

ELECTRONIC SUPPORTING INFORMATION

Covalently functionalized layered MoS₂ supported Pd nanoparticles as highly active oxygen reduction electrocatalyst

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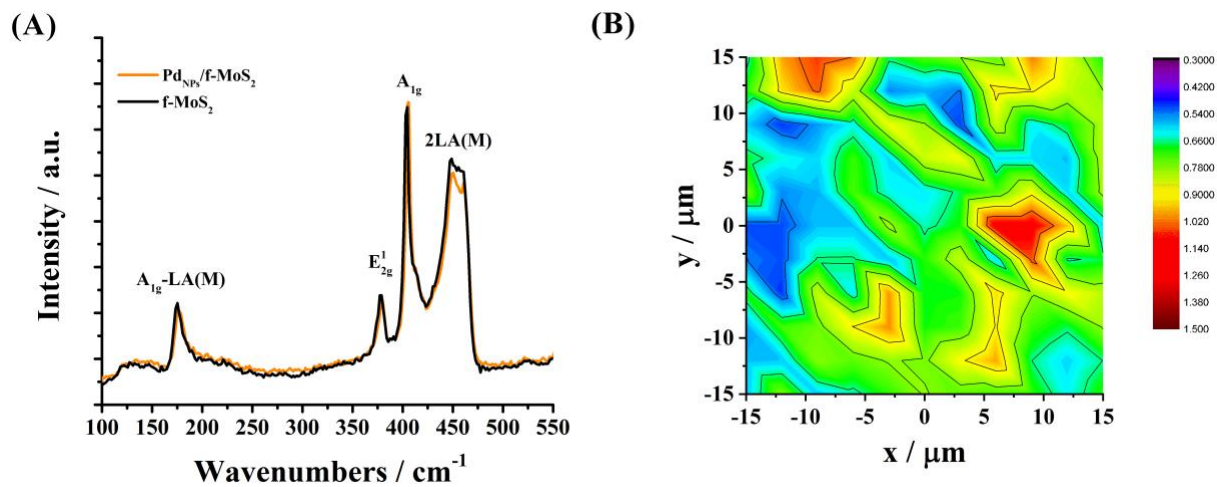


Figure S1. (A) Raman spectra for $Pd_{NPs}/f-MoS_2$ (orange) and functionalized MoS_2 (black); (B) Raman mapping upon excitation at 633 nm of the $2LA(M)/A_{1g}$ intensity ratio of a $30 \times 30 \mu m^2$ area (121 acquisition points in total) for $Pd_{NPs}/f-MoS_2$ hybrid material. In Figure S1A we present a representative Raman mapping, close to the total average of five.

