Electronic Supplementary Information

Experimental section

Materials: CC was purchased from Hongshan District, Wuhan Instrument Surgical Instruments Business, China and was cleaned respectively in water and ethanol to eliminate the surface impurities by ultrasonic processing. H$_2$O$_2$ solution (30 wt % aqueous), thiourea, hydrochloric acid (HCl), sodium chloride (NaCl), phorbol-12-myristate-13-acetate (PMA, 1 μL, 50 μg mL$^{-1}$), L-cysteine (L-Cys), ascorbic acid (AA), uric acid (UA), dopamine (DA), sodium molybdic dihydrat (Na$_2$MoO$_4$·2H$_2$O), sodium dihydrogen phosphate (NaH$_2$PO$_4$) and disodium hydrogen phosphate (Na$_2$HPO$_4$) were purchased from Aladdin Ltd (Shanghai, China). The 0.1 M phosphate buffer solution (PBS, 0.1 M, pH = 7.4) as electrolyte was prepared via dissolving Na$_2$HPO$_4$ and NaH$_2$PO$_4$ in doubly distilled water, then 0.2 M HCl was added to regulate and control the final pH value under mild stirring. All reagents and solvents were of analytical grade and used as received without purification.

Synthesis of the MoS$_2$/CC: The MoS$_2$ nanosheets were synthesized through easy hydrothermal procedure: thiourea (0.61 g) and Na$_2$MoO$_4$·2H$_2$O (0.484 g) were dispersed with 44 mL of deionized water in beaker. After that, the partial homogeneous suspension was transferred a 50 mL Teflon-lined stainless-steel autoclave. Subsequently, a piece of pretreated CC (2 cm × 2 cm) was placed and immersed the above aqueous solution. The autoclave was sealed and maintained for 24 h at the temperature of 220 °C and naturally got coolly at room temperature. At last, the resultant sample was moved out and dried at 60 °C for 6 h in an oven after washed with deionized water and ethanol in succession.

The detection of extracellular H$_2$O$_2$ released from cells: In this work, Lung cancer A549 cells (obtained from Qufu Normal University, Shandong, China) were chosen and cultured in Dulbecco’s Modified Eagle Medium (DMEM) solution consisting of 1% penicillin, 1% streptomycin and 10% (V/V) heat-inactivated fetal bovine serum (FBS) in the Petri dish in a humidified incubator (37 °C, 5% CO$_2$) and subcultured every 3 days. Then the cells being exposed to trypsin and cultured in fresh DMEM.
after growing to 80 %-90 % confluence. After that, the cells were collected from culture medium by centrifugation and washed three times carefully by PBS solution at 1000 rpm for 3 min. Finally, the obtained packed cells were dispersed into 20 mL PBS saturated with nitrogen for preparation of the detection of electrochemical responses. After the background noise was steady, the changed current signal of H₂O₂ released from A549 cells upon injecting PMA was recorded and analyzed. In addition, two control experiments were performed under the identical experimental condition but without living cells or PMA.

**Characterization:** Powder X-ray diffraction (XRD) analysis was obtained on a MiniFlex600 X-ray diffractometer (Rigaku, Japan). Scanning electron microscopy (SEM) images were achieved with a Sigma 500 VP electron microscope (Carl Zeiss AG, Germany). Transmission electron microscopy (TEM) photographs was performed on a 80-200kV/JEM-2100PLUS microscope (JEOL, Japan). X-ray photoelectron spectroscopy (XPS) data of the samples was collected on an ESCALABMK II x-ray photoelectron spectrometer using Mg as the exciting source.

**Electrochemical measurement:** All electrochemical measurements were recorded on a CHI 660E electrochemical workstation (CH Instruments, Shanghai, China). The typical cyclic voltammetry (CV) and current- time curves were carried out with a conventional three-electrode cell system at room temperature: the bare CC and MoS₂/CC electrode with an average area of 0.04 cm² were acted as working electrode respectively as control. A graphite rods electrode was used as the counter electrode and a saturated calomel electrode (SCE) was exploited as the reference electrode.

**LOD calculations:** The LOD is employed by the following equation: LOD = 3σ/b. (σ is the standard deviation of the blank samples (10 times), b is the slope of the calibration curve.
Fig. S1. The SAED pattern of MoS$_2$/CC
Fig. S2. EDS spectrum for MoS$_2$/CC
Fig. S3. (a) XPS survey spectra of MoS$_2$/CC. XPS spectra of MoS$_2$/CC in the (b) Mo 3d and (c) S 2p regions.
Fig. S4. Amperometric responses of MoS$_2$/CC at various potentials with continuous addition of 1 mM H$_2$O$_2$ in 0.1 M PBS (pH = 7.4).
Fig. S5. Amperometric response of MoS$_2$/CC under the addition of 1 mM H$_2$O$_2$ followed by some common interferences in 0.1 M PBS (pH = 7.4). Applied potential: -0.5 V.
**Fig. S6.** The CV measurements obtained at MoS$_2$/CC for 20 successive assays in 0.1 M PBS containing 1.0 mM H$_2$O$_2$. 
Table. S1. Comparison of analytical performances of MoS$_2$/CC with other H$_2$O$_2$ sensors.

<table>
<thead>
<tr>
<th>Electrode</th>
<th>LOD (μM)</th>
<th>Sensitivity (mA mM$^{-1}$ cm$^{-2}$)</th>
<th>Liner range (μM)</th>
<th>Ref</th>
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<tr>
<td>MoS$_2$/CC</td>
<td>1.0</td>
<td>5.3/3.6</td>
<td>5.0–235/435–3000</td>
<td>This work</td>
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<tr>
<td>MoS$_2$@MgFe$_2$O$_4$</td>
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<td>2.5–300</td>
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<td>SDS–MoS$_2$ NPs</td>
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<td>–</td>
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<td>CoTPP/RGO</td>
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<td>0.0042</td>
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<td>NiHCF/CS/CNTs</td>
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<td>Co$_3$N NW/TM</td>
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<td>Pd nanocubes</td>
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<td>0.23–2200/2200–14220</td>
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References


