

## Supporting Information

# **A simple approach to synthesize NiFe LDH-Nb<sub>2</sub>C MXene for enhanced electrochemical nitrogen reduction reactions by synergistic effect**

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## **Catalyst characterization**

XRD with a scanning rate of 5 degrees  $\text{min}^{-1}$  was conducted to characterize the phase composition and crystal structure of Ni@Nb<sub>2</sub>C MXene at accelerating voltage of 35 kV and tube current of 25 mA, respectively. XPS was used to determine the chemical composition and valence of the catalyst. The surface morphology of Ni@Nb<sub>2</sub>C MXene was observed using SEM (Nova Nano SEM 450). Moreover, the nano-structural characteristic and elemental distribution of Ni@Nb<sub>2</sub>C MXene were analyzed by the TEM technique.

## **Electrochemical measurement**

Electrocatalytic performance of Ni@Nb<sub>2</sub>C MXene was evaluated using the CHI760E electrochemical workstation (Shanghai Chenhua) in a three-electrode system with glassy carbon (effective area 0.196  $\text{cm}^2$ ), Ag/AgCl, and platinum wire as the working, reference, and auxiliary electrodes, respectively. The electrolyte was 0.1 M KOH aqueous solution, while the preparation process of work cathode was as follows. For example, 10 mg catalyst and 30  $\mu\text{L}$  Nafion were dispersed into 1 mL isopropanol under the ultrasonic condition to obtain a uniform slurry, and then 20  $\mu\text{L}$  slurry was dropped onto the glassy carbon electrode. Before the test, the ultrapure N<sub>2</sub> was successively bubbled into the electrolyte for 2 h to achieve its saturation.

## **Calculation of NH<sub>3</sub> yield and FE**

The indophenol blue reaction was carried out to determine NH<sub>3</sub> yield.<sup>37</sup> Specifically, a 2 mL electrolyte was firstly extracted from the electrocatalytic NRR system. Subsequently, 2 mL of 1 M sodium hydroxide solution containing 5 wt% (weight

percentage) sodium salicylate and 5 wt% sodium potassium tartrate was added to the above electrolyte, followed by adding 1 mL of 6 wt% sodium hypochlorite solution and 0.2 mL of 1 wt % sodium nitroferricyanide solution. After quiescence for 1 h at room temperature, the absorption peak ( $\lambda \approx 655$  nm) of generated  $\text{NH}_4^+$  was detected with a UV-vis spectrophotometer.

The  $\text{NH}_3$  yield (*Yield*) was calculated by the following equation:

$$\text{Yield} = \frac{c \times V}{t \times A} \quad (1)$$

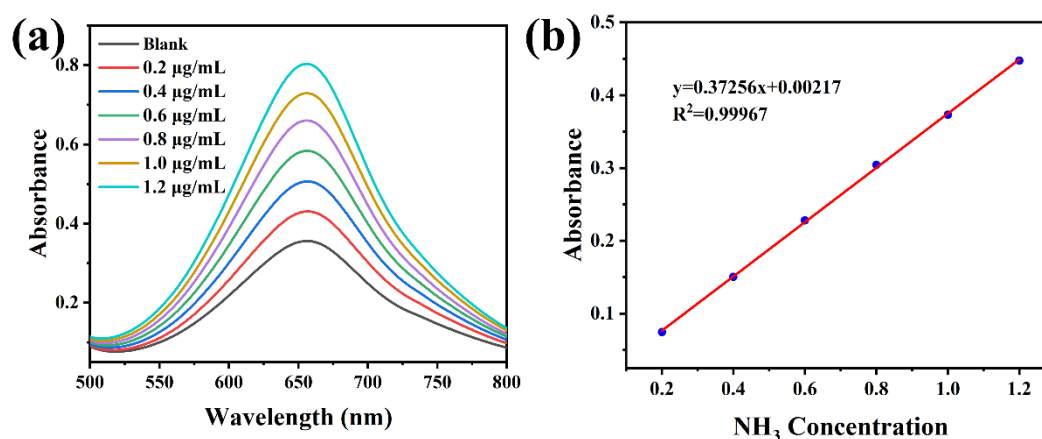
where  $c$  is the  $\text{NH}_4^+$  concentration,  $V$  is the electrolyte volume,  $t$  is the NRR time, and  $A$  is the effective area of electrode.

