Supplementary Information (SI) for Journal of Materials Chemistry A. This journal is © The Royal Society of Chemistry 2025

**Supporting Information** 1 2 3 Highly active and stable PdRE catalysts for enhancing the electrocatalytic methanol and ethanol oxidation 4 5 6 Lingling Li<sup>a, 1</sup>, Mingli Ouyang<sup>a, 1</sup>, Lihua Zhu<sup>a, 1, \*</sup>, Yingliang Feng<sup>a</sup>, An Pei<sup>a, \*</sup>, Guang Li<sup>c</sup>, Tongxiang Liang <sup>b,\*</sup> 7 8 9 a Jiangxi Province Key Laboratory of Functional Crystalline Materials Chemistry, College of 10 Chemistry and Chemical Engineering, Jiangxi University of Science and Technology, Ganzhou 11 341000, Jiangxi, China. 12 <sup>b</sup> College of Rare Earth, Jiangxi University of Science and Technology, Ganzhou 341000, Jiangxi, 13 China. 14 ° College of Chemistry and Chemical Engineering, Xiamen University, Xiamen 361005, Fujian, 15 China. 16 17<sup>1</sup> These authors contributed equally to this work. 18 Corresponding authors: lihuazhu@stu.xmu.edu.cn (Lihua Zhu); anpei03@foxmail.com (An Pei); 19 txliang@jxust.edu.cn (Tongxiang Liang)

## **Characterization of the catalysts**

ICP-OES (PE Optima 8000 ICP-OES) testing was used to analyze the contents of the metals in the catalysts. X-ray diffraction (XRD-Rigaku D/MAX 2000 PC) tests were applied to determine the presence of the crystallite phase and structure in the catalysts. The testing conditions were as follows: Cu Ka as the X-ray source,  $\lambda =$ 0.15406 nm; 20 scanning range: 10°-90°, test voltage: 40 kV; current: 30 mA. X-ray photoelectron spectroscopy (XPS) measurement with a monochromatic Al  $K\alpha$  X-ray source with hv = 1486.6 eV was applied to study the elements in the catalyst as well as their chemical valence states and compositions. The microstructure of the materials could be investigated by acquiring transmission electron microscopy (TEM) and High-resolution TEM (HRTEM) images by TECNAI F30 with an accelerating voltage of 300 KV. Scanning transmission electron microscopy (STEM-TECNAI F30, 300 kV, FEI) combined with STEM-energy dispersive X-ray spectroscopy (STEM-EDX) line scanning and mapping can be used to analyze the elemental composition and nanostructures. Scanning electron microscopy (SEM) images and SEM-energy dispersive X-ray spectroscopy (SEM-EDX) were obtained at an operating voltage of 15 kV (SU8010, Hitachi).

Electrochemical *in-situ* Fourier transform infrared (FTIR) reflection spectroscopy using a Nicolet-8700 spectrometer equipped with a liquid-nitrogencooled MCT-A detector, the details are given as below: Electrochemical *in-situ* Fourier transform infrared (*in-situ* FTIR) reflection spectroscopy was carried out by using a Nicolet-8700 spectrometer included with a liquid-nitrogen-cooled MCT-A detector. The absorbed species and dissolved species could be detected by IR passed through the thin solution layer formed by the working electrode and CaF<sub>2</sub> windows. A Pt foil and a SCE were used as counter and reference electrodes, respectively. The catalyst (2.0 mg) was dispersed in 1.0 mL of mixed solution (ethanol: water = 1: 1), and ultrasonic for 30 mins. 10  $\mu$ L ink was deposited on the working electrode and we added 5  $\mu$ L 0.25 wt% Nafion solution after ink dried. The working electrode was electrochemically cleaned until stable in N<sub>2</sub>-saturated 1.0 M KOH solution, and electrochemical *in-situ* FTIR was measured in 1.0 M KOH + 1.0 M CH<sub>3</sub>CH<sub>2</sub>OH. Multi-stepped FTIR spectroscopy (MS-FTIR) was used to collect spectra from -0.857 V to 0.143 V (vs. SCE) at 0.100 V intervals. The FTIR spectra were recorded from 1000 cm<sup>-1</sup> to 4000 cm<sup>-1</sup> with a spectral resolution of 8 cm<sup>-1</sup>. The spectra were reported as the relative change in reflectivity ( $\Delta$ R/R), calculated by the following equation:

$$\frac{\Delta}{R} = \frac{R}{R} \frac{R}{R} \frac{H_{S}}{R} - R \frac{H_{R}}{R}$$

