

Supporting Information

Facile synthesis of porous worm-like Pd nanotubes for high catalytic activity and stability towards ethylene glycol electrooxidation

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Experimental section

1. Chemicals

Palladous chloride (PdCl_2), dimethylglyoxime (dmg), ethylene glycol, ascorbic acid and NaBH_4 were purchased from Aladdin Co. Ltd. All the reagents were of analytical grade and used without further purification. All aqueous solutions were prepared with twice-distilled water.

2. Synthesis of worm-like PdO nanotubes

For the synthesis of the PdO WNTs, 10 mL PdCl_2 (100 mM) solution was drop-wisely added to a 25 mL dimethylglyoxime solution (100 mM) under stirring. The coordination compound $[\text{Pd}(\text{dmg})_2]_n$ was collected by centrifugation and thoroughly washed with water, calcined at 450°C for 2 h in muffle furnace, and cooled to room temperature naturally.

3. Synthesis of worm-like Pd nanotubes

Typically, 0.02 g PdO WNTs were dropped into a 10 mL NaBH_4 solution (25 mM) under stirring for 2 h. The final black precipitate was collected by centrifugation, thoroughly washed with water and ethanol, and dried at 60°C in vacuum for further characterization.

4. Characterizations

The morphology and size of the samples were characterized by scanning electron

microscopy (SEM, LEO-1530) and transmission electron microscopy (TEM, Hitachi S-4800), respectively. The crystal structures of the samples were examined by X-ray diffraction with Cu K α radiation (XRD, Philips PW3040/60). N₂ adsorption isotherms of the dried products were measured on a Micromeritics ASAP 2020 instrument. The specific surface areas and the pore-size distributions were determined using the Brunauer–Emmett–Teller (BET) and the Barrett–Joyner–Halenda (BJH) methods, respectively.

All electrochemical experiments were performed on a CHI 660D electrochemical workstation (Chenhua Instrument Shanghai Co. Ltd, China), in which a conventional three-electrode cell were used at room temperature, consisting of a glassy carbon electrode (GCE, $d = 0.3$ cm) as working electrode, saturated calomel electrode (SCE) as the reference electrode and a platinum wire as the counter electrode.

5. Preparation of the worm-like Pd nanotubes modified electrode

The Pd WNTs modified electrode was prepared as follows: 30 mg of the Pd WNTs were suspended in 1 mL of water, and then sonicated for 2 min to disperse the suspension. Next, 10 μ L of the suspension was dropped onto the freshly polished electrode surface and dried naturally. Another layer of Nafion (0.05 wt %) was deposited by drop-casting on the electrode to seal the products in place. Similarly, the Pd black (Alfa Aesar, fuel cell grade, BET: 20 m² g⁻¹) modified electrode was prepared under the similar conditions for comparison.

The electrocatalytic activity of the as-prepared electrode was examined by cyclic

voltammetry towards EG oxidation in 1.0 M KOH at a scan rate of 50 mV s⁻¹. For CO-stripping measurements, the electrode was firstly saturated with CO by bubbling CO through 0.5 M H₂SO₄ for 120 min, and quickly changed to a fresh one for further characterization.

