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## Interfacial electric field in Zr-MOF-grafted carbon nitride to boost H<sub>2</sub>O<sub>2</sub> piezo-synthesis under ambient conditions

 Jingyi Yang,<sup>a</sup> Zhi Li,<sup>ib</sup> \*<sup>ac</sup> Huiru Chen,<sup>a</sup> Jie Leng,<sup>a</sup> Li Gong\*<sup>b</sup> and Mingshan Zhu<sup>ib</sup> \*<sup>ac</sup>

**Harnessing interfacial electric fields offers a promising route to boost piezocatalytic efficiency. Here, we grafted Zr-MOF onto carbon nitride to construct an interfacial electric field, leading to a hydrogen peroxide production rate of 4.76 mmol g<sup>-1</sup> h<sup>-1</sup> via water oxidation and the oxygen reduction reaction.**

Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) has emerged as a versatile and environmentally benign oxidant with wide-ranging applications in energy storage, water treatment, and green synthesis.<sup>1</sup> Traditional H<sub>2</sub>O<sub>2</sub> production relies on the energy-intensive anthraquinone process, which suffers from high carbon emissions and complex infrastructure.<sup>2</sup> The direct synthesis of H<sub>2</sub>O<sub>2</sub> from oxygen and water offers a sustainable alternative, but conventional thermostatic approaches often require noble metal catalysts and hazardous conditions.<sup>3</sup> In this context, piezocatalysis, an emerging technology that converts mechanical energy into chemical energy, holds great promise for sustainable H<sub>2</sub>O<sub>2</sub> production.<sup>4,5</sup> Its mechanism relies on stress-induced charge separation within piezoelectric materials, which initiates redox reactions between O<sub>2</sub> and H<sub>2</sub>O to generate H<sub>2</sub>O<sub>2</sub>. However, electrons and holes produced under piezoelectric excitation rapidly recombine, limiting the catalytic efficiency.<sup>6</sup> Therefore, promoting effective charge separation remains a key challenge for advancing piezocatalytic systems.<sup>7–9</sup>

To overcome such rapid recombination of charge carriers, previous works have mainly focused on strategies such as elemental doping<sup>10</sup> and introducing defect sites<sup>11</sup> to improve charge-carrier-separation efficiency.<sup>12</sup> In recent years, constructing an internal electric field (IEF) has been recognized

as an effective method for improving the reactivity and selectivity of catalytic reactions in the energy and environmental fields.<sup>13</sup> Zhang *et al.* prepared a step-scheme heterojunction ZnO/COF(TpPa-Cl) using a simple electrostatic self-assembly method and studied its photocatalytic H<sub>2</sub>O<sub>2</sub> generation performance.<sup>14</sup> However, research on using the IEF for enhancing charge carrier separation in piezocatalysis remains relatively scarce.

Carbon nitride (C<sub>3</sub>N<sub>4</sub>) was first reported in 2019 to have piezoelectrically catalyzed hydrogen-peroxide-producing activity, attributed to its excellent piezoelectric properties.<sup>15–17</sup> However, the poor charge-carrier-separation ability of pure C<sub>3</sub>N<sub>4</sub> limits the efficiency of its piezoelectric-catalyzed synthesis of H<sub>2</sub>O<sub>2</sub>, which may be due to its fast carrier complexation.<sup>18–21</sup> To address this critical issue, we constructed the IEF to efficiently promote piezoelectric carrier separation by grafting MOFs<sup>22,23</sup> onto the carbon nitride surface taking advantage of their tunable electronic structures and catalytic sites.<sup>24,25</sup>

