Metal Atom-Doped Co$_3$O$_4$ Hierarchical Nanoplates for Electrocatalytic Oxygen Evolution

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Electrocatalysts based on hierarchically structured and heteroatom-doped non-noble metal oxide materials are of great importance for efficient and low-cost electrochemical water splitting systems. Herein, the synthesis of a series of hierarchical hollow nanoparticles (NPs) composed of ultrathin Co$_3$O$_4$ nanosheets doped with 13 different metal atoms is reported. The synthesis involves a cooperative etching–coordination–reorganization approach starting from zeolitic imidazolate framework-67 (ZIF-67) NPs. First, metal atom decorated ZIF-67 NPs with unique cross-channels are formed through a Lewis acid etching and metal species coordination process. Afterward, the composite NPs are converted to hollow Co$_3$O$_4$ hierarchical NPs composed of ultrathin nanosheets through a solvothermal reaction, during which the guest metal species is doped into the octahedral sites of Co$_3$O$_4$. Density functional theory calculations suggest that doping of small amount of Fe atoms near the surface of Co$_3$O$_4$ can greatly enhance the electrocatalytic activity toward the oxygen evolution reaction (OER). Benefiting from the structural and compositional advantages, the obtained Fe-doped Co$_3$O$_4$ hierarchical NPs manifest superior electrocatalytic performance for OER with an overpotential of 262 mV at 10 mA cm$^{-2}$, a Tafel slope of 43 mV dec$^{-1}$, and excellent stability even at a high current density of 100 mA cm$^{-2}$ for 50 h.

The oxygen evolution reaction (OER) is of critical importance as the key electrocatalytic reaction in water splitting devices and rechargeable metal-air batteries.[1–7] Nevertheless, the multistep proton-coupled electron transfer processes involved in OER lead to the sluggish kinetics, which severely constrains the energy conversion efficiency.[8] To date, noble metal oxides, in particular RuO$_2$ and IrO$_2$, are demonstrated to be the most effective electrocatalysts for OER.[9] However, due to the scarcity and preciousness of the noble metal constituents, mass production of OER catalysts cannot be fulfilled unless replaced by other earth-abundant materials with comparable OER activity.[10–12] Transition metal oxides (TMOs), in particular Co$_3$O$_4$, which contains highly oxidized redox couples, have recently been considered as promising alternatives to noble metal oxides for efficient OER in alkaline media.[13–15] However, the activity of reported Co$_3$O$_4$-based catalysts can rarely be comparable to the high activity of noble metal oxides. Surface modulation is an efficient way to enhance the intrinsic activity by lowering the high driving force required by potential through tuning their surface structure and electronic properties.[16–18] Among various surface modulation strategies, the incorporation of a secondary metal atom into the lattice of TMOs has been demonstrated to remarkably improve their catalytic performance.[19–23] Therefore, rational introduction of isolated metal atoms into the crystal lattice of Co$_3$O$_4$ host is expected to promote its electrocatalytic OER activity.

In addition, design and construction of peculiar nanoarchitectures for electrocatalysts has been regarded as another efficient strategy for the enhancement of electrocatalytic performance by exposing more electrochemically active sites.[22] Particularly, hierarchical nanostructures can offer great benefits for the construction of advanced electrocatalysts because they combine the advantages of both low-dimensional building blocks and hierarchically porous structures.[23] Low-dimensional nano-sized building blocks, especially ultrathin nanosheets or nanoneedles, can not only expose abundant catalytically active sites, but also shorten the charge transport distance.[24] Moreover, hierarchically macro-/mesoporous structures are particularly beneficial for increasing the efficiency of mass transport, and thus promoting the formation and release of gas-phase catalytic products.[25,26] Based on these design rationales mentioned above, hollow nanostructures assembled by heteroatom-doped Co$_3$O$_4$ ultrathin nanosheets could serve as excellent catalysts for OER.

