Fe-NiSe_2 are featured with porous properties, which will benefit for charge transfer efficiency and mass transport for electrocatalysis. The HRTEM image in Fig. 2b indicated the high crystallinity of Fe-NiSe_2 , in which the d -spacing of 0.292 nm can be indexed to the (200) facet of Fe-NiSe_2 , being consistent with the analysis of SAED pattern (Fig. 2c). The XRD peaks of Fe-NiSe_2 can be well indexed to the NiSe_2 (JCPDS# 41–1495) except for the slight shift due to the larger atomic radius of Fe, while some weak diffraction peaks can be assigned to the NiSe_2 (JCPDS# 21–0432)

(Fig. S2) [34]. Besides the morphology and structure, the chemical composition of the Fe-NiSe_2 is also thoroughly investigated. Figure 2d showed the EDS elemental mapping images of the Fe-NiSe_2 catalyst, where the Fe, Ni, and Se elements are homogeneously dispersed throughout the nanostructure, indicating the successful fabrication of Fe-NiSe_2 catalyst. The EDS spectrum (Fig. 2e) and line-scan profile (Fig. 2f) also suggested the successful doping of Fe into NiSe_2 , with a percentage of 3.44%. The abundant O element is observed in the EDS spectrum of Fe-NiSe_2 , maybe originated from the oxygen adsorbed on the surface of the material and oxygen contained in surface functional groups.

The valence state and compositions of the Fe-NiSe_2 were also analyzed by XPS by using NiSe_2 as a referenced sample. The survey spectra revealed the occurrence of C, Ni, Fe, and Se elements and trace amount of O from air exposure (Fig. S3). The Ni 2p XPS spectrum featured two twist circles of Ni $2p_{3/2}$ and Ni $2p_{1/2}$ connected by two associative satellite peaks (Fig. 3a). Both the peaks for Ni $2p_{3/2}$ and Ni $2p_{1/2}$ can be assigned to the Ni^{2+} and Ni^{3+} . Interestingly, when compared with NiSe_2 , the Ni 2p spectrum of Fe-NiSe_2 can display a slight shift to lower binding energy, suggesting that the dopant of trace amount of Fe can induce the charge transfer, which is indirectly indicating the Fe doping can modify the electronic structure of NiSe_2 [35, 36]. The Se 3d can also fitted to the Se^{2-} of $3d_{5/2}$ and $3d_{3/2}$, respectively (Fig. 3b), indicating the

Fig. 4 a LSV polarization curves of Fe-NiSe₂-3, Fe-NiSe₂-6, Fe-NiSe₂-9, FeSe₂, NiSe₂, and RuO₂. b Histograms of the overpotentials of different products at 10 mA cm⁻². c Tafel plots of different electrocatalysts. d Current density as a function of the scan rate for the different electrodes. e Nyquist plots of different products at 0.5 V. f The prolonged CP of Fe-NiSe₂ at a current density of 10 mA cm⁻²



formation of Ni-Se [37]. Furthermore, we also find the XPS spectra of Se before Fe doping is similar with the spectrum after Fe doping. For Fe 2p (Fig. 3c), the peaks appeared at the binding energies around 707.5 eV and 721.2 eV were compatible with Fe²⁺ 2p_{3/2} and Fe²⁺ 2p_{1/2}, respectively, while the other two peaks presented at the binding energies around 712.7 eV and 728.6 eV were well indexed to the Fe³⁺ 2p_{3/2} and Fe³⁺ 2p_{1/2}, respectively [38]. According to the XPS analysis, it is concluded that the negative shift of Ni 2p suggests the electron transfer from Fe to Ni, suggesting Fe doping can effectively tailor the electronic structure of nickel [39].

The electrocatalytic OER performance of the Fe-NiSe₂ was evaluated in 1 M KOH electrolyze by benchmarking

FeSe₂, NiSe₂, and RuO₂. For comparison, the Fe-NiSe₂ with different Ni/Fe ratios are also synthesized and evaluated. As shown in Fig. S4, both the Fe-NiSe₂-3 and Fe-NiSe₂-9 can also exhibit the coral morphology similar to Fe-NiSe₂-6, suggesting this method is universal for the synthesis of a series of Fe-NiSe₂ nanocatalysts with adjustable Ni/Fe atomic ratio. Figure 4a showed the LSV polarization curves of different electrocatalysts in 1 M KOH solution with a scan rate of 5 mV s⁻¹. As shown in Fig. 4a, it is clearly observed that the Fe-NiSe₂-6 can deliver remarkably higher current density and lower onset potential than other electrocatalysts. As seen from Fig. S5, an onset potential of merely 1.39 V is demanded for Fe-NiSe₂-6 to drive electrocatalytic water oxidation, being much smaller than

