Electronic Supplementary Information

Experimental section

Materials: $Na_2MoO_4 \cdot 2H_2O$, CH_4N_2S, HNO_3, C_2H_5OH, CH_3COOH, NaOH, HCl, Na_2SO_4. CC was provided by Hongshan District, Wuhan Instrument Surgical Instruments business. The water used throughout all experiments was purified through a Millipore system. All the reagents were used as received without further purification.

Preparation of MoN NA/CC: MoN NA/CC was prepared as follows. 0.242 g $Na_2MoO_4 \cdot 2H_2O$ was dissolved in 22 mL deionized water in a 50 mL beaker, into which 0.305 g thiourea was added. After gently stirred for 30 min, the solution was then transferred to a 50 mL Teflon-lined stainless steel autoclave, and a piece of CC (2 cm × 3 cm) was immersed into the autoclave contained solution. The autoclave was sealed and maintained at 220 °C for 24 h in an electric oven. After the autoclave cooled down naturally to room temperature, the precursor on CC was taken out and rinsed with deionized water several times, then dried in air at 60 °C in air. To prepare MoN NA/CC, the precursor was placed in the furnace and heated to 800 °C for 3 h with a heating speed of 5 °C min^{-1} under a flowing NH_3 atmosphere. The system was allowed to cool down to room temperature naturally still under a flowing NH_3 atmosphere. Finally, the black MoN NA/CC was collected.

Characterizations: Powder X-ray diffraction (XRD) data were acquired on a RigakuD/MAX 2550 diffractometer with Cu Kα radiation (λ=1.5418 Å). Scanning electron microscopy (SEM) images were collected from the tungsten lamp-equipped SU3500 scanning electron microscope at an accelerating voltage of 20 kV (HITACHI, Japan). Transmission electron microscopy (TEM) measurements were made on a Hitachi H-8100 electron microscopy (Hitachi, Tokyo, Japan) with an accelerating voltage of 200 kV. X-ray photoelectron spectroscopy (XPS) measurements were carried out on an ESCALABMK II X-ray photoelectron spectrometer using Mg as the exciting source.

Electrochemical measurements: Electrochemical measurements were performed with a CHI 660E electrochemical analyzer (CH Instruments, Inc., Shanghai) in a traditional three electrode system, using MoN NA/CC as working electrode, platinum wire as counter electrode and saturated Ag/AgCl as reference electrode. All tests were carried out at room temperature.

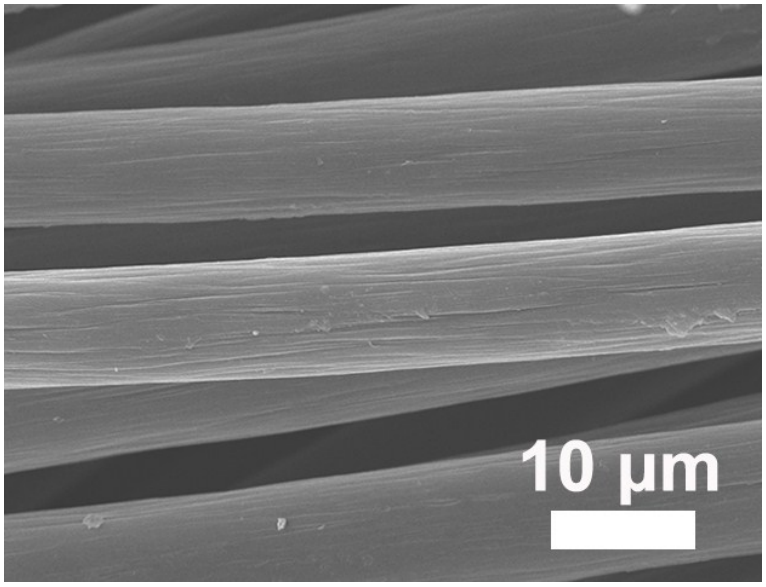

Fig. S1. SEM image for bare CC.

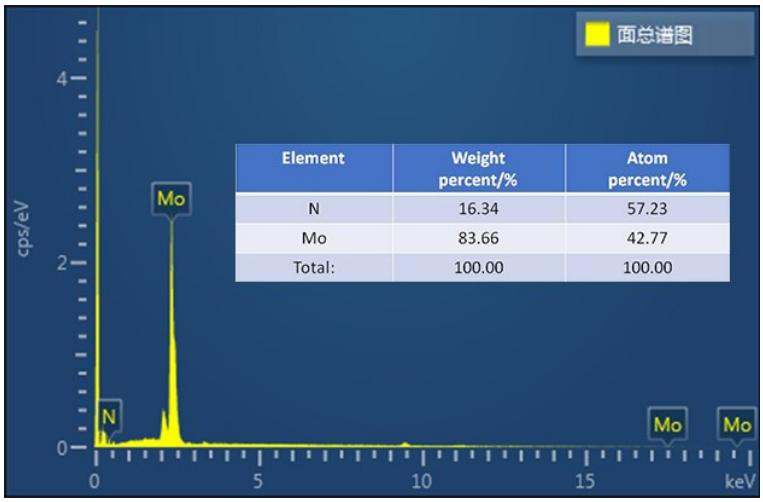

Fig. S2. EDX spectrum of MoN.