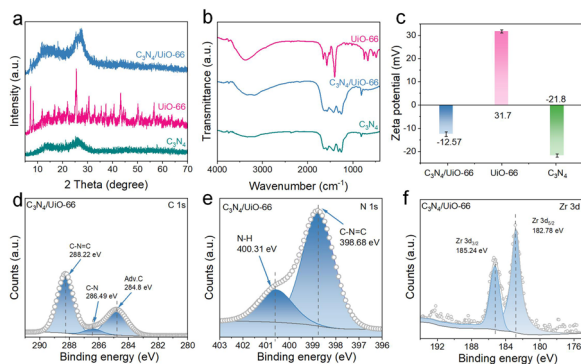
We integrated C<sub>3</sub>N<sub>4</sub> with UiO-66 through electrostatic self-assembly to create a composite exhibiting enhanced interfacial charge modulation. The optimized C<sub>3</sub>N<sub>4</sub>/UiO-66 system demonstrated exceptional piezocatalytic performance, achieving an H<sub>2</sub>O<sub>2</sub> production rate of 4.76 mmol g<sup>-1</sup> h<sup>-1</sup> under ambient conditions, representing 12.1-fold and 11.5-fold improvements over pristine C<sub>3</sub>N<sub>4</sub> and UiO-66, respectively. Electron paramagnetic resonance (EPR) spectra showed the simultaneous presence of \*O<sub>2</sub><sup>-</sup> and \*OH radical species, indicating the cooperative use of oxygen reduction (ORR) and water oxidation (WOR) pathways. The IEF intensity of the C<sub>3</sub>N<sub>4</sub>/MOF was assessed by taking zeta potential measurements, and performing open-circuit voltage tests, and was found to be 1.17 times higher than that of pristine C<sub>3</sub>N<sub>4</sub>. This work has provided a generalizable strategy for developing advanced piezocatalytic WOR materials.

C<sub>3</sub>N<sub>4</sub>/UiO-66 composites were prepared *via* electrostatic self-assembly.<sup>14</sup> Transmission electron microscopy (TEM) showed that UiO-66 indeed grafted onto the C<sub>3</sub>N<sub>4</sub> surface (Fig. S1–S3) and XRD analysis revealed a shift in the (002) diffraction peak

<sup>a</sup> Guangdong Engineering Technology Research Center of Water Treatment Processes and Materials, College of Environment and Climate, Jinan University, Guangzhou 511443, China. E-mail: zhili@jnu.edu.cn, zhumingshan@jnu.edu.cn

<sup>b</sup> Instrumental Analysis and Research Center, Sun Yat-Sen University, Guangzhou, 510275, China. E-mail: gongli2@mail.sysu.edu.cn

<sup>c</sup> Guangdong Provincial Key Laboratory of Spine and Spinal Cord Reconstruction, The Fifth Affiliated Hospital of Jinan University (Heyuan Shenhe People's Hospital), Heyuan 517465, China



**Fig. 1** (a) XRD patterns, (b) FT-IR spectra, and (c) zeta potential values of  $C_3N_4$ /UiO-66, UiO-66 and  $C_3N_4$ . (d) C 1s, (e) N 1s and (f) Zr 3d regions of the XPS spectrum of  $C_3N_4$ /UiO-66.

from  $25.97^\circ$  in  $C_3N_4$  to  $27.3^\circ$  in the composite, indicating a reduced interlayer spacing (Fig. 1a). A shifted (100) peak at  $12^\circ$  and the retained (111) peak of UiO-66 at  $7.2^\circ$  suggested strong interfacial interactions and maintained structural integrity.<sup>26</sup> Fourier-transform infrared spectroscopy (FT-IR) (Fig. 1b) showed characteristic peaks of triazine units at  $810\text{ cm}^{-1}$  and  $1200\text{--}1700\text{ cm}^{-1}$  for both  $C_3N_4$  and the composite, with no evident  $\text{COO}^-$  signals, indicating a predominant  $C_3N_4$  framework.<sup>27</sup> Zeta potential analysis (Fig. 1c) confirmed the occurrence of electrostatic attraction between negatively charged  $C_3N_4$  ( $-21.8\text{ mV}$ ) and positively charged UiO-66 ( $+31.7\text{ mV}$ ), promoting a stable composite formation, and demonstrating their spontaneous electrostatic self-assembly.<sup>14</sup>

