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Water oxidation is key to the production of chemical fuels from electricity. Now, both the simulations and experimental characterizations illustrate that incorporating the hydration-effect-promoting metal cations into the Ni–Fe framework can improve water molecules adsorption functionality proximate to the active sites and the subsequent oxygen evolution reaction catalytic activity in pH-neutral electrolyte.
Hydration-Effect-Promoting Ni–Fe Oxyhydroxide Catalysts for Neutral Water Oxidation

Ning Wang, Zhen Cao, Xueli Zheng, Bo Zhang,* Sergey M. Kozlov, Peining Chen, Chengqin Zou, Xiangbin Kong, Yunzhou Wen, Min Liu, Yansong Zhou, Cao Thang Dinh, Lirong Zheng, Huisheng Peng, Ying Zhao, Luigi Cavallo, Xiaodan Zhang,* and Edward H. Sargent*

Oxygen evolution reaction (OER) catalysts that function efficiently in pH-neutral electrolyte are of interest for biohybrid fuel and chemical production. The low concentration of reactant in neutral electrolyte mandates that OER catalysts provide both the water adsorption and dissociation steps. Here it is shown, using density functional theory simulations, that the addition of hydrated metal cations into a Ni–Fe framework contributes water adsorption functionality proximate to the active sites. Hydration-effect-promoting (HEP) metal cations such as Mg$^{2+}$ and hydration-effect-limiting Ba$^{2+}$ into Ni–Fe frameworks using a room-temperature sol–gel process are incorporated. The Ni–Fe–Mg catalysts exhibit an overpotential of 310 mV at 10 mA cm$^{-2}$ in pH-neutral electrolytes and thus outperform iridium oxide (IrO$_2$) electrocatalyst by a margin of 40 mV. The catalysts are stable over 900 h of continuous operation. Experimental studies and computational simulations reveal that HEP catalysts favor the molecular adsorption of water and its dissociation in pH-neutral electrolyte, indicating a strategy to enhance OER catalytic activity.

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is several orders of magnitude lower than in the alkaline electrolyte.\cite{31} OER in pH-neutral electrolyte therefore requires additional water adsorption and dissociation processes to provide the adsorbed water molecules (H$_2$O$^+$) and the OER steps under neutral-pH conditions.\cite{32}

\[
\begin{align*}
\text{H}_2\text{O}^+ & \rightarrow \text{OH}^- + \text{H}^+ + e^- \quad (1) \\
\text{OH}^- & \rightarrow \text{O}^- + \text{H}^+ + e^- \quad (2) \\
\text{O}^- + \text{H}_2\text{O}^- & \rightarrow \text{OOH}^- + \text{H}^+ + e^- \quad (3) \\
\text{OOH}^- & \rightarrow \cdot \text{O}_2 + \text{H}^+ + e^- \quad (4)
\end{align*}
\]

Catalysts including iridium oxide (IrO$_2$)\cite{13} as well as Co-based,\cite{14} Ni-based,\cite{15,16} and Mn-based\cite{17} materials, have each shown recent progress in OER performance in pH-neutral electrolytes. Nevertheless, these catalysts have yet to fulfill the requirement of sub-350 mV overpotentials and prolonged durability (2500 h typically for initial stability studies).\cite{18}

We took the view that efficiently capturing and dissociating water molecules at the catalyst/water interface could improve OER performance in pH-neutral electrolytes. Prior studies have shown that noncovalent interactions between hydrated metal cations (M$^{(n)}$) and $\cdot$OH form OH$_{ad}$M$^{(n)}$(H$_2$O)$_x$ and enhance the adsorption of OH$^-$ at the catalyst/electrolyte interface. These methods can accelerate the oxygen reduction reaction.\cite{19-24}

Mg$^{2+}$ is an example of a metal that can support another beneficial strategy: it possesses a high hydration energy that offers the prospect of enhancing the bond strength between the catalyst surface and molecular water.\cite{25} We hypothesized that introducing ions of this kind into the catalyst framework could lead to a higher hydration level of the system, improving thereby the OER rate.

Theoretical studies of the hydration effect on Ni–Fe based catalyst. Using the Ni–Fe system as a prototypical framework,\cite{25,26} we investigated the effect of introducing metal cations with strong hydration capacity and small atomic diameters. We focus on whether water adsorption onto the surface of Ni–Fe catalyst can be enhanced.

We first studied the effect of Mg$^{2+}$ (a relatively small size cation) and compared it with Ba$^{2+}$ (a relatively large size cation) doping (Figure 1). In each case we examined the water adsorption properties of the doped Ni–Fe catalyst.

