

Electronic Supplementary Information

Preparation of Fe₂O₃/MIL-53(Fe) composite by partial thermal decomposition of MIL-53(Fe) nanorods and its photocatalytic activity†

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Preparation of Photocatalysts

All chemicals were analytical grade. MIL-53(Fe) was prepared by using FeCl₃, terephthalic acid (H₂BDC) and DMF with a molar ratio 1:1:30 was mixed in a 100 mL Teflon lined stainless steel autoclave, the mixture was then heated at 180 °C for 24 h. A yellow MIL-53 (Fe) powder having foreign molecules in the pores was obtained by centrifuge at 6000 rpm for 10 min. To remove the foreign DMF, the solid was washed with deionized water followed by continuous stirring in methanol for 3 days. Then the solid was filtered and dried at 150 °C in air oven for 12 h to obtain MIL-53 (Fe). Fe₂O₃/MIL-53(Fe) composites were prepared in accordance to thermogravimetric analysis (TGA) curve. FM-1 was prepared by thermal decomposition of MIL-53 (Fe) up to 450 °C at a heating rate of 5 °C/min in a N₂ atmosphere in a tubular furnace, whereas FM-2 was prepared by thermal decomposition of MIL-53(Fe) up to 800 °C under similar condition.

Characterization

Powder X-ray diffraction (XRD) patterns of all catalysts were obtained by using a PAN analytical diffractometer (model: PW-3050/60), using Cu-K α radiation at 40 kV and 30mA with 2 θ angle scanning from 5° to 70°. A TGA Q50 instrument was used for thermogravimetric analysis from 150°C to 850°C. A heating rate of 5 °C/min was used in a N₂ atmosphere. The surface morphology of catalysts was analysed using a scanning electron microscope (JEOL, model ESM-5800). Different chemical bonds in the catalysts were analyzed using Fourier transform infrared (FTIR) spectroscopy (PerkinElmer, model: Spectrum 100). A UV-Vis spectrophotometer (Perkin Elmer lambda 25, Germany) was used to record the absorbance spectra of all samples. Specific surface area and pore size distribution were calculated from the N₂ adsorption-desorption

isotherms, according to Brunauer-Emmett-Teller (BET) method and Barrett-Joyner-Halenda (BJH) method respectively. N₂ adsorption- desorption isotherms were collected using a Quanchrome instruments (model: AUTOSORB iQ, USA) by adsorption of nitrogen at 77K on 200 mg of sample previously degassed at 180 °C under vacuum.

Calculation of percent composition of FM-1

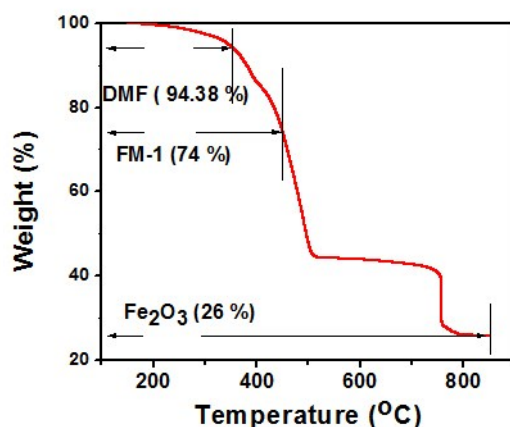


Fig. S1: Thermogravimetric Analysis of MIL-53(Fe).

Assumptions:

1. At 800 °C, MIL-53(Fe) totally converts to Fe₂O₃
2. Composites contains only MIL-53(Fe) and Fe₂O₃ as component (No intermediate product was formed)

Let us suppose total weight of catalyst sample is 1 gm.

0.9438 gmMIL-53(Fe) forms 0.2626 gm Fe₂O₃ at complete decomposition (0.0562 gm DMF).