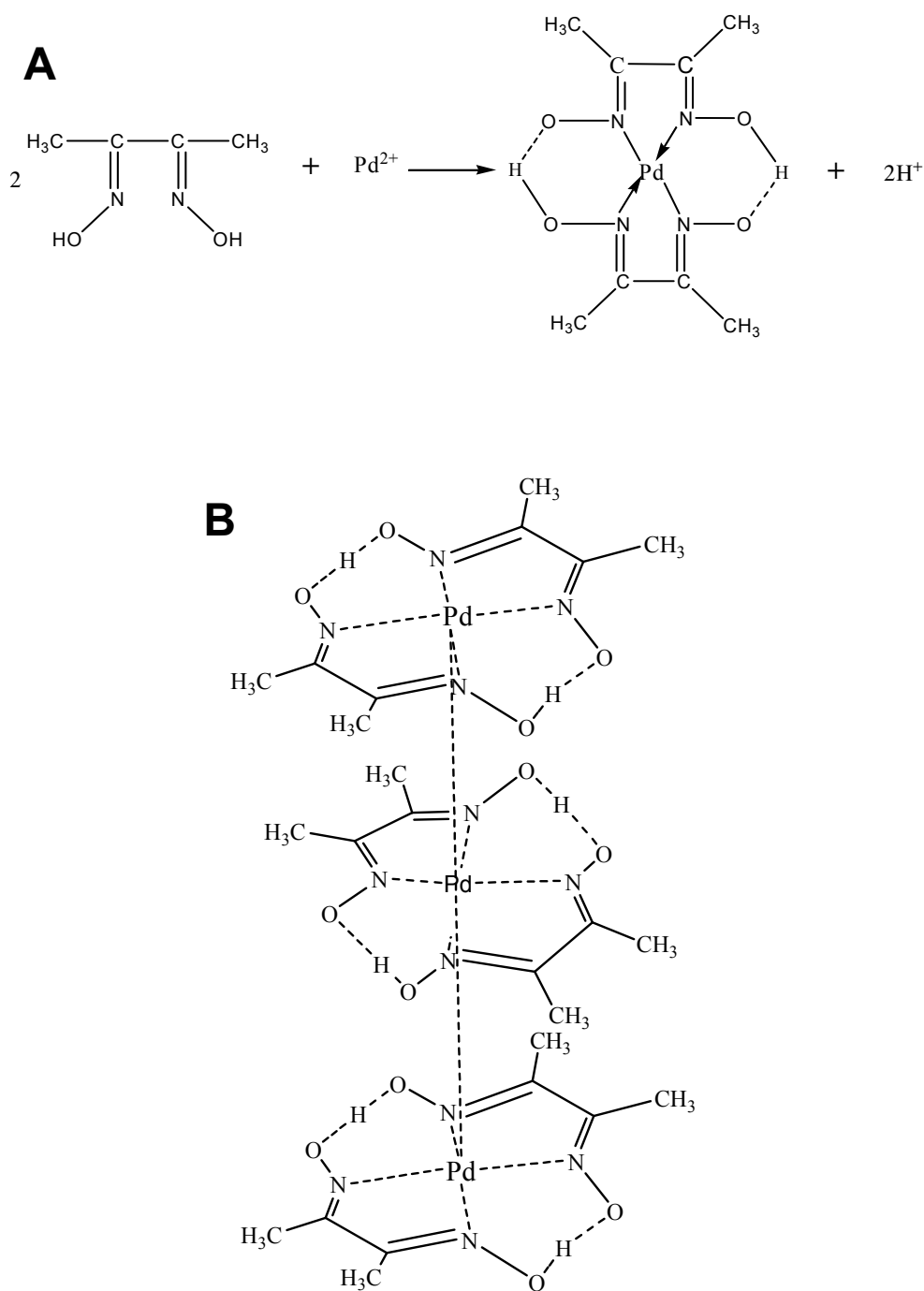


Fig. S1 (A) The forming process of $\text{Pd}(\text{dmg})_2$. (B) Columnar structure of $[\text{Pd}(\text{dmg})_2]_n$ nanowires.

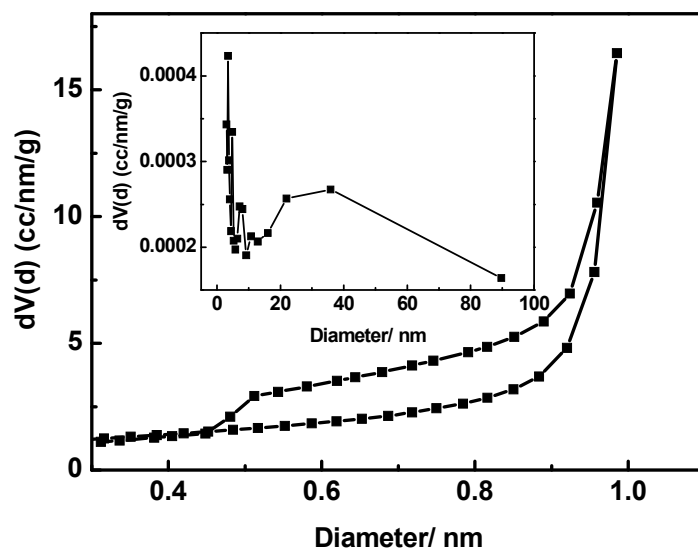


Fig. S2 Typical N₂ adsorption-desorption isotherm of the Pd WNTs sample. Inset shows the corresponding pore-size distribution.

