Electronic Supplementary Material (ESI) for Physical Chemistry Chemical Physics.

Metal Ni-loaded g-C3N4 for enhanced photocatalytic H2 evolution activity: the change of surface band bending

Lingling Bi,a Dandan Xu,a Lijing Zhang,a Yanhong Lin,a Dejun Wang,a,b and Tengfeng Xie*a

a College of Chemistry, Jilin University, Changchun 130012, People's Republic of China.

b Department of Chemistry, Tsinghua University, Beijing 100084, People's Republic of China.

Photocatalytic activity of the Ni10-P

As for the Ni10 placed about one month later, named as Ni10-P, we carried out the photocatalytic H_2-production activity in the same condition of Ni10. The photocatalytic H_2-production performance is shown in Fig. S1. We can easily find that the activity of H_2-production of Ni10-P is higher than the Ni10. May be with the reason of synergistic effect of Ni and NiO[1-2].

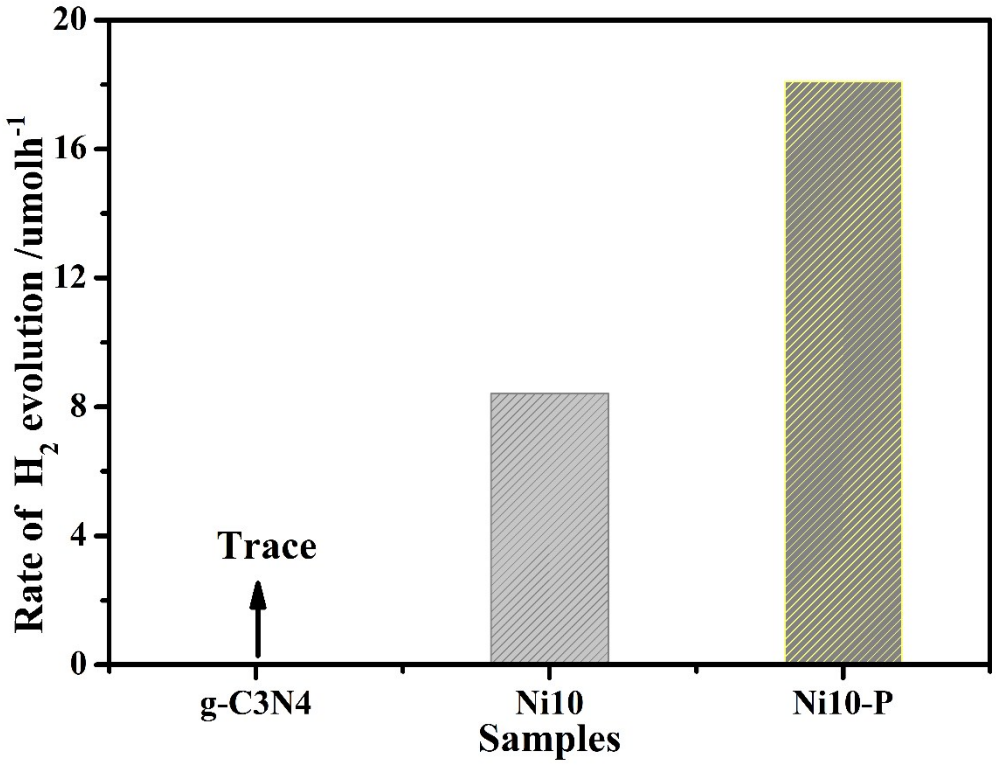

Fig. S1 Comparison of the photocatalytic activity of g-C_3N_4, Ni10 and Ni10-P for H_2 production using triethanolamine as scavenger under simulated sunlight irradiation (500W Xe lamp).

XPS measurements of Ni10-P

Then the XPS was measured to analyze chemical composition and identify the chemical status of Ni element in the sample. As shown in Fig. S2a, the fully scanned spectra indicates the Ni, O, C and N exist in Ni10-P. As shown in Fig. S2b, Ni 2p signal could be deconvoluted into five peaks[3-4]. The binding energies at 855.6 eV and 861.7 eV are attributed to the Ni $2p_{3/2}$ peaks, and the 880.8 eV and 873.3 eV are assigned to the Ni $2p_{1/2}$ peaks, which confirms the presence of Ni (II). Meanwhile, the peak at 874.2 eV may be corresponding to the Ni^o. The XPS dates show that the Ni is partly oxidated to NiO after placed a period of time. Then the other peaks of Ni10-P are similar to the Ni10.