So, x amount of MIL-53(Fe) forms 0.2755x (Fe₂O₃) ----- eq(1)

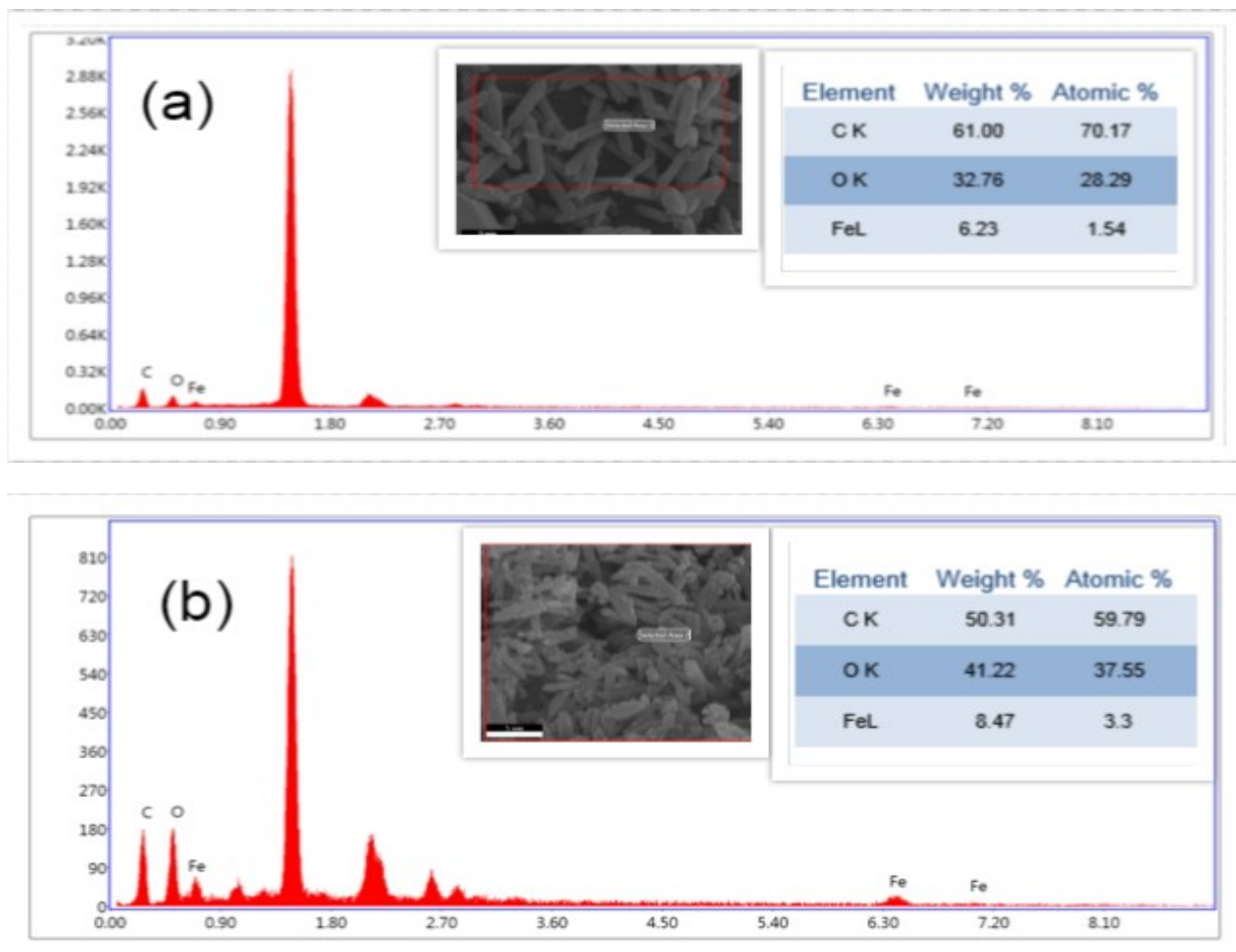
For FM-1 at 450 °C, let suppose amount of MIL-53(Fe) is x and amount of Fe₂O₃ is y

So, x + y = 0.74 -----eq(2),

Using eq(1),It can be rewritten as $x + (0.9438-x) * 0.2755 = 0.74$ (as $0.9438-x$ forms Fe_2O_3)

By solving equation 2, $x = 0.66$ and $y = 0.08$ ($\text{MIL-53}(\text{Fe}) = 89\%$ and $\text{Fe}_2\text{O}_3 = 11\%$)