X-ray photoelectron spectroscopy (XPS) analysis further confirmed the formation of a strong electronic interface between  $C_3N_4$  and UiO-66. Compare to the C 1s region of the spectrum (Fig. 1d and Fig. S4a and S5a), the composite yielded an intensified peak at  $288.22\text{ eV}$ , attributed to C–N–Zr bonding, along with a shift in the C–N bond energy ( $286.49\text{ eV}$ ), indicating interfacial charge redistribution. The N 1s region of the spectrum of the composite revealed an increased binding energy for N–H ( $400.31\text{ eV}$ ) and decreased binding energy for C=N–C ( $398.68\text{ eV}$ ) compared to those of pure  $C_3N_4$ , as shown in Fig. 1e and Fig. S4b and S5b, suggesting coordination between nitrogen atoms and Zr–O clusters.<sup>28,29</sup> These changes, together with the preserved pyrrole-N peak ( $401.35\text{ eV}$ ), confirmed the structural integrity of the  $C_3N_4$  framework and a modified electronic environment.<sup>30</sup> Compare to the Zr 3d region of the spectrum of the composite (Fig. 1f and Fig. S4c and S5c), a slight downshift in Zr  $3d_{5/2}$  ( $182.78\text{ eV}$ ) and upshift in Zr  $3d_{3/2}$  ( $185.24\text{ eV}$ ) were observed, and further indicated electronic interactions at the interface, leading to increased electron density around Zr sites.<sup>31,32</sup> These observations highlight the formation of a well-defined metal–semiconductor interface, which facilitates charge separation and enhances piezocatalytic activity.<sup>33</sup>

The piezocatalytic performance of the  $C_3N_4$ /UiO-66 composite showed a significant enhancement in  $\text{H}_2\text{O}_2$  generation efficiency, demonstrating a production rate substantially surpassing those observed for the individual  $C_3N_4$  and UiO-66 components under identical test conditions (Fig. 2a). As shown



**Fig. 2** (a) Curves for piezoelectric  $\text{H}_2\text{O}_2$  production in the presence of, respectively,  $C_3N_4$ /UiO-66, UiO-66 and  $C_3N_4$ . Experimental conditions: catalyst ( $0.1\text{ g L}^{-1}$ ) under ultrasound (Us),  $T = 25^\circ\text{C}$ . (b) Piezoelectric  $\text{H}_2\text{O}_2$  production for 6 h. (c) and (d)  $\text{H}_2\text{O}_2$  production in the presence of  $C_3N_4$ /UiO-66 (c) recycled every hour and (d) in different real water samples under Us. (e) Curves for  $\text{O}_2$ -TPD of  $C_3N_4$ /UiO-66, UiO-66 and  $C_3N_4$ . (f) Phase hysteresis loop and amplitude butterfly loop when using  $C_3N_4$ /UiO-66. (g) Nyquist plots with the fitted equivalent circuit based on EIS simulation. (h) Transient current density time curves over time under periodic Us signals and (i) LSV for  $C_3N_4$ /UiO-66, UiO-66 and  $C_3N_4$ .