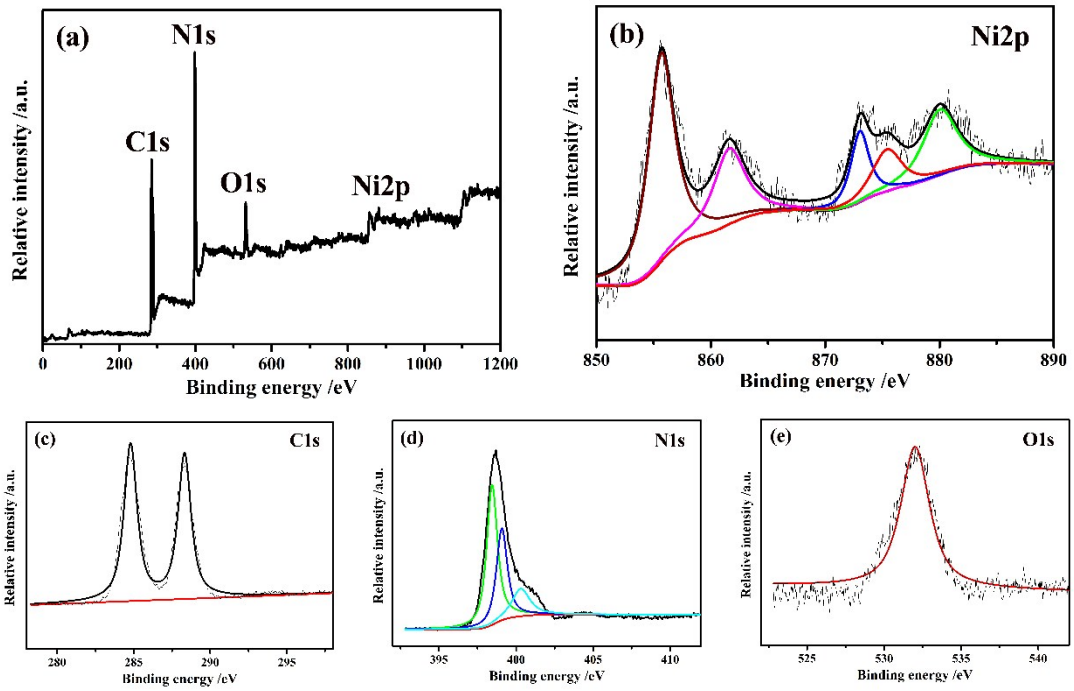

Fig. S2 XPS spectrum of (a) survey scan; (b) Ni2p; (c) C 1s; (d) N 1s and (e) O 1s binding energy regions of Ni10-P.

Quantum efficiency of Ni10

We use a method of the light quantum of ferric oxalate, measured absolute numbers of photons by means of chemical actionometry known quantum yield and calculate the quantum efficiency of hydrogen production.[5,6]

The fundamental is Fe^{3+} is reduced to Fe^{2+} after absorbing a certain wavelength, and then generates red solution after the Fe^{2+} encounters the 1, 10-phenanthroline. Quantitative analysis was made by the spectrophotometer.

The experimental method is following:

The 300 W mercury lamp used as light source irradiated on the photocatalytic reactor with $K_3[Fe(C_2O_4)_3]$ solution (V_0=100 mL) for 60 s and 20 s, respectively. Took 5 mL (V_1) out of the V_0 to a brown glass flask volumetric, added 10 mL 1, 10-phenanthroline and 10 mL HAc-NaAc buffer solution, then diluted to 50 mL (V_2=50 mL). Spectrophotometer was used to measure its absorbance A_t about λ max = 510 nm after placed 30 min in the dark. Finally measured the absorbance A_0 of the samples without irradiation, with the same steps above. Furthermore, the photocatalytic H_2 production also was carried out at the same under the same condition of the 300 W Hg irradiation, and hydrogen content was analyzed by gas chromatograph (GC-2014C, Shimadzu, Japan, TCD, argon as a carrier gas and 5 Å molecular sieve column).

The quantum yield of photocatalytic hydrogen production (ϕ) was about 2.19%, calculated based on the equation:

$$\phi_{H_2} = \frac{2N_{H_2}N_A}{N} \times 100\% \quad (1\text{-}1)$$

$$N = \frac{(A_t - A_0)V_2V_0N_A}{\varepsilon L V_1 \phi_{Fe^{2+}} t} \quad (1\text{-}2)$$

where "N_{H_2}" is the rate of photocatalytic H_2 production, mol/s; "N_A" is the avogadro constant; "N" is the number of incident photon ; "ε" is the molar absorption coefficient of Fe^{2+}; "L" is the thickness of cuvette; "$\phi_{Fe^{2+}}$" is 1.21 (the quantum efficiency of 300 W mercury lamp at λ_{max} = 365nm); "t" is the time for irradiation, s.

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