Metal-organic frameworks (MOFs) have become a rapidly expanding research area in the past two decades.[27–30] The most distinctive advantage of MOFs is their designable frameworks...
formed via modular self-assembly reactions of metal ions/clusters and organic ligands.\textsuperscript{31–33} Although their chemical instability often prevents them from being used directly as electrocatalysts for OER, it offers us many possibilities to chemically convert to various nanostructures, in which some advantages of MOFs can be well inherited.\textsuperscript{34,35} Here, we report a cooperative etching–coordination–reorganization strategy for the introduction and stabilization of isolated metal atoms in hierarchical Co$_3$O$_4$ hollow nanoplates (NPs) with cross-channels (Figure 1a). Novel NPs of Co-based zeolitic imidazolate framework-67 (ZIF-67) are employed as the precursors for the synthesis of metal atom doped Co$_3$O$_4$ electrocatalysts. By introducing the guest metal salt as a Lewis acid into the reaction system, the acid etching reaction induces the transformation of ZIF-67 from solid NPs to hollow NPs with unique cross-channels. Meanwhile, the guest metal species is adsorbed on the inner and outer surfaces of the hollow ZIF-67 NPs. After a solvothermal reaction, these hollow ZIF-67 NPs are transformed into hierarchical Co$_3$O$_4$ NPs assembled by ultrathin nanosheets. Moreover, the adsorbed metal atoms are successfully incorporated into the octahedral sites of Co$_3$O$_4$. Owing to the structural and compositional merits, the Fe-doped Co$_3$O$_4$ hierarchically hollow NPs (designated as Fe-Co$_3$O$_4$ HHNPs) exhibit superior OER performance with a small overpotential of 262 mV to reach the
current density of 10 mA cm\(^{-2}\), enhanced kinetics with a Tafel slope of 43 mV dec\(^{-1}\), and excellent stability at ultrahigh current densities, which are much better than that of Co\(_3\)O\(_4\) HHNPs and even commercial RuO\(_2\) catalysts in alkaline solution.

ZIF-67 NPs are first prepared through a surfactant-assisted hydrothermal reaction. Field-emission scanning electron microscopy (FESEM) and transmission electron microscopy (TEM) images reveal that these ZIF-67 NPs are uniform with an average side length of about 900 nm and thickness of about 160 nm (Figure 1b–d). After a Lewis acid etching treatment in 0.8 mg mL\(^{-1}\) FeCl\(_2\) ethanol solution for 10 min at room temperature, orthogonal channels are formed in ZIF-67 NPs. Figure 1e shows a typical FESEM image of the as-formed NPs with internal cross-channels. The average particle size does not change significantly during the etching process. TEM images confirm the existence of orthogonal channels in each NP (Figure 1f–g). Energy-dispersive X-ray (EDX) spectroscopy reveals that a small amount of Fe species is incorporated into the as-formed cross-channeled ZIF-67 NPs (thereafter denoted as Fe-ZIF-67), where the molar ratio of Fe to Co is about 1:22.9 (Figure S1, Supporting Information). Next, the as-prepared Fe-ZIF-67 NPs are refluxed in a mixed solvent of ethanol and water to gradually form Fe-Co\(_3\)O\(_4\) nanosheets. Finally, Fe-ZIF-67 NPs are completely transformed into Fe-Co\(_3\)O\(_4\) HHNPs with cross-channels. EDX analysis of the product shows the existence of Fe, Co, and O elements in the sample (Figure S2, Supporting Information). The disappearance of the N element verifies that the Fe-ZIF-67 sample is completely converted to Fe-Co\(_3\)O\(_4\) HHNPs after the solvothermal treatment. The low Fe/Co molar ratio of 1:30.6 suggests that the Fe atoms might be doped into the lattice of Co\(_3\)O\(_4\) in the form of isolated atoms. The content of Fe and Co is also revealed to be 3.1 and 70.2 wt% with an atomic ratio of 0.05 by inductively coupled plasma atomic emission spectroscopy (ICP-AES) measurement. Figure 1h shows an FESEM image of the as-derived Fe-Co\(_3\)O\(_4\) HHNPs at a low magnification. The sample retains its NP morphology, but the surface becomes very rough after the solvothermal treatment. The structure of the Fe-Co\(_3\)O\(_4\) HHNPs is further elucidated by TEM, showing the hierarchically porous structure with orthogonal channels and nanosheet subunits (Figure 1i,j). A closer examination of the nanosheet subunits reveals that the thickness of the nanosheets is only about 2 nm (Figure 1k).

