Supplied Materials

Large–area, three–dimensional interconnected graphene oxide intercalated with self–doped polyaniline nanofibers as a free–standing electrocatalytic platform for adenine and guanine

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1. XPS characterization of GNO-SPAN

The C 1s XPS spectrum of GNO-SPAN shown in Figure S1A clearly indicated a certain extent of oxidation of graphite upon chemical exfoliation. The three prominent peaks (284.5, 285.3, 287.0 eV) observed on GNO-SPAN present the existence of a certain amount of functional groups on the surface[1,2]. From N 1s XPS spectrum (Figure S1B), the benzenoid amine with binding energy (BE) centered at 399.5 eV and the nitrogen cationic radical (N + ·) with BE at 401.9 eV are clearly identified[3]. As shown in Fig. S1C (S 2p XPS spectrum), the S 2p peaks appeared at 167.4 eV and 168.4 eV are consistent with the existence of -SO$_3^-$ groups in the GNO-SPAN, which is further confirmed by O 1s XPS spectrum (Fig. S1D, 532.7 eV) [4]. In addition, the peak at 531.1 eV in O 1s XPS spectrum is corresponding to C-O entity[5].

Fig. S1 The C 1s(A), N 1s(B), O 1s (C) and S 2p (D) XPS spectrum of GNO-SPAN. *Note: count per second (CPS)*
2. Optimization of determination conditions

**Fig. S2** (A) Representative DPV of 2×10⁻⁴ mol/L adenine in ABS (a), B–R (b) and PBS (c) buffer solutions. (B), (C), (D) The effect of pH on the electrooxidation of 5.0×10⁻⁵ mol/L adenine. (a)–(g) 5.5, 6.0, 6.5, 7.0, 7.5, 8.0 and 8.5. *Note:* Each point is the mean of three measurements, and the error bars correspond to the standard deviation.
**Fig. S3** Representative DPV in B–R solution with $2 \times 10^{-4}$ mol/L guanine recorded at GNO–SPAN with different ultrasonic time (a) 10 min, (b) 20 min, (c) 30 min, (d) 40 min.
3. Determinations of guanine and adenine

Fig. S4 Representative DPV of guanine (A) and adenine (B) (from a to j: 0.5, 1, 5, 20, 40, 60, 80, 120, 150 and 200 μmol/L) at GNO–SPAN/CPE in B–R. Calibration curves of the oxidation peak current versus different concentration of guanine (C) and adenine (D), respectively. Note: Each point is the mean of three measurements, and the error bars correspond to the standard deviation.
Fig. S5 (A) Representative DPV of guanine (from a to j: 0.5, 1, 5, 20, 40, 60, 80, 120, 150 and 200 μmol/L) in B–R (pH 7.0) with coexistence of $5.0 \times 10^{-5}$ mol/L adenine at GNO–SPAN/CPE. (B) Representative DPV of adenine (from a to j: 0.5, 1, 5, 20, 40, 60, 80, 120, 150 and 200 μmol/L) in B–R (pH 7.0) with coexistence of $5.0 \times 10^{-5}$ mol/L guanine at GNO–SPAN/CPE. (C) Calibration curve of guanine. (D) Calibration curve of adenine. Note: Each point is the mean of three measurements, and the error bars correspond to the standard deviation.