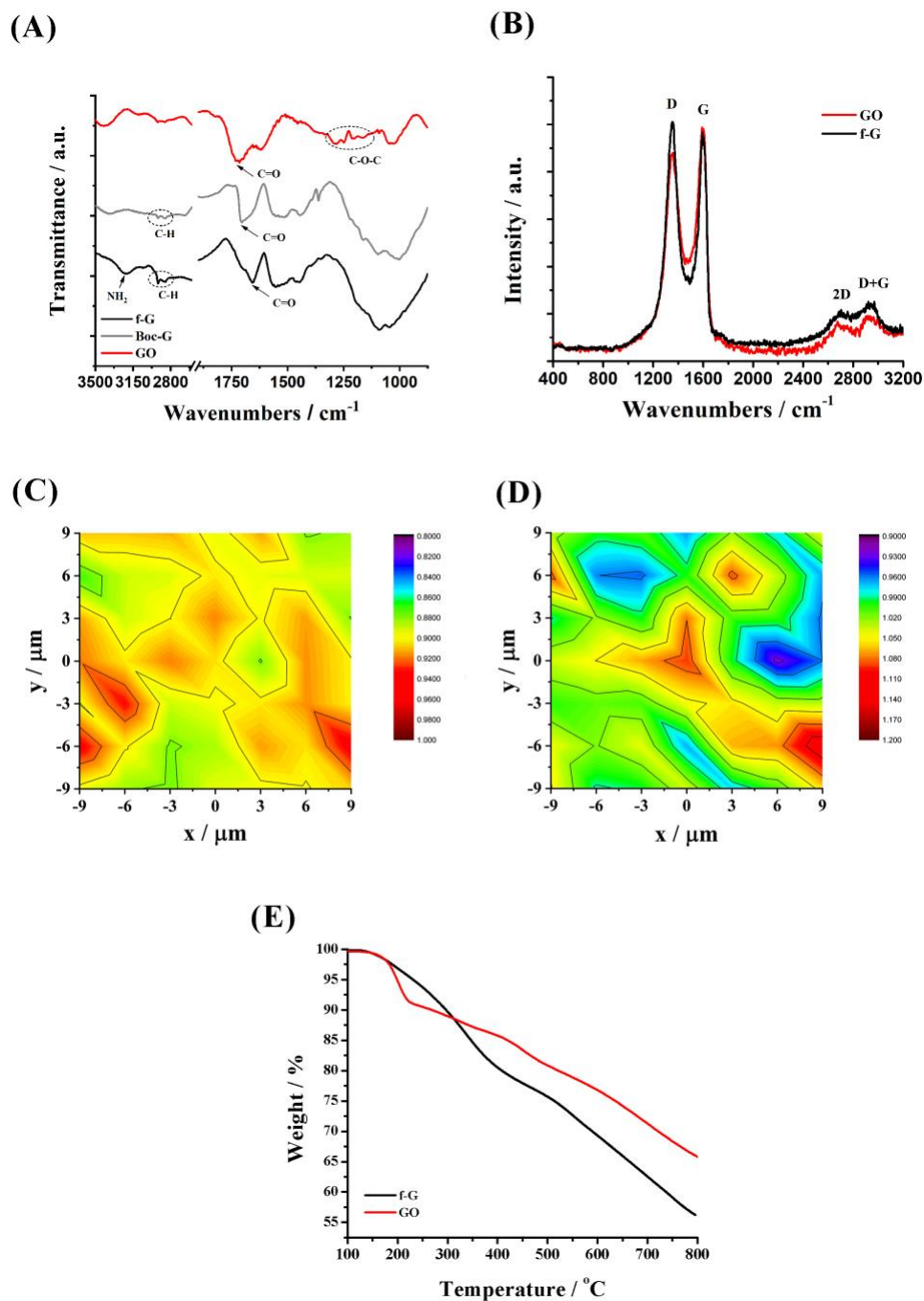


Figure S2. (A) ATR-IR spectra for amino functionalized graphene (black), BOC modified graphene (gray) and GO (red); (B) Raman spectra for f-G (black) and GO (red) upon excitation at 514 nm; (C, D) Raman mapping upon excitation at 514 nm of the D/G intensity ratio of a 18 x 18 mm² area (49 acquisition points in total) for GO and f-G, respectively. In Figure S2B we present a representative Raman mapping, close to the total average of five. (E) Thermographs for f-G (black) and GO (red).

