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## **Figure Captions**

Fig. S1. The work function (W) determined by the difference between the photo energy and the binding energy of the secondary cutoff edge. (W = 21.2 eV - 16.6 eV = 4.6 eV)

**Fig. S2.** (a) Typical C-V curve of 3D Ni/NiO/MoS<sub>2</sub> foam with continuous addition of DA under continuous agitation. (b) Typical C-V curve of rGO with continuous addition of DA under continuous agitation.

The scan rage is from -0.6V to 0.6V at a rate of 100 mV·S<sup>-1</sup> and the number of sweep segments is 4.

**Fig. S3.** (a) The electrochemical response of the single  $MoS_2$  to 1 mM DA, UA and AA in 0.01 M PBS at pH = 7.4. (b) The electrochemical response of the single rGO to 1 mM DA, UA and AA in 0.01 M PBS at pH = 7.4. The scan rage is from -0.6V to 0.6V at a rate of 100 mV·S<sup>-1</sup> and the number of sweep segments is 4.

**Fig. S4.** CV curves of Ni/NiO (a), Ni/NiO/MoS<sub>2</sub> (b), and Ni/NiO/MoS<sub>2</sub>/rGO (c) against different scan rates.

**Annotation:** The the actual electrochemically active surface area was performed at room temperature in 0.1 M PBS (pH 7.4) using a standard three-electrode cell, where a Ag/AgCl electrode served as the reference electrode, a Pt wire served as the counter electrode, and the Ni/NiO or Ni/NiO/MoS<sub>2</sub> or Ni/NiO/MoS<sub>2</sub>/rGO (typically 1×1.5 cm<sup>2</sup>) covered with products served as the working electrode. All potentials were measured against the Ag/AgCl electrode and converted to the reversible hydrogen electrode (RHE) reference scale using ERHE (V) =  $E_{Ag/AgCl} + 0.197 + 0.059 \times pH$ . Cyclic voltammetry was measured from 0 to 0.1 V vs RHE at different scan rates from 20 mV·s-1 to 200 mV·s<sup>-1</sup>.

**Fig. S5.** Nyquist plots of EIS of the Ni/NiO (a), the Ni/NiO/MoS<sub>2</sub> (b) and the Ni/NiO/MoS<sub>2</sub>/rGO (c) with the addition of  $3 \mu M$  DA in the frequency ranges from 0.01 Hz to 100 KHz.

**Fig. S6.** Reproducibility of 3D Ni/NiO/MoS<sub>2</sub>/rGO foam after 100 successive cyclic voltammograms measurement for 1 mM DA.

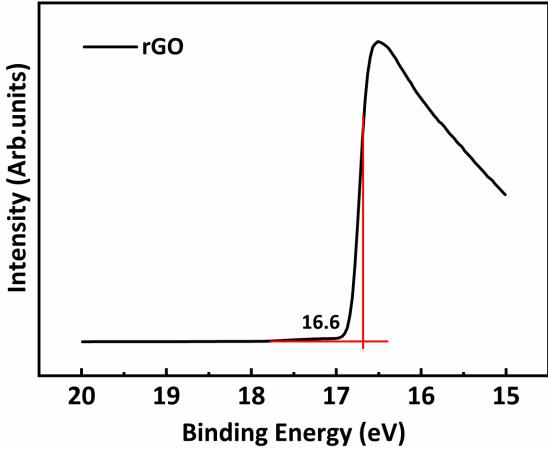


Fig.S1

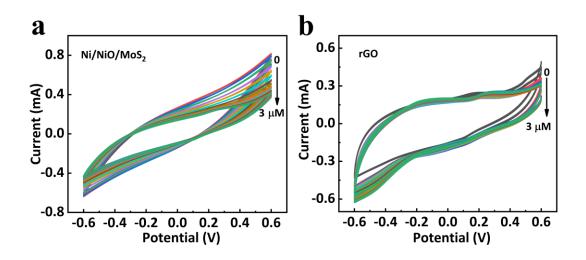
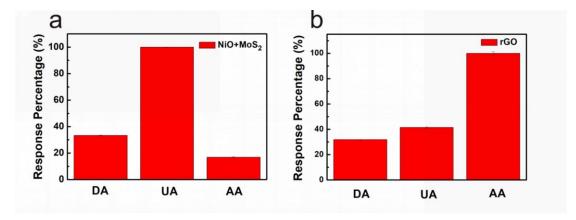
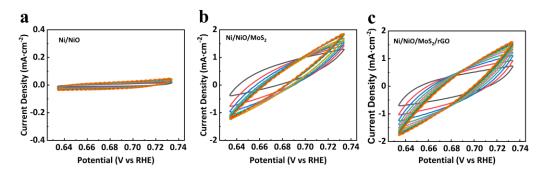


Fig.S2









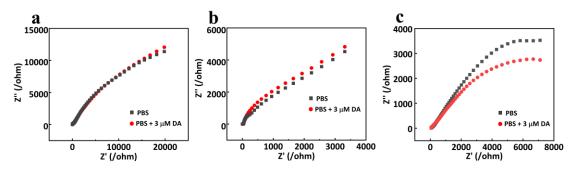


Fig.S5

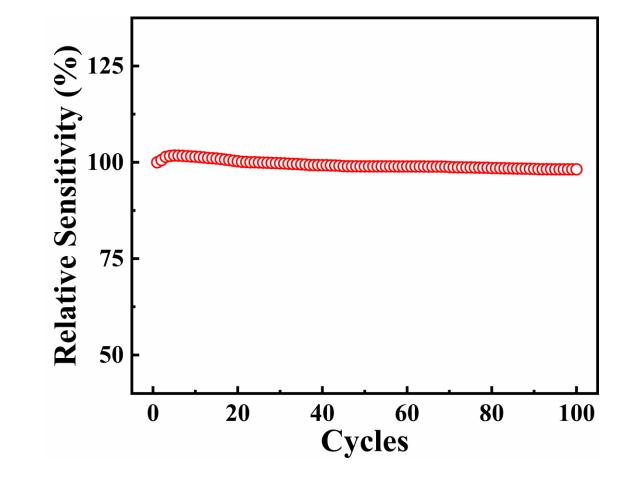


Fig. S6