 $R(E_S)$  and  $R(E_R)$  represented the single-beam spectra recorded at the sample potential ES and the reference potential  $E_R$ , respectively. The downward bands and upward bands in the resulting spectra indicated the formation of products and the consumption of reactants. The  $E_R$  was fixed at -0.757 V.

## Electrode preparation and electrochemical studies

The electrochemical tests were performed by dropping a certain amount of the catalyst on the surface of a glassy carbon electrode, and the electrocatalytic methanol and ethanol oxidation reactions were carried out by setting up a classical three-electrode system (working electrode-glassy carbon electrode, counter electrode-Pt foil, reference electrode-saturated calomel electrode (SCE)) on an electrochemical workstation CHI 760E, and the catalytic performance, stability, electrochemical impedance and anti-CO poisoning performance were tested. All potentials were corrected according to the Nernst equation:

$$E_{RHE} = E_{SCE} + 0.0591 * pH + 0.2415$$

The catalyst ink of 2 mg/mL was prepared by adding 0.1 mL of ultrapure water and 0.9 mL of isopropanol to 2 mg of the catalyst, and after sonication for 30 min, 5  $\mu$ L of catalyst solution was taken from the surface of the glassy carbon electrode with a pipette gun and a total of 10  $\mu$ L of catalyst solution was dropped on the surface of the electrode, after the electrode surface was completely dry, then 5  $\mu$ L of 0.25 wt% Nafion solution was dropped on the surface of the glassy carbon electrode. After the Nafion solution dried, a layer of Nafion cation exchange membrane could be formed on the surface, which played the role of proton transport and prevented the diffusion of methanol from the anode to the cathode. The glassy carbon electrode dripping with catalyst was then placed in 48 mL of saturated nitrogen 1.0 M KOH electrolyte solution and activated by scanning 80 cycles at a rate of 200 mV s<sup>-1</sup>, followed by 5 cycles at a rate of 50 mV s<sup>-1</sup>. After activation, 2.0 mL of methanol was added to the electrolytic cell to form 1.0 M KOH + 1.0 M CH<sub>3</sub>OH solution for CV performance testing. The electrode for the ethanol test was prepared in the same way as above, except that the electrolyte solution was 47 mL of saturated nitrogen 1.0 M KOH, followed by the addition of 3 mL of CH<sub>3</sub>CH<sub>2</sub>OH after activation to form 1.0 M KOH + 1.0 M CH<sub>3</sub>CH<sub>2</sub>OH solution. Both methanol and ethanol oxidation reaction stability tests were carried out using timed currents with a constant potential set at 0.769 V (vs. RHE-the reversible hydrogen electrode) to detect the change in current. Electrochemical impedance spectroscopy (EIS) was obtained by testing at opencircuit voltage in the frequency interval of 0.01-100 kHz with an amplitude of 5 mV. For the CO-stripping tests, 20 µL (2 mg/mL) of catalyst solution was dripped onto the electrode surface, and then CO was passed into 1 M KOH solution and the CV curves were measured in the potential range of  $0.050 \sim 1.200$  V (vs. RHE) to obtain CO stripping peaks. The CO oxidation peak was integrated and the electrochemically surface areas (ECSAs) of the sample was obtained from the following equation:

$$ECSA = \frac{Q_e}{M \times 0.42 \ (mC \ cm^{-2})}$$

$$Q_e = \frac{\int IdE}{v}$$

where  $\int IdE$  was the integral of the shaded area under the CV curve after

deducting the double layer current and  $\nu$  was the scanning speed (50 mV s<sup>-1</sup>). A monolayer of adsorbed CO electrooxidation requires 0.42 mC cm<sup>-2</sup>.

