α-Fe_2O_3 Nanoparticles/Iron-Containing Vermiculite Composites: Structural, Textural, Optical and Photocatalytic Properties

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Supplementary materials

1. Photocatalytic experiment

The photocatalytic experiments were carried out in a homemade batch mixed photoreactor (Figure S1).

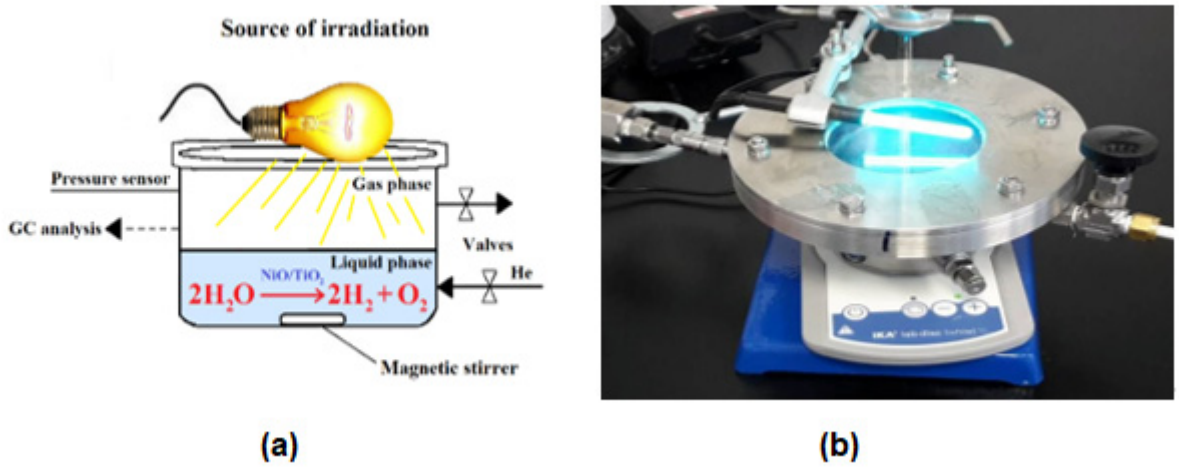

Figure S1. Scheme (**a**) and real photo (**b**) of photocatalytic experiment.

The photoreactor was made from stainless steel. Photoreactor diameter and height was 0.075 and 0.06, respectively. The volume of photoreactor was 348 mL. Reaction mixture contained 100 mL of 50% methanol with a photocatalyst (0.1 g) and was saturated by helium, to purge the air. An 8W Hg lamp (peak intensity at 254 nm wavelength; Ultra-Violet Products Inc.) was used as the irradiation source which was placed on a quartz glass window in the top of the photoreactor in horizontal position. Lamp intensity was 1.2 $W.m^{-3}$ ($J . s^{-1} . m^{-2}$). The photoreactor was tightly closed and before the start of the reaction (switching on the UVC lamp), a gaseous sample was taken (at time 0 h) through septum by syringe. The all gaseous samples were analyzed by a gas chromatograph (Shimadzu Tracera GC-2010Plus) equipped with BID (barrier discharge ionization detector). The reaction mixture was irradiated at certain time intervals (0-4 h) and samples were taken at 1, 2, 3 and 4 h for analysis. Each measurement was repeated until its reproducibility.

2. Results of photocatalytic experiments

To confirm that hydrogen is generated only in the presence of photocatalyst and UV irradiation, two blind experiments were performed: (i) methanol-water mixture and photocatalyst without UV irradiation and (ii) methanol-water mixture and UV irradiation without photocatalyst. In neither case was hydrogen detected.

It is important to mention that all tests (including repeated tests) were always conducted with the same batch of each photocatalyst in order to determine at least short time stability and reusability.