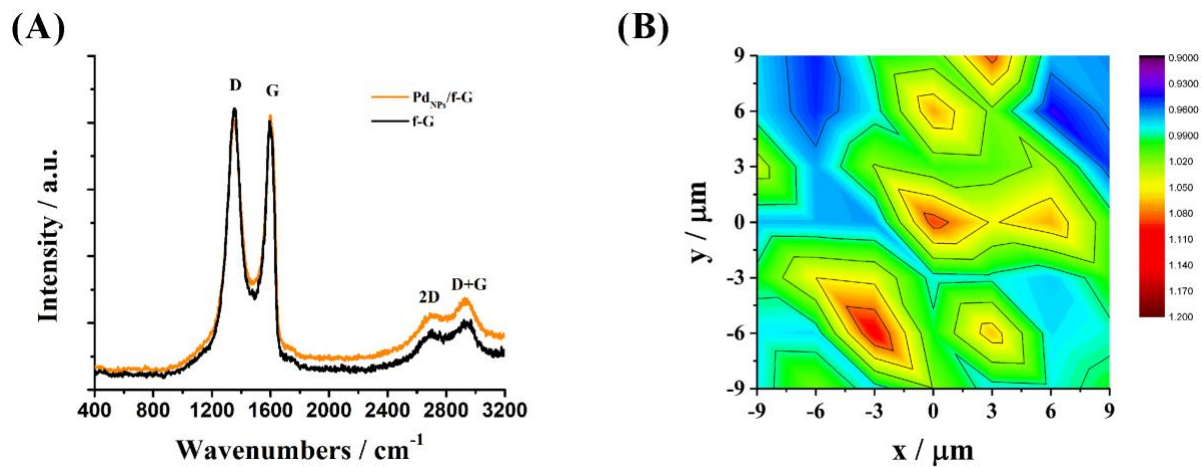


Figure S3. (A) Raman spectra for Pd_{NPs}/f-G (orange) and f-G (black); (B) Raman mapping upon excitation at 514 nm of the D/G intensity ratio of a 18 x 18 mm² area (49 acquisition points in total) for Pd_{NPs}/f-G hybrid material. In Figure S3A we present a representative Raman mapping, close to the total average of five.