Among them, the electrochemical workstation was purchased from Shanghai Chenhua Instrument Co., Ltd, the metal foil for the counter electrode and the glassy carbon electrode for the working electrode in the three-electrode system were purchased from Shanghai Yuejie Electronic Technology Co., Ltd, and the reference electrode, the calomel electrode, came from Wuhu Vulank New Materials Co., Ltd.



Fig. S1. XPS survey spectra of (a) PdLa/C, (b) PdNd/C, and (c) PdDy/C.



**Fig. S2.** STEM-EDX elemental maps of Pd + La.



Fig. S3. SEM images of (a) PdLa/C, (b) PdNd/C and (c) PdDy/C. Scale bar: 200 nm.



Fig. S4. SEM image and SEM-EDX elemental mapping of PdLa/C: (b) C (red), (c) Pd (green) and

(d)

La

(blue).



Fig. S5. SEM image and SEM-EDX elemental mapping of PdNd/C: (b) C (red), (c) Pd (green) and (d) Nd (purple).



Fig. S6. SEM image and SEM-EDX elemental mapping of PdDy/C: (b) C (red), (c) Pd (green) and (d) Dy (yellow).



Fig. S7. CV curves in (a) 1.0 M KOH solution.



**Fig. S8.** (a) the CA curves of the catalysts for the MOR, (b) CV curves of PdLa/C, PdNd/C, PdDy/C and Pd/C-JM normalized to MA after MOR stability measurement in 1.0 M KOH + 1.0 M CH<sub>3</sub>OH, (c) comparison plots of the electrocatalytic activity of PdLa/C, PdNd/C, PdDy/C and Pd/C-JM catalysts before and after stability, (d) CA testing at 0.77 V (vs. RHE) for 4000 s, (e) CV curves of PdLa/C, PdNd/C, PdDy/C and Pd/C-JM normalized to MA after EOR stability measurement in 1.0 M KOH + 1.0 M CH<sub>3</sub>CH<sub>2</sub>OH, (f) comparison plots of the electrocatalytic activity of PdLa/C, PdNd/C, PdDy/C and Pd/C-JM catalysts before and after stability.



Fig. S9. (a, b) TEM and HRTEM images of PdLa/C after 4000 s CA test in 1 M KOH + 1 M CH<sub>3</sub>OH, (c, d) TEM and HRTEM images of PdLa/C after 4000 s CA test in 1 M KOH + 1 M  $CH_3CH_2OH$ .