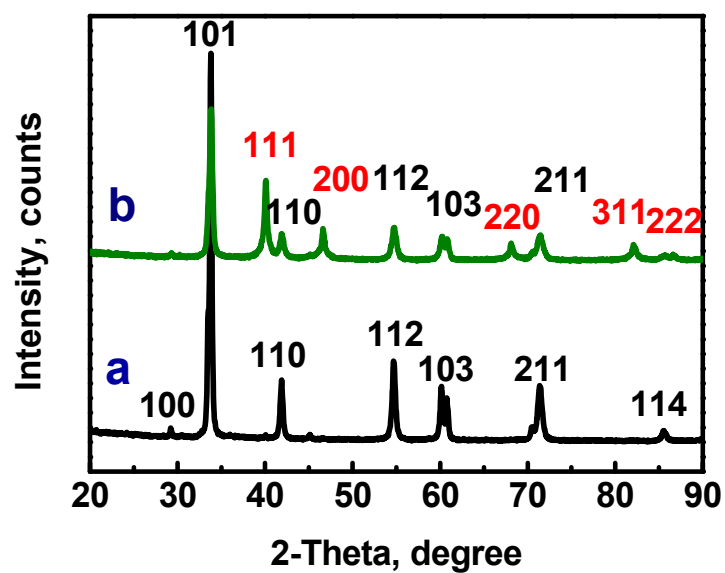


Fig. S3 XRD patterns of the products reduced by ascorbic acid (curve a) and hydrazine (curve b), respectively.

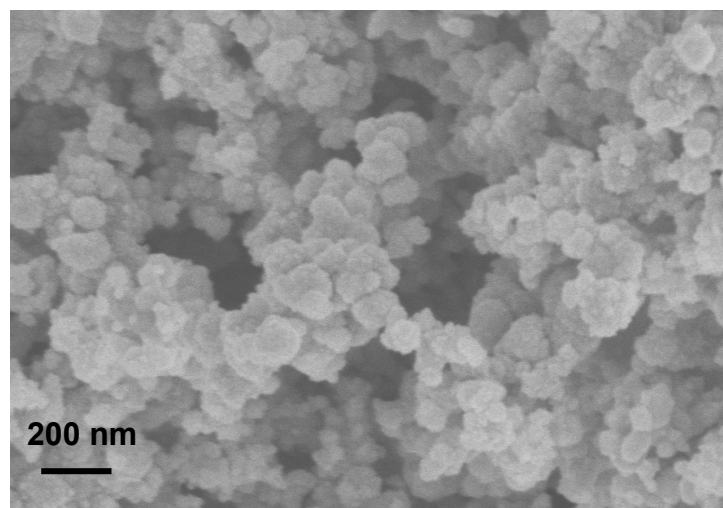


Fig. S4 SEM image of the Pd black sample.

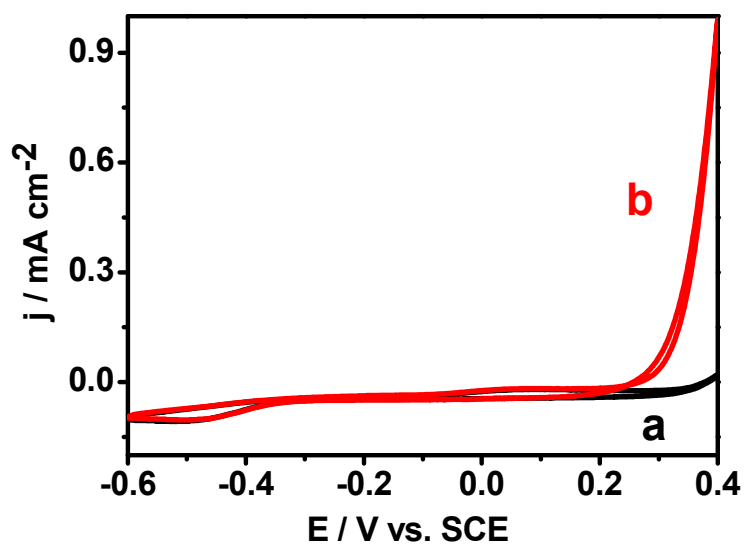


Fig. S5 The CVs obtained on the PdO WNTs modified electrode without (curve a) and with (curve b) 0.5 M EG in 1.0 M KOH solution.