EDS



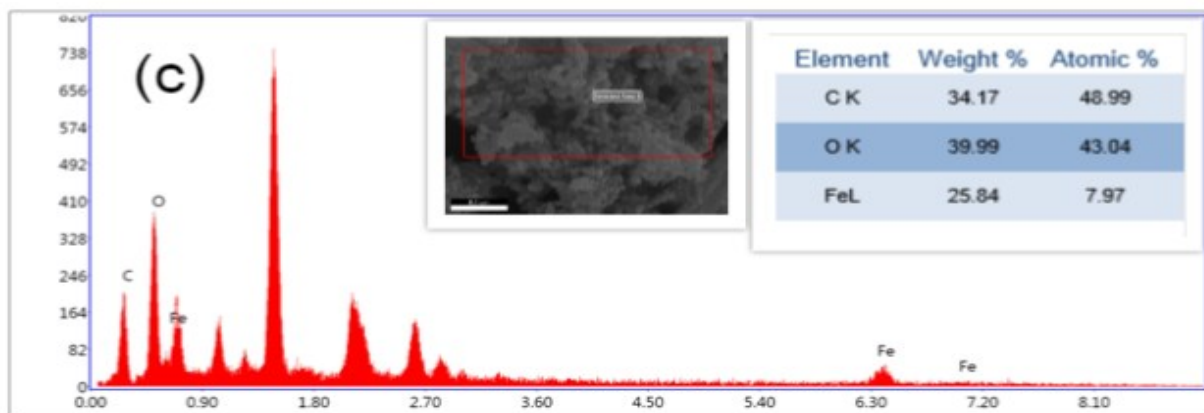


Fig. S2:EDS elemental mapping of (a) MIL-53(Fe) (b) FM-1 and (c) FM-2(Unnecessary peaks are due to presence of Aluminum foil, which was used for drop cast).

Table 1 : BET Surface Area and Pore Volume

S. No	Sample Name	Surface Area (m ² /g)	Pore Volume (cm ³ /g)
1	MIL-53(Fe)	47.9	0.06
2	FM-1	47.4	0.08
3	FM-2	12.8	0.02

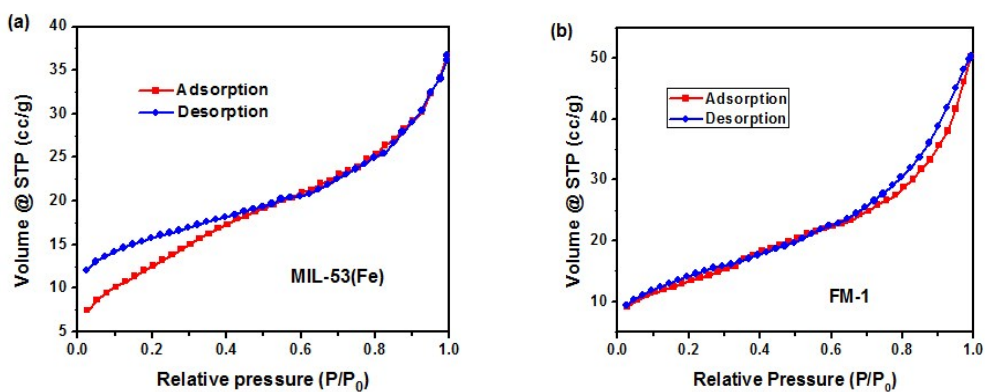


Fig. S3: N₂ isotherms of (a) MIL-53(Fe) and FM-1.



Fig. S4: Photo comparing appearance of MIL-53(Fe), FM-1 and FM-2.