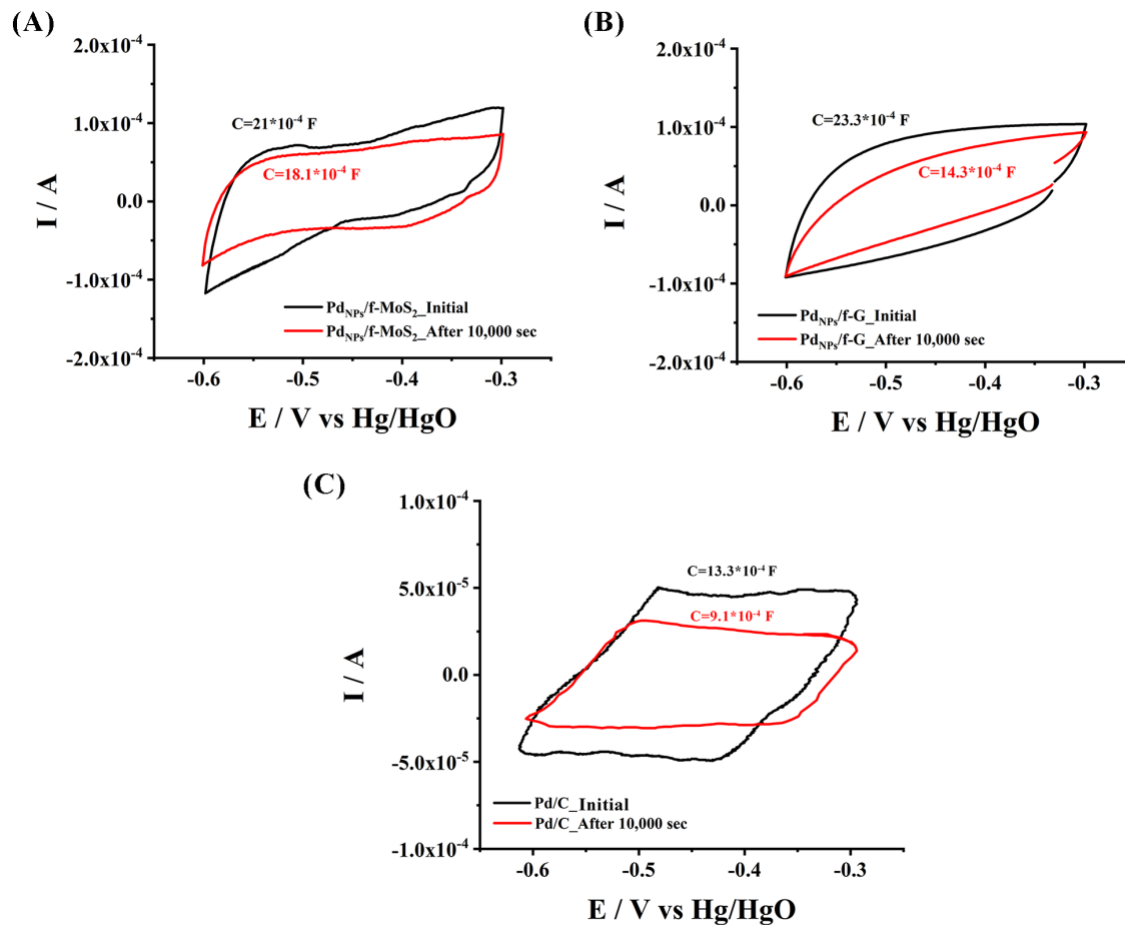


Figure S4. Cyclic voltammetry curves for (A) $\text{Pd}_{\text{NPs}}/\text{f-MoS}_2$, (B) $\text{Pd}_{\text{NPs}}/\text{f-G}$ hybrids and (C) Pd/C before and after the chronoamperometric essays. The CV curves obtained at the “double layer region” ($-0.6 - 0.3$ V vs Hg/HgO) recorded in N_2 saturated aqueous 0.1 M KOH solution at a scan rate 50 mV/sec. The capacitance values were calculated for each case using the Equation 6.

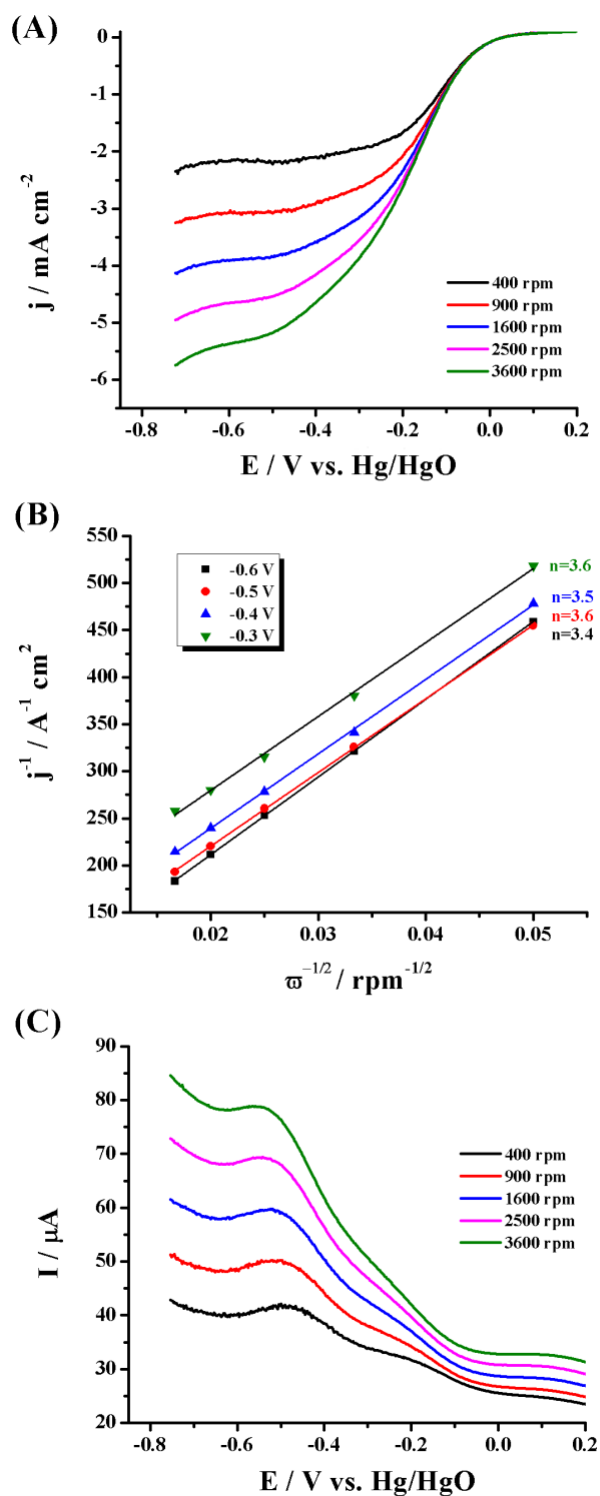


Figure S5. (A) ORR polarization curves at different rotation rates (400-3600 rpm) for PdNPs/f-G, (B) the corresponding K-L plots and (C) ring response. All measurements were conducted in O₂-saturated aqueous 0.1M KOH electrolyte, and the corresponding LSV polarization curves were recorded at a scan rate of 5 mVs⁻¹. The ring potential was fixed at 1.0 V vs Hg/HgO. The current densities in (A) are normalized to the geometric electrode area.

