## Supplemental Information for: Nanofibers Decorated with High-Entropy Alloy Particles for the Detection of Nitrites

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Figure S1 ICP results of the FeCoNiCuAl/CNFs.



**Figure S2** XPS spectra of the synthesized FeCoNiCuAl/CNFs: (a)C 1s, (b) N 1s, (c) O 1s.



**Figure S3** The CV diagram of GCE, CNFs/GCE, FeCoNi/CNFs/GCE, FeCoNiCu/CNFs/GCE, FeCoNiAl/CNFs/GCE, FeCoNiCuAl/CNFs/GCE in 0.10 M PBS (pH=7) without nitrite, and the scanning rate was 100 mV/s.



**Figure S4** (a) CV diagram of FeCoNiCuAl/CNFs/GCE in PBS solution with pH=5-9 and nitrite concentration of 1 mM. (b) Change of peak current.



**Figure S5** The linear relationship between the peak current of the anode and the square root of the scanning rate.



Figure S6 (a) DPV spectrum of 0-200  $\mu$ M nitrite. (b) Linear curve of peak current and nitrite concentration.



**Figure S7** (a) DPV diagram of FeCoNiCuAl/CNFs at five glassy carbon electrodes at 1 mM nitrite concentration. (b) Peak current.



**Figure S8** CV diagram and peak current of FeCoNiCuAl/CNFs at 1 mM nitrite concentration on five glassy carbon electrodes (scan rate 50 mV/s).



Figure S9 The weekly DPV peak current of FeCoNiCuAl/CNFs/GCE in 0-28 days.



Figure S10 The weekly CV peak current of FeCoNiCuAl/CNFs/GCE in 0-28 days.



Figure S11 DPV spectra of FeCoNiCuAl/CNFs/GCE in 100  $\mu M$  NaNO\_2 and 2000  $\mu M$  interfering substances.



**Figure S12** DPV spectra of FeCoNiCuAl/CNFs/GCE in 1mM NaNO2 and 5 mM interfering substances.



**Figure S13** (a) The amperometric response of FeCoNiCuAl/CNFs/GCE in 0.10 M PBS (pH=7) at 0.72, 0.74, 0.76, 0.78 V, respectively. (b) Ampere current response diagram at 0.76 V.



**Figure S14** The linear curve of the amperometric response of FeCoNiCuAl/CNFs/GCE to nitrite concentration.



Figure S15 The current response of FeCoNiCuAl/CNFs/GCE to 2000  $\mu M$  nitrite for 1800 s.



**Figure S16** Amperometric response of FeCoNiCuAl/CNFs/GCE in 0.10 M PBS (pH=7) with 0.4 mM nitrite and different 2 mM Common electrochemical detection substances.

**Table S1** Analysis of nitrite in real samples by DPV method based onFeCoNiCuAl/CNFs/GCE.

Sample	Nitrite concentration (µM)	Spiked nitrite concentration (µM)	Measured nitrite concentration (µM)	Recovery (%)
Tap water	Not detected	100	97.2	97.2
River water	Not detected	100	96.5	96.4
Milk	Not detected	100	95.7	95.7

**Table S2**Analysis of nitrite in real samples by I-t method based onFeCoNiCuAl/CNFs/GCE.

Sample	Nitrite concentration (µM)	Spiked nitrite concentration (µM)	Measured nitrite concentration (µM)	Recovery (%)
Tap water	Not detected	100	100.4	100.4
River water	Not detected	100	93.0	93.0
Milk	Not detected	100	92.0	92.0