The *FE* value was calculated according to the following equation:

$$\text{FE} = \frac{3F \times c \times V}{17Q} \quad (2)$$

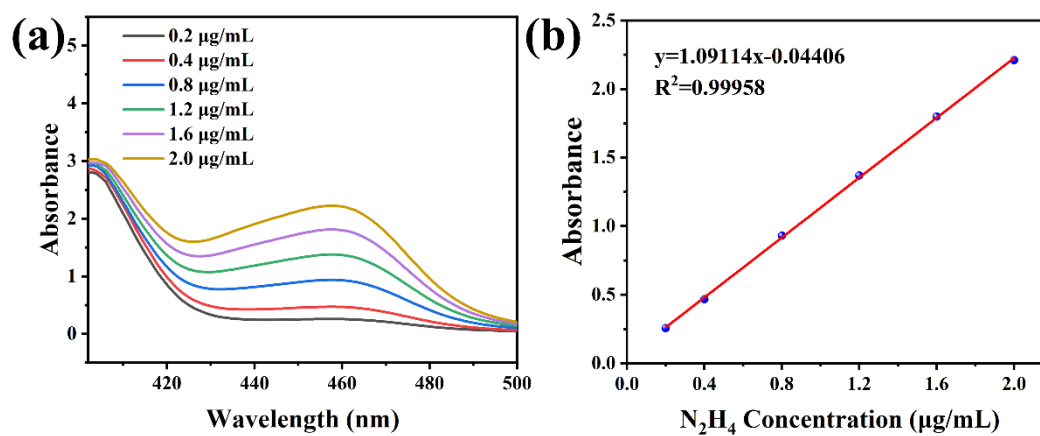
where  $F$  is the Faradic constant, and  $Q$  is the total quantity of electric charge.

A series of standard  $\text{NH}_4\text{Cl}$  solutions was used to calibrate the concentration–absorbance curve, giving the following linear fitting curve:  $y = 0.37256x + 0.00217$ ;  $R^2 = 0.99967$ .

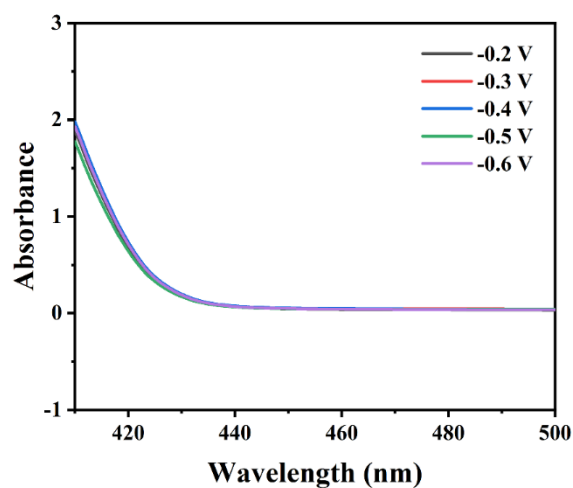


**Figure S1.** (a) UV-Vis absorption spectra of standard  $\text{NH}_4^+$  solutions with different concentrations; (b) Standard curve for calculating  $\text{NH}_4^+$  concentration

A series of standard  $N_2H_4$  solutions was used to calibrate the concentration–absorbance curve, giving the following linear fitting curve:  $y = 1.09114x + 0.04406$ ;  $R^2 = 0.99958$ .



**Figure S2.** (a) UV-Vis spectra for the known concentration of  $N_2H_4$  solutions, and (b) Standard curve for calculating  $N_2H_4$  concentration



**Figure S3.** UV-Vis absorption spectra of  $N_2H_4$  at different potentials