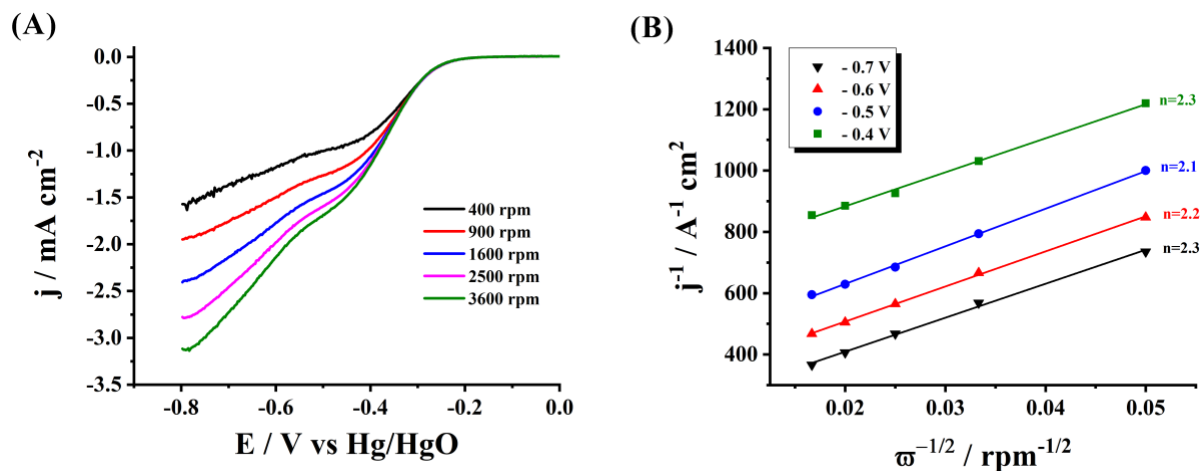


Figure S6. (A) ORR polarization curves at different rotation rates (400-3600 rpm) for *f*-MoS₂ and (B) the corresponding K-L plots. All measurements were conducted in O₂-saturated aqueous 0.1 M KOH electrolyte, and the corresponding LSV polarization curves were recorded at a scan rate of 5 mV/s. The current densities are normalized to the geometric electrode area.

Table S1. Performance analysis of recently developed hybrid MoS₂-based electrocatalysts for the ORR.

PdNPs (~9nm) on MoS ₂ nanosheets	O ₂ -saturated 0.1 M KOH	Onset potential: -0.10 V vs. SCE Half-wave potential: -0.20 V vs. SCE Diffusion limited current density: 5.2 mA cm ⁻² at 1600 rpm Mass activity: 0.55 mA μgPd ⁻¹ @ -0.26 V vs. SCE Specific activity: 1.02 mA cm ² EC _{SA} ⁻² @ -0.26 V vs. SCE Electron transfer number: 3.6 – 4.0 Stability: after 4,000 cycles, no significant activity loss	1
PdSNPs (40-50nm) on MoS ₂ /N-G heterostructure	O ₂ -saturated 0.1 M KOH	Onset potential: -0.141 V vs. SCE Half-wave potential: -0.214 V vs. SCE Diffusion limited current density: 4.1 mA cm ⁻² at 1600 rpm Electron transfer number: 3.75 – 3.82 Stability: after 1,000 cycles / 16,000 sec , no significant activity loss Methanol tolerance: up to 0.1 M CH ₃ OH	2
PdNPs (2-3nm) on covalently functionalized MoS ₂ nanosheets	O ₂ -saturated 0.1 M KOH	Onset potential: +0.066 V vs. Hg/HgO Half-wave potential: -0.116 V vs. Hg/HgO Diffusion limited current density: 5.7 mA cm ⁻² at 1600 rpm Kinetic current density: 2.65 mA cm ⁻² at -60 mV vs. Hg/HgO Electron transfer number: 3.6 – 4.0 Stability: after 10,000 sec, 16.5 % loss of its initial activity	This work

1. L.-X. Zuo, L.-P. Jiang and J.-J. Zhu, *Ultrason. Sonochem.*, 2017, **35**, 681-688.
2. L. G. Bach, M. L. N. Thi, Q. B. Bui and H. T. Nhac-Vu, *Synth. Met.*, 2019, **254**, 172-179.