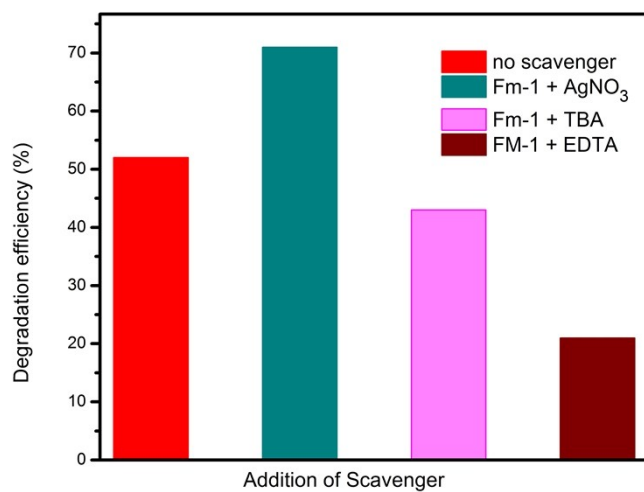


Fig. S5: Trapping experiment

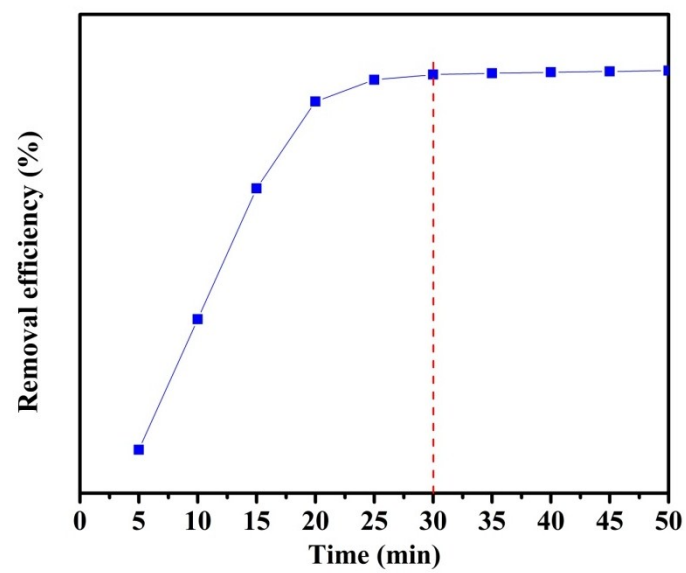


Fig. S6: Adsorption equilibrium study

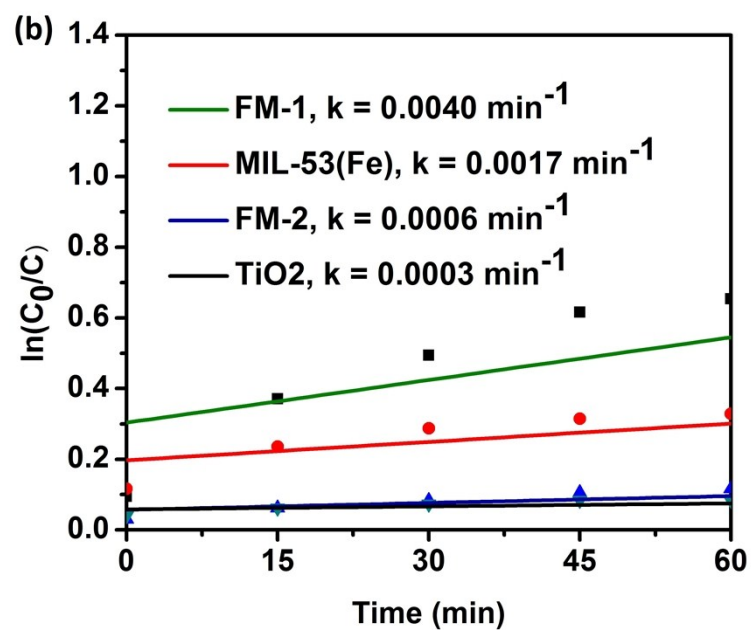
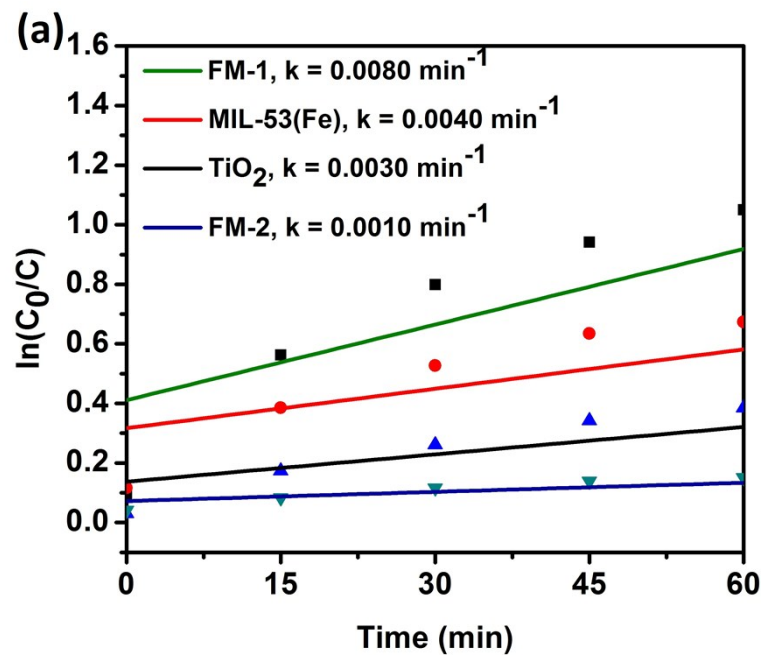