N\(_2\) sorption measurements reveal that the Fe-Co\(_3\)O\(_4\) HHNPs possess a porous structure with a relatively high specific surface area of about 60 m\(^2\) g\(^{-1}\) (Figure S3, Supporting Information). High-resolution TEM (HRTEM) image (Figure 1l) shows the lattice fringes of the ultrathin nanosheets, which can be ascribed to the (311) planes of cubic spinel Co\(_3\)O\(_4\) (JCPDS card No. 42–1467). As revealed by high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) and elemental mapping results, Fe, Co, and O elements are evenly distributed throughout the Fe-Co\(_3\)O\(_4\) HHNP (Figure 1m).

To further investigate the impact of the etching–coordination process on ZIF-67 NPs, X-ray diffraction (XRD) and X-ray absorption fine structure (XAFS) spectroscopies are conducted to characterize the crystalline phase and coordination configurations of Co and Fe in Fe-ZIF-67 NPs. As shown in Figure 2a, XRD pattern of the Fe-ZIF-67 NPs is the same as that of ZIF-67 NPs, suggesting that the Fe species is doped into the ZIF-67 NPs. The existing forms of the metal elements are first revealed by

![Figure 2](image-url)

**Figure 2.** Phase and structural characterizations of Fe-ZIF-67 NPs and Fe-Co\(_3\)O\(_4\) HHNPs. a) XRD patterns of ZIF-67 NPs and Fe-ZIF-67 NPs. b) FT of EXAFS \(k^2\chi(k)\) spectra of Fe-ZIF-67 with Fe foil and ZIF-67 as the references. c) Normalized Fe K-edge (up), Co K-edge (bottom) XANES spectra of Fe-ZIF-67 with Fe foil, Co foil, and ZIF-67 as references. d) XRD patterns of Co\(_3\)O\(_4\) HHNPs and Fe-Co\(_3\)O\(_4\) HHNPs together with the standard pattern of Co\(_3\)O\(_4\). e) FT of EXAFS \(k^2\chi(k)\) spectra of Fe-Co\(_3\)O\(_4\) with Fe foil and Co\(_3\)O\(_4\) as references. f) Normalized Fe K-edge (up), Co K-edge (bottom) XANES spectra of Fe-Co\(_3\)O\(_4\) with Fe foil, Co foil, and Co\(_3\)O\(_4\) as the references.
extended X-ray absorption fine structure (EXAFS) analysis. In Figure 2b, the Fourier transform (FT) of Fe K-edge EXAFS of Fe-ZIF-67 shows a dominating peak of Fe-N at 1.53 Å, which overlays with the Co-N peak at 1.56 Å for the CoN₄ moiety, suggesting the incorporated Fe centers partially substitute for the Co sites in the CoN₄ imidazole tetrahedra.[36] The similar pattern in EXAFS further confirms that Fe atoms have taken the Co positions (Figure S4a, Supporting Information). The FT of EXAFS profile of Fe-ZIF-67 also exhibits minor signals located at 2.60 and 3.04 Å, which respectively arise from Fe–C and Fe–Fe from the second neighbor correlation.[24] The FT of Co K-edge EXAFS (Figure 2b) and the normalized X-ray absorption near-edge structure (XANES) spectra (Figure 2c) of Fe-ZIF-67 are observed in excellent accordance with unetched ZIF-67, confirming the good reservation of the original coordination structure of ZIF-67 after the etching–coordination treatments.

We next investigate the local structure of Fe and Co centers in Fe-Co₃O₄. Both XRD patterns of the obtained Co₃O₄ and Fe-Co₃O₄ products can be well indexed to the cubic spinel Co₃O₄ phase (JCPDS card No. 42–1467; Figure 2d). X-ray photoelectron spectroscopy (XPS) analysis is further carried out to study the surface chemical states of Fe-Co₃O₄ HHNPs. The survey spectrum (Figure S5a, Supporting Information) shows the presence of C, O, Co, and Fe, which is consistent with the EDX result. The peak-fitting analysis of Co 2p spectrum (Figure S5b, Supporting Information) suggests that there are two chemical states, corresponding to Co²⁺ at 781.6 and 797.3 eV and Co³⁺ at 780.4 and 796.0 eV.[37] The high-resolution O 1s spectrum (Figure S5c, Supporting Information) can be deconvoluted into two peaks, namely the Co–O peak at 530.4 eV and Co–O–H peak at 531.7 eV.[36] The Fe 2p spectrum (Figure S5d, Supporting Information) shows a pair of relatively weak peaks for Fe²⁺ at 711.7 and 724.9 eV, which also confirms the successful incorporation of Fe atoms.