Fig. 5 a Schematically presenting the electrochemical overall water splitting based on Fe-NiSe₂-6||Pt/C couple. b LSV polarization curves of Fe-NiSe₂-6||Pt/C and RuO₂||Pt/C toward water electrolysis. c The long-time CP curve of Fe-NiSe₂-6||Pt/C couple at 10 mA cm⁻²

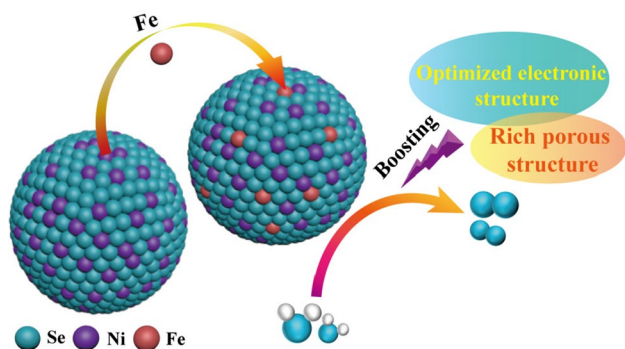
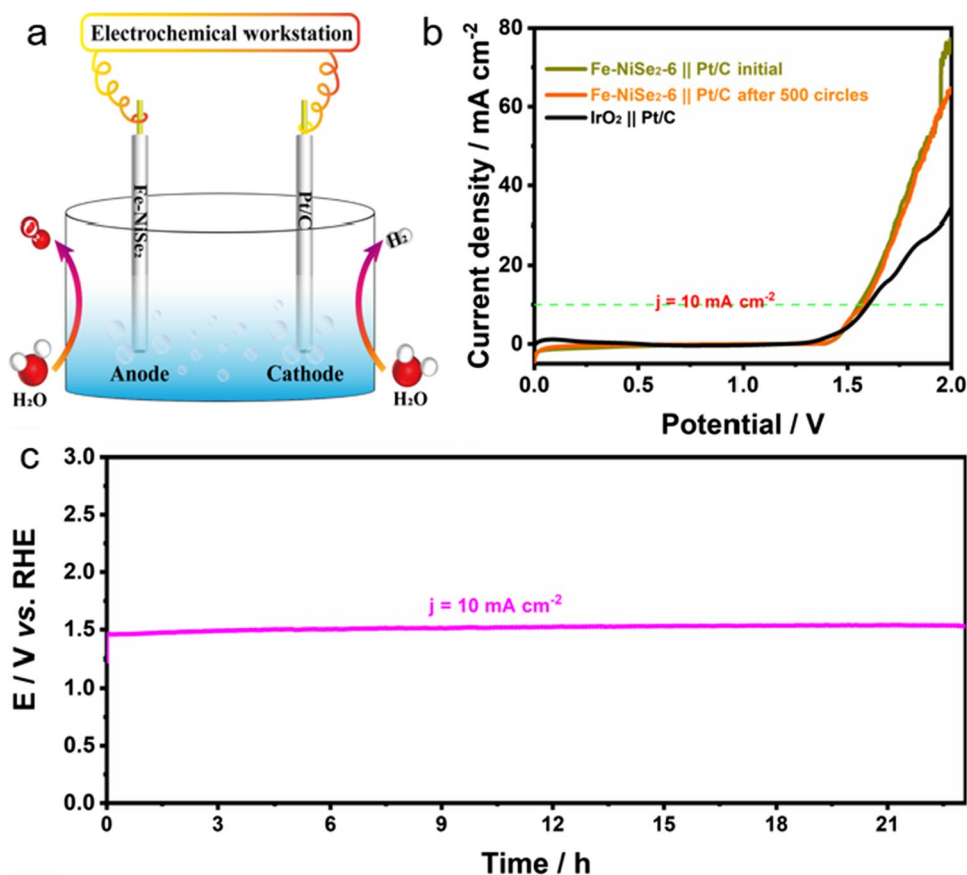