Starting from a stable metal oxyhydroxide model, we constructed the slab $\beta$-NiOOH as the catalyst framework (Figure S1, Supporting Information).\cite{27,28} Mg atoms remain at the catalyst surface, and are directly exposed to the aqueous solution during OER (Figure S2, Supporting Information).

We then considered the immersion of this catalyst into aqueous solution: we assumed that two (0T5) facets were exposed to interfacial water. One of the two (0T5) facets was modified to replace half of the surface Ni$^{2+}$ with the relatively small Mg$^{2+}$ or the larger Ba$^{2+}$ cations (Figure 1a,d), while the other facets of the catalyst were kept the same as controls. Water adsorption on each catalyst was investigated using molecular dynamic simulations in the canonical ensemble at 300 K (details in the Supporting Information). We accumulated a 12 ps trajectory and recorded the final 10 ps trajectory to produce a map of the water-catalyst distance under dynamic conditions. Specifically, we report \( \left( \frac{1}{d^2} \right)^{1/2} \exp \left( -\frac{\|\mathbf{r}_i - \mathbf{r}_a\|}{\xi} \right) \), where \( d \) is the shortest distance between the interfacial water oxygen and any metal ion at slab surface, \( x_a \) is the \( x-y \) coordinate of this water oxygen, \( a_i \) is the grid on the surface, and \( \xi = 1.0 \) Å determines the resolution of the map (Figure 1b,c,e,f).

In the absence of the Mg$^{2+}$ cation, the surface OH terminal groups of the Ni–Fe catalyst determine the interfacial water
distribution via hydrogen-bonding interactions. When surface Mg$^2+$ is added, it attracts interfacial water molecules, leading to a shorter distance between the oxygen atom in water and the metal ions (red patches in Figure 1c). These interfacial water molecules no longer reflect the structure of surface OH terminal groups. By contrast, when Ba$^{2+}$ is introduced, the water-catalyst distance is dominated by the hydrogen-bonding network (Figure 1f).

Using this simple model, we are able to suggest a trend: pursuing experimental doping using cations having a high hydration capacity could potentially offer more water molecules for dissociation reactions.

Synthesis and OER activity of hydration-effect catalysts. We synthesized hydration-effect-promoting (HEP) oxyhydroxides and hydration-effect-limiting (HEL) Ni–Fe–Ba oxyhydroxides. We used a room-temperature sol–gel process previously reported.[29] Energy-dispersive X-ray spectroscopy mapping shows a uniform distribution of Ni, Fe, and Mg (Figure S3, Supporting Information). From inductively coupled plasma optical emission spectra, we determined the molar ratio of Ni:Fe:Mg to be 7:1:0.2 (Figure S4, Supporting Information). The catalysts present a porous structure (Figure S5, Supporting Information) without evidence of crystallinity (Figure S6, Supporting Information).

We evaluated catalytic properties using a three-electrode electrochemical cell containing CO$_2$-saturated 0.5 m KHCO$_3$ electrolyte. Compared with the HEL catalyst, the HEP exhibited a significant improvement in catalytic activity on glass carbon electrode (GCE). Linear sweep voltammetry (LSV) polarization measurements indicate that optimized Ni–Fe–Mg requires only 514 mV overpotential to reach 10 mA cm$^{-2}$ on GCE, which outperforms the best oxide catalysts previously reported (Figure 2a; Table S1 and Figure S7, Supporting Information). The decreased Tafel slope (210 mV dec$^{-1}$→150 mV dec$^{-1}$, Table S2, Supporting Information) indicates a higher OER reaction rate when Mg$^{2+}$ is incorporated (Figure 2b). Charge-transfer resistance ($R_{ct}$) determined using electrochemical impedance spectroscopy (Figure 2c), shows that incorporating Mg$^{2+}$ decreases the $R_{ct}$ (580 → 480 $\Omega$) and gives rise to faster electrode kinetics. The Ni is in an oxidation state higher than 3$^+$ as seen in previous reports (Figure S8, Supporting Information).[30,31]

Next, we investigated further electrochemical behaviors of the best catalysts compared to controls in order to characterize intrinsic activity. First, we normalized the current density using the electrochemically active surface area (ECSA) obtained using the double-layer capacitance ($C_{dl}$) technique.[32,33] The ECSA-normalized current density of the HEP catalyst is fully 1.3-times and 1.8-times higher than those of reference Ni–Fe catalyst and HEL Ni–Fe–Ba catalyst, respectively, at 1.7 V versus RHE (Figure S9, Table S3, Supporting Information). The activation energy (Figure 2d) and turnover frequency $^{[29,31,34–36]}$.