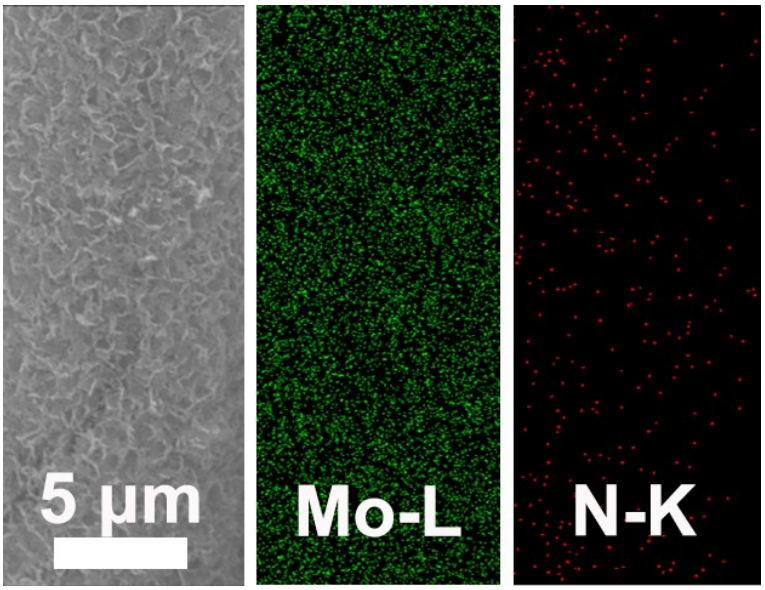

Fig. S3. SEM and EDX element mapping images of Mo and N elements in MoN.

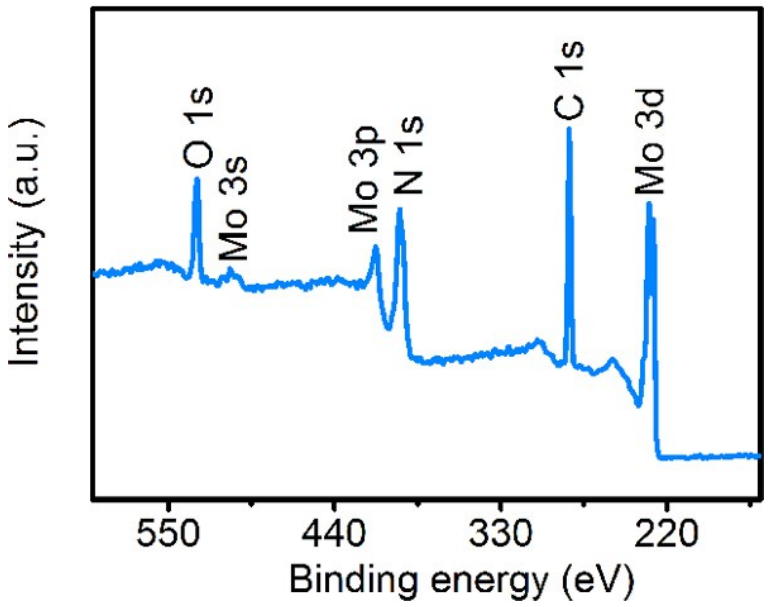

Fig. S4. XPS survey spectrum for MoN.

Table S1. Comparison of the MoN NA/CC performances for nitrite sensing with reported electrocatalysts.

Catalyst	Sensitivity	linear range (μM)	Detection limit(μM)	Ref.
MoN NA/CC	**4319 μA mM^{-1} cm^{-2}**	**1.0–5000**	**0.003**	This work
Cu–NDs/rGO	214 μA mM^{-1} cm^{-2}	1.25–13000	0.4	1
Nano Au/Ch	354 μA mM^{-1} cm^{-2}	0.4–750	0.1	2
Cu–CoTCPP/MWCNTs/GCE	439 μA mM^{-1} cm^{-2}	2.5–1100	0.17	3
PTB/GCE	470 μA mM^{-1} cm^{-2}	0.1–15.2	0.05	4
RGO–MWCNT–Pt/Mb/RDGCE	165.1±26 μA mM^{-1} cm^{-2}	1–12000	0.93	5
CuS–MWCNT	131.2 μA mM^{-1} cm^{-2}	1–8100	0.33	6
Ag–PAMAM	265 μA mM^{-1} cm^{-2}	4–1440	0.4	7
EAG/SPCE	126 μA mM^{-1} cm^{-2}	0.1–16400	0.038	8
Au/ZnO/MWCNTs	/	0.78–400	0.4	9
AuCu NCNs	17.55 μA mM^{-1}	10–4000	0.2	10
CR–GO/GCE	26.7 μA mM^{-1}	8.9–167	1	11
AuNPs/SG	45.44 μA mM^{-1}	10–3960	0.2	12

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