Fig. 6 Improving mechanism of the electrocatalytic OER performance of Fe-NiSe₂

other electrocatalysts. For systematically comparing the OER activity of different catalysts, a histogram of the overpotentials of different electrocatalysts at 10 mA cm⁻² is summarized in Fig. 4b. As shown in Fig. 4b, it is clearly observed that the Fe-NiSe₂-6 can archive 10 mA cm⁻² with the overpotential of only 277 mV, which is the smallest among these electrocatalysts investigated and surpass more reported Fe-NiSe₂ OER electrocatalysts [40]. In order to investigate the influence of the electronic structure optimization on the reaction

kinetics of OER, we have calculated the Tafel slope of different electrocatalysts. As displayed in Fig. 4c, the Tafel slope of Fe-NiSe₂-6 is 88.4 mV dec⁻¹, which is much smaller than that of FeSe₂ and NiSe₂. In addition, both the Fe-NiSe₂-3 and Fe-NiSe₂-9 can also exhibit much smaller Tafel slope than that of FeSe₂ and NiSe₂, further implying the positive influence of the electronic structure optimization on the acceleration of reaction kinetics [41]. The electrochemical active surface areas of these electrocatalysts are further evaluated by electrochemical double-layer capacitance (Fig. 4d). Herein, the capacitance of Fe-NiSe₂-6 is as high as 2.74 mF cm⁻², suggesting abundant electrochemically active sites are available for oxygen intermediates. To evaluate the electron transfer capability of Fe-NiSe₂-6, the electrochemical impedance spectra are also obtained, where the Fe-NiSe₂-6 shows a remarkably smaller impedance arc diameter than NiSe₂ (Fig. 4e), suggesting the high electrical conductivity and good electron transfer capability [42]. Besides the electrochemical activity, catalytic stability is also crucial for the practical application of electrocatalyst. To this end, we have thus carried out a chronopotentiometry (CP) test to evaluate the electrochemical stability. As shown in Fig. 4f, the Fe-NiSe₂-6 can run stably over 35 h at a current density of 10 mA cm⁻² without amplification in overpotential. The negligible variation in overpotential further confirm its superb electrochemical stability [43].

To investigate the potential for driving electrochemical overall water splitting, a two-electrode water electrolysis system equipped by Fe-NiSe₂ as anode and Pt/C as cathode is assembled (Fig. 5a). As shown in Fig. 5b, the Fe-NiSe₂-6 || Pt/C electrode exhibited obviously higher current density than that of the IrO₂ || Pt/C electrode, which requires a cell voltage of only 1.57 V to deliver a current density of 10 mA cm⁻². More importantly, the LSV polarization curve after 500 potential CV cycles is almost overlapped with the initial one, suggesting the high electrochemical durability for overall water splitting. More importantly, this couple can also exhibit superb voltage stability by maintaining a stable cell potential at the current density of 10 mA cm⁻² without obvious amplification for more than 25 h (Fig. 5c). These electrochemical tests have further indicated the great potential of Fe-NiSe₂-6 for the practical application in driving electrochemical water splitting (Fig. 6) [44].

Upon the combination of characterizations and electrochemical tests, it is concluded that the outstanding electrocatalytic OER performance of the Fe-NiSe₂ is primarily attributed to the electronic structure modification after Fe doping, which greatly optimizes the binding strength with oxygen intermediates [45]. Moreover, the porous structure also contributes to the substantial improvement in OER activity and stability [46, 47].

Conclusion

In summary, a MOF-derived strategy has been proposed for the fabrication of Fe-NiSe₂ with modified electronic structure and porous architecture. It is demonstrated that a trace amount of Fe doping can greatly modulate the electronic structure of NiSe₂, enabling them to possess an optimized binding strength with oxygen intermediates. Moreover, the porous structure can also expose more accessible active sites for intermediates. Benefitting from the modulated electronic structure and porous architecture, such Fe-NiSe₂ can exhibit superb catalytic OER performance with an overpotential of 277 mV at 10 mA cm⁻², and a Tafel slope of 46.3 mV dec⁻¹ in 1.0 M KOH, as well as remarkably high cycle stability. This work demonstrated the power of electronic structure engineering in designing advanced OER electrocatalysts with super catalytic activity, and it is highly anticipated that more work on electronic structure engineering of other potential materials may be stimulated.

Supplementary Information The online version contains supplementary material available at <https://doi.org/10.1007/s11581-022-04875-y>.

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