Electronic Supplementary Material (ESI) for Dalton Transactions. This journal is © The Royal Society of Chemistry 2023

# Supplementary Material for PEDOT-embellished Ti<sub>3</sub>C<sub>2</sub>Tx Nanosheets Supported Pt-Pd Bimetallic Nanoparticles as an Efficient and Stable Methanol Oxidation Electrocatalyst

Shuyue Xie<sup>a</sup>, Fangfei Liu<sup>a</sup>, Tursun Abdiryim<sup>a,\*</sup>, Xiong Liu<sup>a</sup>, Ruxangul Jamal<sup>b,\*</sup>,

Yanyan Song<sup>a</sup>, Mariyam Niyaz<sup>a</sup>, Yajun Liu<sup>a</sup>, Hujun Zhang<sup>a</sup>, Xinsheng Tang<sup>a</sup>

<sup>a</sup> State Key Laboratory of Chemistry and Utilization of Carbon Based Energy Resources;
College of Chemistry, Xinjiang University, Urumqi, 830017, Xinjiang, P.R. China;
<sup>b</sup> State Key Laboratory of Chemistry and Utilization of Carbon Based Energy Resources;
School of Chemical Engineering and Technology, Xinjiang University, Urumqi, 830017, Xinjiang, P.R. China.

\*Corresponding author: tursunabdir@sina.com.cn; jruxangul@xju.edu.cn.

#### **1.1 Characterization**

The micromorphology and microstructure of the synthetic electrocatalysts were observed by field emission scanning electron microscopy (FE-SEM, JSM-7610FPlus) combined with the elemental mapping analysis and high-resolution transmission electron microscopy (HR-TEM, JEM-2100). X-ray powder diffraction (XRD, Bruker D8 advance), X-ray photoelectron spectroscopy (XPS, Thermo Scientific ESCALAB 250Xi) and Fourier transform infrared spectroscopy (FT-IR, VERTEX 70 RAMI) were used to analyze the chemical structure and composition of the electrocatalysts. The content of noble metal elements in the electrocatalyst composites was accurately analyzed using inductively coupled plasma optical emission spectrometer (ICP-OES, Optima 8000).

#### **1.2 Electrochemical measurements**

All electrocatalyst samples were evaluated for their electrocatalytic performance via CHI 660C electrochemical workstation (ChenHua Instrument Co., Ltd., Shanghai, China) under a standard three-electrode system. A typical three-electrode system combines a glassy carbon electrode (GCE, diameter of 4.0 mm) coated with active catalyst, a saturated calomel electrode (SCE) and a platinum electrode as the working electrode, the reference electrode and the counter electrode, respectively. Before electrocatalytic testing, the working electrodes need to be activated by cyclic voltammetry (CV) in N<sub>2</sub>-saturated 0.5 M H<sub>2</sub>SO<sub>4</sub> solution to a stable curve. The electrochemically active surface area (ECSA) values of the as-prepared electrocatalysts were first evaluated using CV in 0.5 M H<sub>2</sub>SO<sub>4</sub> solution. The working electrodes were

then switched to a mixed solution of  $0.5 \text{ M H}_2\text{SO}_4/1 \text{ M CH}_3\text{OH}$  for subsequent MOR evaluations by CV, chronoamperometry (CA), and electrochemical impedance spectroscopy (EIS).

#### **1.3 Preparation of the working electrode**

The working electrode was prepared as follows: first, a glassy carbon electrode (GCE) (d = 4.0 mm) was wet-polished with alumina slurry on the fleece, then the polished electrode was sonicated and the its surface was blown dry with nitrogen. Next, weigh 5 mg of Ti<sub>3</sub>C<sub>2</sub>Tx/PEDOT/Pt-Pd catalyst powder into a centrifuge tube, add 0.5 mL of ultrapure water and 0.5 mL of isopropanol, and sonicate for 5 min to prepare a suspension. Then use a micro injector to draw 1  $\mu$ L of Nafion solution (5 wt%) and 5  $\mu$ L of the above suspension, and drop them onto the smooth GCE surface, so that the catalyst mixture is evenly spread on the GCE surface. Finally, the obtained working electrodes were naturally air-dried at room temperature for subsequent testing.

### **1.4 The formula for calculating the ECSA value of the catalysts**

The electrochemically active surface area (ECSA) values of the catalysts were evaluated by cyclic voltammetry (CV) in  $0.5 \text{ M} \text{ H}_2\text{SO}_4$  solution at 50 mV s<sup>-1</sup>. The ECSA values for the catalysts were calculated according to the following formula:

$$ECSA = \frac{Q_{H}}{0.21^{*}[Pt]}$$

where  $Q_H$  is the actual charge for hydrogen adsorption in the CV curve (mC), 0.21 represents the charge adsorbed by the hydrogen single atomic layer on the Pt unit surface (mC cm<sup>-2</sup>), and [Pt] represents the actual loading of platinum (Pt) on the GCE substrate (g).

## **1.5** The formula for calculating the MA value of the catalysts

The mass activities (MA) values of the catalysts were evaluated by CV in a mixture contained 0.5 M  $H_2SO_4$  and 1 M CH<sub>3</sub>OH at 50 mV s<sup>-1</sup>. The MA values of the catalysts were calculated according to the following formula:

$$MA = \frac{If}{[Pt]}$$

where  $I_f$  is the forward sweep peak current density in the CV curve (mA) and [Pt] represents the actual loading of Pt on the GCE substrate (g).



Fig. S1. the SEM image of  $Ti_3C_2Al$  powder.



Fig. S2. the TEM image of  $Ti_3C_2Tx$  nanosheets.



Fig. S3. the SEM image of  $Ti_3C_2Tx/Pt$ -Pd sample.



Fig. S4. the particle size histogram of Pt-Pd NPs in Ti<sub>3</sub>C<sub>2</sub>Tx/Pt-Pd

sample.



Fig. S5. the particle size histogram of Pt-Pd NPs in  $Ti_3C_2Tx/PEDOT/Pt$ -

Pd sample.