The FT of Fe K-edge EXAFS spectrum of Fe-Co₃O₄ illustrates a dominating peak centered at 1.50 Å, which is assigned to the Fe–O bonding at the first shell (Figure 2e). In contrast to Fe foil, the Fe–Fe coordination peaks at 2.20 and 3.47 Å are absent in Fe-Co₃O₄, ruling out the existence of aggregated Fe clusters or particles. From EXAFS spectra (Figure S4b, Supporting Information) of Fe-Co₃O₄, the Fe K-edge exhibits similar oscillation shape to Co K-edge ranging from 3 to 10 Å⁻¹ by wavenumber (k), which suggests that Fe atoms have been successfully doped into the Co₃O₄ lattice. The disordered structure of Fe centers is verified by the lower magnitude of Fe K-edge than the Co K-edge. The main peak of the Fe K-edge has shifted to lower k compared with the Co K-edge in EXAFS spectra of Fe-Co₃O₄, implying that the Fe dopants mainly occupy the octahedral sites of high oxidation state Co(III), which has been demonstrated by the similar magnitude of Co EXAFS spectra of Fe-Co₃O₄ and Co foil.[38] The Co K-edge XANES profiles of Fe-Co₃O₄, Co₃O₄, and Co foil are also evaluated to study the local structure regarding Fe coordination in Co₃O₄. As shown in Figure 2f, the Co XANES spectrum of Fe-Co₃O₄ resembles that of Co₃O₄, indicating the similar valence of Co species in both samples.

To investigate the structural evolution process and conversion mechanism, the etching reaction is carried out in FeCl₂ ethanol solutions with different ion concentrations. When FeCl₂·4H₂O is added into ethanol, it will hydrolyze to generate the acidic environment for the etching process. When the ZIF-67 NPs (Figure 3a) are dispersed in 0.4 mg mL⁻¹ FeCl₂ ethanol solution for 10 min, the etching process begins selectively at the center of four side surfaces forming a thin cross in each NP (Figure 3b). When the concentration is increased to 0.8 mg mL⁻¹, the orthogonal channels are enlarged (Figure 3c). By increasing the FeCl₂ concentration to 1.2 then to 1.6 mg mL⁻¹, the orthogonal channels are further expanded (Figure 3d) until the nanostructure breaks down into four separate parts (Figure 3e). In addition, we have also investigated the transformation process from Fe-ZIF-67 NPs to Fe-Co₃O₄ HHNPs under the solvothermal treatment. As elucidated by the TEM images of the samples collected at different reaction stages, the hydrolysis process starts at

Figure 3. Synthetic etching–coordination and hydrolysis processes. a–e) TEM images of single ZIF-67 NP (a) and b–e) the corresponding products obtained by etching in 10 mL of FeCl₂·4H₂O ethanol solution with different concentrations for 10 min: b) 0.4 mg mL⁻¹, c) 0.8 mg mL⁻¹, d) 1.2 mg mL⁻¹, and e) 1.6 mg mL⁻¹. f–j) TEM images of the products obtained by hydrolysis of cross-channeled Fe-ZIF-67 NPs etched in 0.8 mg mL⁻¹ for different durations: f) 0 min, g) 3 min, h) 6 min, i) 9 min, and (j) 15 min.
the outer surface and channel walls of the Fe-ZIF-67 precursor simultaneously, and the Fe-ZIF-67 residue is gradually consumed and converted into hierarchical hollow NPs constructed from ultrathin Fe-Co3O4 nanosheets (Figure 3f). It is found that the etching process is largely ascribed to the Lewis acidity of FeCl2 salt. Control experiments are conducted by replacing the FeCl2 ethanol solution with a low concentration HCl solution. As expected, the exactly same etching processes can be observed, resulting in cross-channeled ZIF-67 NPs (Figure S6, Supporting Information). With this understanding, the present method can easily be extended for incorporation of many other metals into ZIF-67 NPs, ranging from III B to V A group in the periodic table, to generate metal-doped ZIF-67 (M-ZIF-67) NPs with cross-channels. During this process, the ZIF-67 NPs are steadily etched by protons released from the hydrolysis of metal cations, generating a cross-channeled structure for M-ZIF-67 NPs. Remarkably, similar nanostructures can be formed with the addition of 13 metal salts in total, which can be transformed to corresponding M-Co3O4 HHNPs after the solvothermal treatment. Figure 4 presents the overview of M-ZIF-67 NPs and corresponding M-Co3O4 HHNPs (M = Mn, Fe, Co, Ni, Cu, Zn, Zr, Nb, W, In, Sn, Bi, and La). TEM images of M-ZIF-67 NPs and M-Co3O4 HHNPs are displayed in Figure 4b. In particular, for the Co3O4 HHNPs decorated with six first row transition elements (M = Mn, Fe, Co, Ni, Cu, and Zn), electrocatalytic OER tests are carried out (Figure S7, Supporting Information). From the criteria of overpotential and Tafel slope, the Fe-Co3O4 electrocatalyst exhibits the best performance and thus is selected as the main sample for further detailed investigation of the effects of atomic doping and nanostructure engineering on the OER performance.