	Electrocatalysts	Electrolyte	Mass activity (A mg <sub>metal</sub>	Reference
			1)	
1	Pd <sub>45</sub> Pt <sub>42</sub> Ni <sub>13</sub> /C <sup>1</sup>	0.1 M KOH + 0.5 M CH <sub>3</sub> OH	16.2	<i>Chem. Eng. J.</i> , 2024, <b>483</b> , 149113.
2	Pd/Cu <sub>2</sub> O/Cl-GDY <sup>2</sup>	1 M KOH + 1 M CH <sub>3</sub> OH	4.0	J. Am. Chem. Soc., 2024, 147, 436-445.
3	Pd-UNs/Cl-GDY <sup>3</sup>	1 M KOH + 1 M CH <sub>3</sub> OH	3.6	Angew. Chem. Int. Ed., 2023, 62, e202308968.
4	PdLa/C	1 M KOH + 1 M CH <sub>3</sub> OH	2.28	This work
5	CrO <sub>x</sub> -Pd/C <sup>4</sup>	1 M KOH + 1 M CH <sub>3</sub> OH	2.05	Nano Lett., 2023, 23, 9555-9562.
6	Pd-Ni <sub>83</sub> Cu <sub>17</sub> /NiOOH <sup>5</sup>	1 M KOH + 1 M CH <sub>3</sub> OH	1.565	Appl. Surf. Sci., 2023, 614, 156214.
7	Pd(20%)-CeO2(20%)/NMCS6	1 M KOH + 1 M CH <sub>3</sub> OH	1.5	ACS Catal., 2019, 9, 6362-6371.
8	Pt-NGDY <sup>7</sup>	1 M KOH + 1 M CH <sub>3</sub> OH	1.4493	Adv. Sci., 2022, 9, 2104991.
9	$Pd_{3}Ru_{1}P_{1.5}^{8}$	1 M KOH + 1 M CH <sub>3</sub> OH	1.260	Int. J. Hydrogen Energ., 2017, <b>42</b> , 11229-11238.
10	Pt/Ni(OH) <sub>2</sub> /rGO-4 <sup>9</sup>	1 M KOH + 1 M CH <sub>3</sub> OH	1.236	Nat. Commun., 2015, 6, 1-8.
11	Pd/PdO nanotubes <sup>10</sup>	1 M KOH + 1 M CH <sub>3</sub> OH	1.1113	Adv. Funct. Mater., 2020, 30, 2000534.
12	Co-Rh <sub>2</sub> <sup>11</sup>	1 M KOH + 1 M CH <sub>3</sub> OH	0.889	Adv. Funct. Mater., 2023, 33, 2209134.
13	$Pd_1Ni_1/C^{12}$	1 M KOH + 1 M CH <sub>3</sub> OH	0.84737	Int. J. Hydrogen Energ., 2020, 45, 32022-32038.
14	Pd <sub>10</sub> Ag <sub>10</sub> /CNT <sup>13</sup>	$1 \text{ M KOH} + 0.5 \text{ M CH}_3\text{OH}$	0.731	ACS Appl. Energy Mater., 2018, 1, 3763-3770.
15	Rh NWs <sup>14</sup>	1 M KOH + 1 M CH <sub>3</sub> OH	0.722	Nano. Res., 2019, 12, 211-215.
16	PtCo CNCs <sup>15</sup>	1 M KOH + 1 M CH <sub>3</sub> OH	0.692	Appl. Catal. B-Environ., 2020, 277, 119135
17	PdNi <sup>16</sup>	1 M KOH + 1 M CH <sub>3</sub> OH	0.67708	Colloids and Surfaces A, 2017, <b>529</b> , 651-658.
18	$Pd_3Cu_1^{17}$	1 M KOH + 1 M CH <sub>3</sub> OH	0.6	Appl. Catal. B-Environ., 2016, 191, 76-85.
19	PdNW/cCuOx <sup>18</sup>	1 M KOH + 1 M CH <sub>3</sub> OH	0.55	Small, 2020, 16, 1904964.
20	Pd/Mxene <sup>19</sup>	1 M KOH + 1 M CH <sub>3</sub> OH	0.4	ACS Appl. Mater. Interfaces, 2019, 12, 2400-
				2406.

 Table S1. MA comparison of PaLa/C and some reported state-of-the-art catalysts for alkaline MOR.