![Figure 2](https://www.advancedsciencenews.com/doi/abs/10.1002/adma.201906806)

**Figure 2.** Performance of Ni–Fe–Mg catalyst and controls in a three-electrode configuration in CO$_2$ saturated 0.5 m KHCO$_3$ aqueous electrolyte. a) OER polarization curves for catalysts loaded on GCE. The data were obtained using 1 mV s$^{-1}$ scan rate and are presented without iR correction (catalyst loading 0.21 mg cm$^{-2}$). b) Tafel plot of catalysts loaded on GCE. c) Electrochemical impedance spectroscopy (EIS) data for Ni–Fe–Mg catalysts and controls in three-electrode configuration in CO$_2$ saturated 0.5 m KHCO$_3$ aqueous electrolyte. The data were collected for the electrodes under 1.6 V versus RHE. The inset provides the equivalent circuit: $R_s$; series resistance; $R_{ct}$; charge-transfer resistance; CPE; constant-phase element related to the double-layer capacitance. d) Activation energy of samples on Ni foam obtained from the Arrhenius relationship. e) The OER polarization curves of Ni–Fe–Mg catalysts and controls loaded on Au-coated Ni foam in a three-electrode configuration in CO$_2$ saturated 0.5 m KHCO$_3$ aqueous electrolyte. f) Chronopotentiometric curves obtained from the Ni–Fe–Mg catalyst on Ni foam electrode with constant current densities of 10 mA cm$^{-2}$, and the corresponding Faradaic efficiency from gas chromatography measurement of evolved O$_2$. 

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Figure 3. OER polarization curves of Ni–Fe–Mg catalyst and relevant controls. The materials were loaded on GCE in (a) 0.5 M KHCO₃ (pH = 8.5) and (b) 1 M KOH electrolyte (pH = 14). (c) The corresponding Tafel curves of Ni–Fe–Mg catalyst in 0.5 M KHCO₃ (pH = 8.5) and 0.5 M KOH electrolyte (pH = 14). (d) Arrhenius plot of the inverse temperature versus the log of the exchange current for Ni–Fe–Mg and Ni–Fe–Ba catalyst in electrolyte with H₂O and D₂O.

We characterized the operating stability of the HEP catalyst at 10 mA cm⁻² and found that it retained its overpotential to within 10 mV following 900 h of continuous water splitting operation (Figure 2f). The Faradaic efficiency for oxygen production remained at 98% ± 2% throughout.

Investigations of the HEP effect. To explore further the relationship between the hydration effect and catalytic activity, we examined the role of Mg and Ba in water adsorption and water dissociation. When we increase pH, the catalytic advantage from HEP Ni–Fe–Mg is reduced, consistent with the abundance of OH⁻ at high pH (Table S7, Supporting Information; Figure 3a,b), and also in accordance with the viewpoint that alkaline earth cations cannot improve the alkaline catalytic performance. [37,38] The Tafel slope decreased from 150 mV dec⁻¹ (pH = 7.2) to 120 mV dec⁻¹ (pH = 8.5) and 80 mV dec⁻¹ (pH = 14, Figure 3c), which again supports the view that the HEP advantage is occurred in neutral electrolyte. [39–41]

We then characterized the activation energy (Eₐ) for HEP and HEL catalysts in H₂O and D₂O to evaluate the role of OH/OD bond breaking throughout the OER process based. We leveraged the kinetic isotope effect in these studies. We found that reactions in D₂O present a larger activation energy compared to those in H₂O (Eₐ(D₂O) = 1.4 Eₐ(H₂O), Figure 3d). This difference in Eₐ indicates that OH (or OD) bond breaking is the rate-limiting step in the neutral OER process. [42]

We next proceeded to characterize the effect of HEP/HEL metal cations on water adsorption. Fourier transform infrared (FTIR) spectroscopy and X-ray photoelectron spectroscopy...
In summary, a series of Ni–Fe based catalysts were prepared via a sol–gel method at room temperature, and then used as electrocatalysts for OER in neutral electrolyte. By combining electrochemical characterization with density functional theory (DFT) studies, we found that incorporating hydration effect metal cations into a Ni–Fe framework—namely, the Ni–Fe–Mg catalysts—influences water adsorption and enhances OER performance in neutral electrolytes. Specifically, OER activities on the RHE scale for Ni–Fe based catalysts are increased when we decrease the metal–oxygen distance, consistent with the view that the hydration-effect-promoting water adsorption triggers more favorable OER reaction pathways. In the future, the hydration-effect-promoting phenomenon can be extended to provide enhanced water adsorption for other electrochemical reactions, such as in CO2 reduction to hydrocarbons, N2 reduction to ammonia, and other reactions involving water molecules.

Experimental Section

See the details in the Supporting Information.

Supporting Information

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

hydration effect, neutral electrolyte, Ni–Fe, oxygen evolution reaction

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