The electrocatalytic activity of Fe-Co3O4, Co3O4 and commercial RuO2 catalysts toward OER is investigated in alkaline solution (1.0 M KOH). Figure 5a shows the iR-compensated linear sweep voltammetry (LSV) curves. The Fe-Co3O4 catalyst requires an overpotential of 262 mV to reach the current density of 10 mA cm−2, which is 52 and 126 mV lower than that required for the Co3O4 and commercial RuO2 catalysts, respectively, to achieve the same current density. Moreover, the current density of the Fe-Co3O4 catalyst can reach 100 mA cm−2 at the overpotential of 303 mV, which is 120 mV lower than that of Co3O4 sample. In sharp contrast, the commercial RuO2 catalyst is not able to deliver a current density of 100 mA cm−2 in the applied potential range. The lowest overpotential and fastest current density rise of Fe-Co3O4 demonstrate the importance of heteroatom doping and nanostructure engineering for enhancing the electrocatalytic activity. The Tafel plots of these catalysts are investigated to get some insight into their OER kinetics. As can be seen in Figure 5b, the Fe-Co3O4 catalyst exhibits enhanced reaction kinetics with the lowest Tafel slope of 43 mV dec−1 compared with 72 mV dec−1 (for Co3O4) and 83 mV dec−1 (for RuO2), which is consistent with the fastest current density rise of Fe-Co3O4 in Figure 5a. Electrochemically active surface area (ECSA) of the catalysts is calculated based on the double-layer capacitance (Cdl). The cyclic voltamograms (CVs) are obtained in the voltage range of 1.18–1.28 V in which there are no redox processes. From the CVs, the Cdl is extracted by plotting (j−jR)/2 versus scan rate, where jR and j are the anode and cathode current densities at 1.23 V, respectively (Figure S8, Supporting Information). The Fe-Co3O4 catalyst exhibits the highest Cdl of 20.2 mF cm−2, which is much higher than that of Co3O4 (70 mF cm−2) and commercial RuO2 (1.7 mF cm−2) catalysts (Figure 5c). As expected, the Fe-Co3O4 catalyst possesses the largest ECSA of 504 cm2 (normalized per cm2 of electrode area), which is much higher than that of Co3O4 (175 cm2) and commercial RuO2 (42 cm2) (Figure 5d). Importantly, from the ECSA-normalized LSV curves (Figure 5d), the Fe-Co3O4 catalyst still exhibits the highest activity. To further elucidate the intrinsic mechanism for the enhancement of OER activity, per-site turnover frequency (TOF) is employed to compare the practical performance of catalysts. The TOF value is calculated based on the assumption that all metal atoms are active sites, and the number of metal atoms is obtained from ICP-AES results.[40] The corresponding potential-dependent TOF curves show the enhanced intrinsic activity per site in Fe-Co3O4 compared to Co3O4 and RuO2 (Figure 5e). We have also provided a bar graph of ECSA-normalized current densities and TOF values at an overpotential of 300 mV (Figure 5f). As can be seen, the Fe-Co3O4 catalyst has shown much higher current density and TOF value (0.033 mA cm−2; 0.0169 s−1) than Co3O4 (0.006 mA cm−2; 0.0016 s−1) and RuO2 (0.003 mA cm−2; 0.0003 s−1), suggesting that the Fe dopants significantly influence the coordination environment and symmetry of Co sites, thus greatly improve the intrinsic activity of active Co sites. Greatly increased TOF values by this heteroatom doping strategy are commonly observed in previous studies.[24,41] Clearly the enhanced electrocatalytic activity of Fe-Co3O4 can be mainly attributed to the Fe doping. This is also verified by electrochemical impedance spectroscopy (EIS) that the Fe-Co3O4 catalyst exhibits nearly identical charge transfer resistance as Co3O4 (Figure 5g).[42]