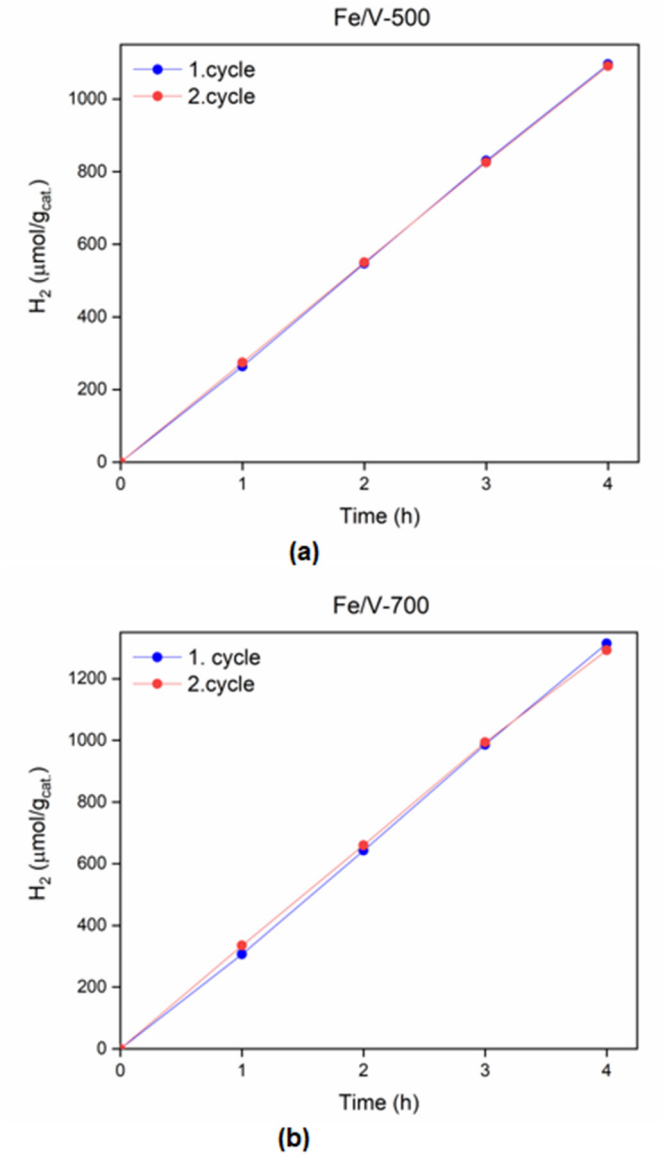

Figure S2. Repeated photocatalytic experiments in presence Fe/V-500 (**a**) and Fe/V-700 photocatalysts (**b**).

3. Example of calculation for Fe/V-700:

The gas chromatograph gives the results of H_2 amount in the ppm (parts per million) unit. However, it is necessary to have the results in μmol per gram of the photocatalyst to construct a graph. That was calculated using the ideal gas law equation where p is the pressure in Pa, V is the volume in m^3, R is the ideal gas constant (8.314 $J \times K^{-1} \times mol^{-1}$), T is the temperature in K, and n is the number of moles in mol (Equation 1).

$$p \times V = n \times R \times T \quad (1)$$

By rearranging, we get the equation (Equation 2).

$$n = \frac{p \times V}{R \times T} \quad (2)$$

Because we need the results in μmol per gram of the photocatalyst, this information has to be added to the equation along with the amount of H_2.

As a result, we get the equation where c is the concentration of H_2 in ppm, m is the mass of the photocatalyst in g, V is the volume in cm^3, and n is the amount of H_2 in $\mu mol/g_{cat}$ (Equation 3).

$$n = \frac{p \times V \times 10^{-6} \times c}{R \times T \times m} \quad (3)$$

For example, to calculate the amount of H_2 produced after 4 hour of photocatalysis with the Fe-V/700 photocatalyst, we will construct equation (Equation 4).

$$n = \frac{129800 \times 247.8 \times 10^{-6} \times 10296}{8.314 \times 0.1 \times 303.15} \quad (4)$$

$$n \approx 13159\ \mu mol/g_{cat}$$