	Electrocatalysts	Electrolyte	Mass activity (A $mg_{metal}$ <sup>-1</sup> )	Reference
1	icosa-Au@PdAu <sub>1.8L</sub> <sup>20</sup>	1 M KOH + 1 M CH <sub>3</sub> CH <sub>2</sub> OH	47.8	ACS Nano, 2024, 18, 18701-18711.
2	$Pd_{45}Pt_{42}Ni_{13}/C^1$	$0.1 \text{ M KOH} + 0.5 \text{ M CH}_3\text{CH}_2\text{OH}$	8.9	Chem. Eng. J., 2024, 483, 149113.
3	PdRhFeCoMo HEM <sup>21</sup>	1 M KOH + 1 M CH <sub>3</sub> CH <sub>2</sub> OH	7.47	Adv. Sci., 2024, 11, 2409109.
4	Ru <sub>0.04</sub> @Pd CUB <sup>22</sup>	1 M KOH + 1 M CH <sub>3</sub> CH <sub>2</sub> OH	6.53	Nano, Res., 2024, 17, 3863-3871.
5	PdLa/C	1 M KOH + 1 M CH <sub>3</sub> CH <sub>2</sub> OH	5.67	This work
6	$Pd_{1.5}Au^{23}$	$0.5 \text{ M KOH} + 2 \text{ M CH}_3\text{CH}_2\text{OH}$	5.3	J. Power Sources, 2013, 232, 99-105.
7	PtBi <sup>24</sup>	1 M KOH + 1 M CH <sub>3</sub> CH <sub>2</sub> OH	4.549	Nano Res., 2019, 12, 429-436.
8	$Pd_{28}Au_{32}Ag_{10}Cu_{30}^{25}$	1 M NaOH + 1 M CH <sub>3</sub> CH <sub>2</sub> OH	4.38	J. Colloid Interf. Sci., 2023, 633, 932-947.
9	Pd@CoNi/rG <sup>26</sup>	1 M KOH + 1 M CH <sub>3</sub> CH <sub>2</sub> OH	3.917	Chem. Eng. J., 2024, 502, 158010.
10	PdCuNi/C <sup>27</sup>	1 M KOH + 1 M CH <sub>3</sub> CH <sub>2</sub> OH	3.74	Geeen Chem., 2022, 24, 2438-2450.
11	Pd/AG-Ni <sub>3</sub> N <sup>28</sup>	1 M NaOH + 1 M CH <sub>3</sub> CH <sub>2</sub> OH	3.415	J. Energy Chem., 2021, 55, 48-54.
12	PdBi NRs <sup>29</sup>	1 M KOH + 1 M CH <sub>3</sub> CH <sub>2</sub> OH	3.21	Mater. Horiz., 2023, 10, 1416-1424.
13	c-Pd-Ni-P@a-Pd-Ni-P 170	1 M KOH + 1 M CH <sub>3</sub> CH <sub>2</sub> OH	3.05	Adv. Mater., 2020, <b>32</b> , 2000482.
	$^{\circ}\mathrm{C}^{30}$			
14	PdPtCu NSs <sup>31</sup>	1 M KOH + 1 M CH <sub>3</sub> CH <sub>2</sub> OH	2.67	Green Chem., 2019, 21, 2367-2374.
15	Pd NPs@Ni SAC <sup>32</sup>	1 M KOH + 1 M CH <sub>3</sub> CH <sub>2</sub> OH	1.093	J. Mater. Chem. A, 2022, 10, 6129-6133.
16	$Pd_1Cu_1^{17}$	1 M KOH + 1 M CH <sub>3</sub> CH <sub>2</sub> OH	1	Appl. Catal. B-Environ., 2016, 191, 76-
				85.
17	PdCuSn/CNTs <sup>33</sup>	$1 \text{ M KOH} + 0.5 \text{ M CH}_3\text{CH}_2\text{OH}$	0.8727	J. Power Sources, 2013, 242, 610-620.
18	Pd-FNMs <sup>34</sup>	$0.5 \text{ M NaOH} + 1 \text{ M CH}_3\text{CH}_2\text{OH}$	0.6	Adv. Mater., 2012, 24, 1594-1597.
19	Pd(56)Ni(44) <sup>35</sup>	1 M NaOH + 1 M CH <sub>3</sub> CH <sub>2</sub> OH	0.03	Electrochim. Acta, 2019, 295, 751-758.

 Table S2. MA comparison of PaLa/C and some reported state-of-the-art catalysts for alkaline EOR.



**Fig. S10.** (a) Identification of carbonate as the product of ethanol electrocatalytic oxidation. *In-situ* FTIR spectrum of the electrooxidation of 0.1 M ethanol at 1.20 V. Transmission IR spectra of (b) 0.1 M CH<sub>3</sub>COONa and (d) 0.1 M Na<sub>2</sub>CO<sub>3</sub>. (c) The resulting spectrum of a-b\*0.0405. The band at 1415 cm<sup>-1</sup> and 1550cm<sup>-1</sup> can be assigned to CH<sub>3</sub>COO<sup>-</sup> as compared with spectrum b. And the band at 1390 cm<sup>-1</sup> can be assigned to  $CO_3^{2-}$  as compared with spectrum d.

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