The long-time operational stability is a major challenge for the practical application of electrocatalysts, which is more severe under a large catalytic current density. As shown in the chronopotentiometry tests (Figure 5h), the potential of commercial RuO2 increases rapidly within 0.6 h even at a small current density of 10 mA cm−2, which could be attributed to the formation of soluble high oxidation state Ru species under highly anodic potential.[43] In contrast, benefiting from the high stability and reversibility of the spinel structure, the Fe-Co3O4 catalyst shows a negligible change of OER potential under the same constant current density after continuous operation for 50 h (Figure 5i), suggesting the superior structural and phase stability of the Fe-Co3O4 catalyst. We have also carried out water oxidation at higher current densities of 50 and 100 mA cm−2, where only insignificant potential rise is observed after 50 h (Figure 5i). The structural and compositional evolutions during the electrochemical tests are also investigated for further understanding of the high stability. The Fe-Co3O4 catalyst is harvested from the electrode after the stability test for 50 h at 10 mA cm−2. It is observed from FESEM and TEM images that the structure of HHNPs is well preserved (Figure S9, Supporting Information). Moreover, post-mortem XRD characterization shows that no additional crystalline phase is observed and the overall crystal structure remains as Co3O4 after the long-term OER test (Figure S10, Supporting Information). XPS Co 2p spectra show that the 2p1/2 and 2p3/2 peaks of Fe-Co3O4 after the stability test are shifted toward lower binding energy, suggesting partial surface oxidation of Co2+ to Co3+ (Figure S11, Supporting Information). HRTEM images
of Fe-Co₃O₄ before and after the stability test show that some crystalline Co₃O₄ on surface is oxidized to form a layer of amorphous high-valent oxyhydroxide (Figure S12, Supporting Information), which is consistent with results reported in literature.[44]

The electrocatalytic performance of these Fe-Co₃O₄ HHNPs in alkaline electrolyte is among the best when compared with numerous Co-based electrocatalysts (Table S1, Supporting Information). The enhanced OER performance is mainly attributed to the Fe doping of Co₃O₄ ultrathin nanosheets, as well as the hierarchical structure. Some preliminary density functional theory (DFT) calculations are performed to understand the effect of Fe doping on the enhanced electrocatalytic activity. For Co₃O₄ catalysts, the adsorption energy difference from the O* to OH* intermediates at the reactive sites is suggested to have dominant effect on the overall OER activity.[45,46] After incorporating isolated Fe atoms into the lattice of Co₃O₄, the DFT result shows that the adsorption energy difference from OH* to O* is significantly reduced from 1.90 to 1.54 eV (Figures S13 and S14, Supporting Information), suggesting the rate-determining step (RDS) is OH⁻ + HO* = O* + H₂O(l) + e⁻.

In view of the ultrathin (~2 nm) nature of these Fe-Co₃O₄ nanosheets, all doped Fe atoms should exist near the...
It is thus easy to understand that the doping of even small amount of Fe atoms will have profound enhancement in electrocatalytic activity as suggested by the DFT results.

In summary, hierarchical cross-channeled hollow NPs constructed with ultrathin Co₃O₄ nanosheets have been prepared by a facile etching−coordinated−reorganization method. Importantly, this method allows simultaneous incorporation of a secondary metal species. In total, 13 metal elements have been successfully doped into the ultrathin Co₃O₄ nanosheets in the form of isolated metal sites. As an example, the doping of Fe atoms in the octahedral sites of Co₃O₄ has greatly enhanced the intrinsic electroactivity of the obtained Fe-Co₃O₄ catalyst for the OER. The ultrathin nanosheet subunits and highly porous structure with cross-channels expose abundant active sites and facilitate the charge and mass transport. Significantly, the as-prepared Fe-Co₃O₄ electrocatalyst exhibits remarkable catalytic activity toward OER with an overpotential of 262 mV to reach the current density of 10 mA cm⁻², and shows excellent stability even at a high current density of 100 mA cm⁻² for 50 h.

Supporting Information
Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest
The authors declare no conflict of interest.