in Fig. 2b and c, the composite demonstrated remarkable stability, sustaining continuous operation over 6 h without significant activity loss and retaining over 90% of its initial performance after six cycles. Furthermore, the composite maintained good  $\text{H}_2\text{O}_2$ -generation capability in real water matrices such as tap water, seawater, river water, and rainwater, with only slight reductions in yield compared to pure water (Fig. 2d), primarily due to the presence of background organic and inorganic constituents that may have scavenged radicals or competed for active sites, highlighting its potential for practical applications. Mechanistic studies indicated the enhanced performance to be due to improved  $\text{O}_2$  activation and interfacial charge dynamics. The obtained  $\text{O}_2$  temperature-programmed desorption ( $\text{O}_2$ -TPD) results (Fig. 2e) revealed a significantly higher capacity of the composite than of the components to adsorb oxygen, while piezoresponse force microscopy (PFM) yielded a pronounced butterfly-shaped amplitude loop with a peak amplitude of  $182.04\text{ pm}$  (Fig. 2f), confirming strong piezoelectric activity. Electrochemical impedance spectroscopy showed the composite having displayed the lowest charge-transfer resistance (Fig. 2g),<sup>34</sup> and piezoelectric current measurements indicated it to have the highest response (Fig. 2h) among all the samples.<sup>35</sup> Linear sweep voltammetry (LSV) further demonstrated a strong current response in the  $0.7\text{--}1\text{ V}$  range (Fig. 2i). Consistent with these results, the open-circuit voltage (OCV) of  $C_3N_4$ /UiO-66 ( $0.347\text{ V}$ ) was significantly higher than those of the individual components ( $0.112$  and  $0.166\text{ V}$ ) (Fig. S6), confirming a stronger charge-separation driving force. To further evaluate the performances of the three materials, their IEF strengths were quantified using the Gouy–Chapman formula (Eqn. S1–S5).<sup>36</sup> The quantitative analysis demonstrated an IEF strength 1.17-times

higher for the  $C_3N_4$ /UiO-66 composite than for  $C_3N_4$  (Fig. S7)—with the strong IEF enhancing charge separation, as further evidenced by the poor performance of physically mixed samples (Fig. S8). These findings collectively confirmed that interface engineering can significantly enhance charge separation and transport, enabling efficient piezocatalytic  $H_2O_2$  generation.

Electron paramagnetic resonance (EPR) spectroscopy was employed to investigate the surface electronic states of the catalysts. As shown in Fig. 3a, all samples exhibited a Lorentzian signal centered at  $g = 2.003$ , corresponding to unpaired, non-localized electrons in the heptazine ring structure of  $C_3N_4$ .<sup>37</sup> Notably, the  $C_3N_4$ /UiO-66 composite showed a stronger signal intensity, which was not simply the sum of the individual contributions from  $C_3N_4$  and UiO-66, indicating a higher content of localized unpaired electrons. This enhancement was attributed to interfacial ion-exchange interactions that promoted charge separation, thereby increasing the population of unpaired electrons and enhancing catalytic activity.

A predominantly ORR mechanism for the function of  $C_3N_4$ /UiO-66 was further supported the higher yield of  $H_2O_2$  in the presence of  $O_2$  and a significant decrease in  $H_2O_2$  production in Ar (Fig. 3b).<sup>38</sup> To elucidate the reactive species involved, sacrificial agent experiments were conducted.<sup>39</sup> As shown in Fig. 3c, the addition of *p*-benzoquinone (*p*-BQ), a scavenger for superoxide radicals ( $*O_2^-$ ), led to a 94% decrease in  $H_2O_2$  production, indicating  $*O_2^-$  to be the primary active species. The introduction of *tert*-butanol (TBA), a scavenger for hydroxyl radicals ( $*OH$ ), resulted in a 36.1% decrease, suggesting  $*OH$  to be a secondary contributor. Furthermore, introduction of sodium iodate ( $NaIO_3$ ), an electron scavenger, reduced the yield

by 45.8%, whereas introducing the hole scavenger EDTA-2Na caused only a 7% reduction. These findings confirmed that  $H_2O_2$  production proceeded predominantly *via* a  $2e^-$  ORR pathway, with additional contributions from  $2e^-$  and  $4e^-$  WOR processes. EPR measurements using spin-trapping agents further validated the generation of  $*O_2^-$  and  $*OH$  radicals (Fig. 3d and e). The  $C_3N_4$ /UiO-66 composite exhibited stronger  $*O_2^-$  signals than did the individual components, highlighting its superior ability to activate  $O_2$  and produce reactive intermediates. These results were found to be consistent with those from atmospheric control and quenching experiments, collectively confirming that the enhanced piezocatalytic  $H_2O_2$  production arose from efficient interfacial charge separation and a predominant  $*O_2^-$ -driven ORR pathway.<sup>38,40</sup>