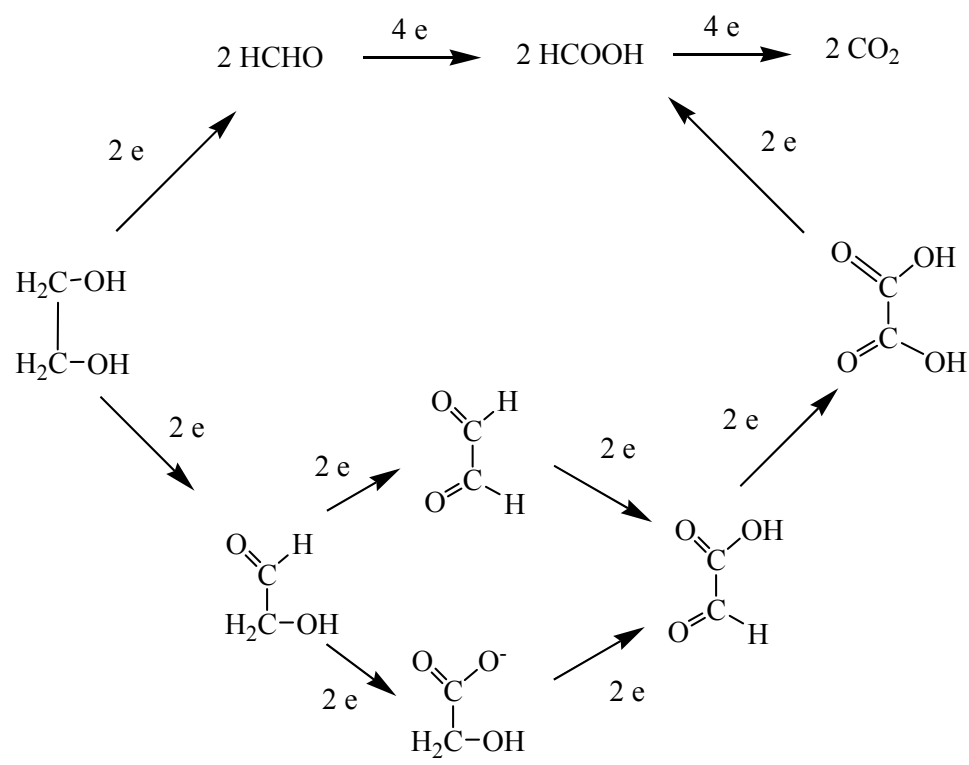


Fig. S6 Reaction pathways of the Pd WNTs for the oxidation of EG.

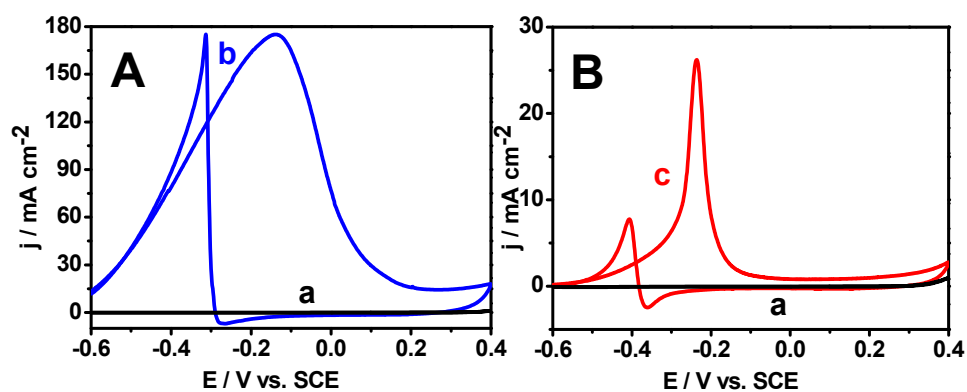


Fig. S7 The CVs obtained on the Pd WNTs modified electrode without (curve a) and with 0.5 M ethanol (curve b) or methanol (curve c) in 1.0 M KOH solutions.