Electronic Supplementary Information

Microwave-assisted, environmentally friendly, one-pot preparation of Pd nanoparticles/graphene nanocomposites and their application in electrocatalytic oxidation of methanol

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Experimental Section

Materials Graphite powder, TA, H_2O_2 (30 wt%), H_2PdCl_4, H_2SO_4, NaOH, CH_3OH, Na_2HPO_4, and NaH_2PO_4 were purchased from Aladdin Reagent Database Inc. (Shanghai, China) and used as received without further purification. Pd/C catalysts were purchased from Alfa Aesar. The water used throughout all experiments was purified through a Millipore system.

Preparation of GO GO was prepared from natural graphite powder through a modified Hummers method. In a typical synthesis, 1 g of graphite was added into 23 mL of 98 % H_2SO_4, followed by stirring at room temperature over a period of 24 h. After that, 100 mg of $NaNO_3$ was introduced into the mixture and stirred for 30 min. Subsequently, the mixture was kept below 5 ºC by ice bath, and 3 g of $KMnO_4$ was

slowly added into the mixture. After heating to 35-40 ºC, the mixture was stirred for another 30 min. After that, 46 mL of water was added into above mixture during a period of 25 min. Finally, 140 mL of water and 10 mL of H_2O_2 were added into the mixture to stop the reaction. After the unexploited graphite in the resulting mixture was removed by centrifugation, as-synthesized GO was dispersed into individual sheets in distilled water at a concentration of 0.5 mg/mL with the aid of ultrasound for further use.

One-pot synthesis of PdNPs-G nanocomposites In a typical experiment, 0.5 mL of GO was dispersed in 0.5 mL of 15 mg/mL TA aqueous solution under ultrasonic irradiation for 30 min. Next, 200 μL of 9.3 mM H_2PdCl_4 aqueous solution was added into the above TA-GO dispersion. The resulting mixture was sonicated for about 2 min and then placed in microwave oven for 1 min or 2min (powder: 750 W). The products in the black dispersion were centrifuged and washed twice with distilled water to remove the excess TA and redispersed in water for characterization and further use. Hereafter, the similar experiments were also performed with GO solution volume decreased to 0.25 mL and 0.125 mL, respectively.

Characterization Transmission electron microscopy (TEM) measurement was made on a HITACHI H-8100 EM (Hitachi, Tokyo, Japan) with an accelerating voltage of 200 kV. Raman spectra were obtained on J-Y T64000 Raman spectrometer with 514.5-nm wavelength incident laser light. X-ray photoelectron spectroscopy (XPS) analysis was measured on an ESCALABMK II X-ray photoelectron spectrometer using Mg as the exciting source. Electrochemical measurements are performed with a

CHI 660D electrochemical analyzer (CH Instruments, Inc., Shanghai). A conventional three-electrode cell was used, including a GCE (geometric area=0.07 cm^2) as the working electrode, a Ag/AgCl (saturated KCl) electrode as the reference electrode, and platinum foil as the counter electrode. The potentials are measured with a Ag/AgCl electrode as the reference electrode. All the experiments were carried out at ambient temperature.

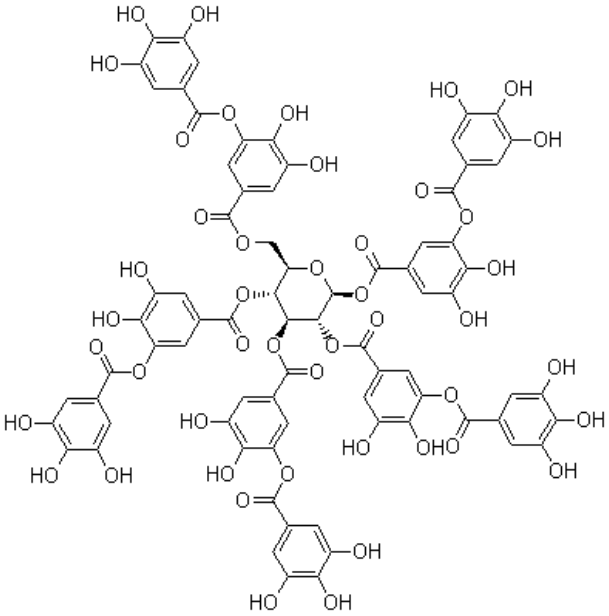

Fig. S1 Chemical structure of tannic acid (TA).

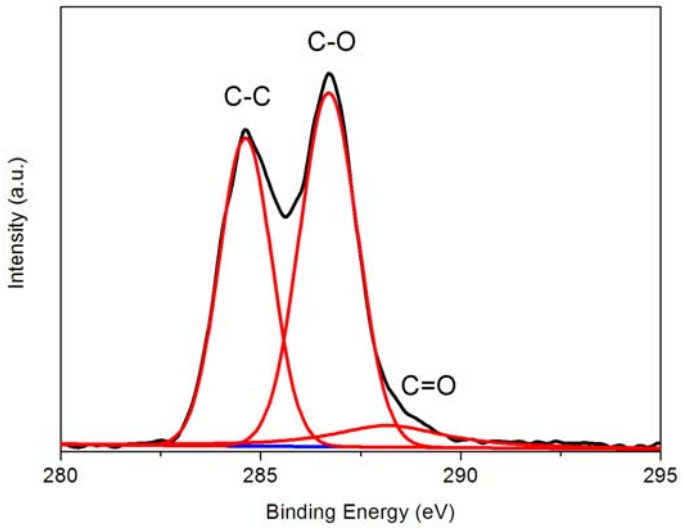

Fig. S2 C1s spectra of the products after 2-min microwave irradiation in the absence of TA.