Based on the previous discussions, a plausible piezoelectric catalytic mechanism for the  $C_3N_4$ /UiO-66 composites was derived, and is outlined in Fig. 4. The  $C_3N_4$ /UiO-66 composite material was concluded to generate  $H_2O_2$  through a piezoelectric-field-driven dual pathway.<sup>41</sup> Under mechanical stress, such as ultrasound, the piezoelectric field generated by the piezoelectric material  $C_3N_4$  interacts with the internal electric field at the interface, creating a coupled enhancement effect: the piezoelectric field drives the efficient separation of electron-hole pairs, with electrons ( $e^-$ ) migrating directly to the Zr active sites of UiO-66 under the guidance of the IEF, generating  $H_2O_2$  through the  $2e^-$  ORR pathway; and holes ( $h^+$ ) accumulate on the  $C_3N_4$  surface under the dual electric field, producing  $*OH$  through the WOR pathway, with  $*OH$  then indirectly converted into  $H_2O_2$  through radical coupling. This synergistic effect not only continuously provides a driving force through the piezoelectric field to overcome charge recombination but also precisely regulates the spatial distribution of  $e^-/h^+$  *via* the IEF, enabling the ORR and WOR pathways to proceed efficiently in parallel, ultimately achieving a significant increase in  $H_2O_2$  yield.

In summary, we successfully developed a  $C_3N_4$ /UiO-66 composite *via* electrostatic self-assembly, which exhibited a significantly enhanced IEF that promoted efficient piezocatalytic production of  $H_2O_2$ . The optimized composite achieved an outstanding  $H_2O_2$ -generation rate under ambient conditions, superior to those of  $C_3N_4$  and UiO-66. The excellent performance was attributed to the strong IEF at the  $C_3N_4$ /UiO-66 interface, which promotes directional charge separation and facilitates carrier transport. Mechanistic studies revealed the engineered IEF not only facilitating effective charge separation



**Fig. 3** (a) Plots of EPR intensity versus  $g$ -factor. (b) Piezoelectric  $H_2O_2$  generation under different atmospheres. (c) Values of piezocatalytic  $H_2O_2$  production at 60 min when including indicated scavengers (*p*-BQ for  $*O_2^-$ , TBA for  $*OH$ ,  $NaIO_3$  for  $e^-$ , EDTA-2Na for  $h^+$ ). (d) and (e) Plots of EPR intensity versus  $G$  for the detection of (d) DMPO- $*O_2^-$  and (e) DMPO- $*OH$ , each in the presence of  $C_3N_4$ /UiO-66, UiO-66 and  $C_3N_4$ , respectively.



**Fig. 4** Proposed reaction mechanism of  $H_2O_2$  production piezocatalyzed by  $C_3N_4$ /UiO-66.

but also simultaneously promoting both ORR and WOR pathways, as evidenced by EPR detection of  $\bullet\text{O}_2^-$  and  $\bullet\text{OH}$  radicals. This work presents a promising strategy for designing advanced piezocatalytic systems through interfacial engineering, paving the way for sustainable chemical synthesis using mechanical energy.

J. Y.: validation, investigation, writing – original draft. H. C.: formal analysis, methodology. J. L.: investigation, methodology. L.G.: writing – review & editing, supervision. Z. L.: conceptualization, writing – review & editing, supervision. M. Z.: resources, funding, writing – review & editing, supervision.

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## Conflicts of interest

There are no conflicts to declare.

## Data availability

The data supporting this article have been included as part of the supplementary information (SI). The experimental methods and characterisation data (TEM and XPS) supporting this article have been included in the SI. See DOI: <https://doi.org/10.1039/d5cc04517g>.

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