Fig. S7: Kinetic study under (a) UV irradiation (b) Visible irradiation.

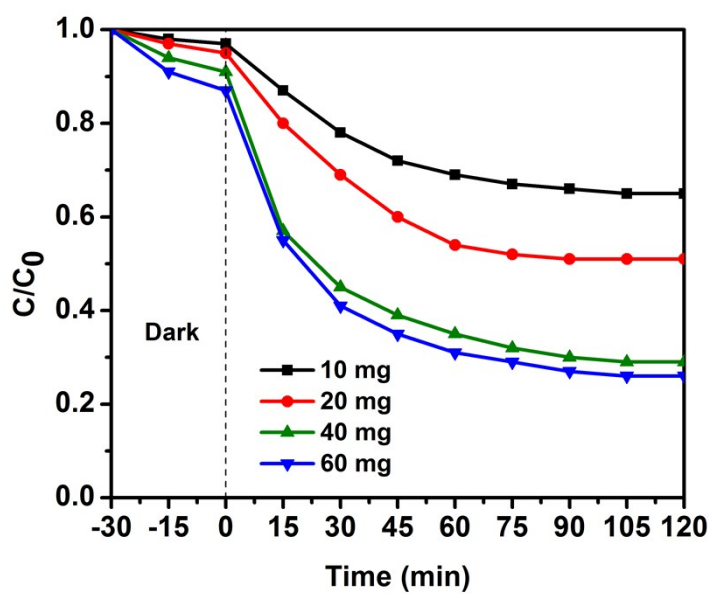


Fig. S8: Effect of Catalyst dose on degradation of MB.

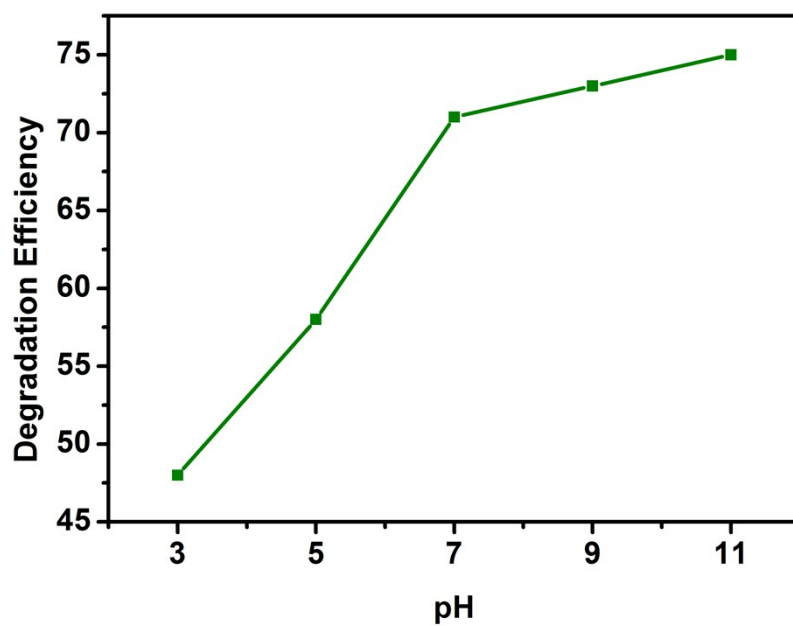


Fig. S9: Effect of pH on degradation of MB

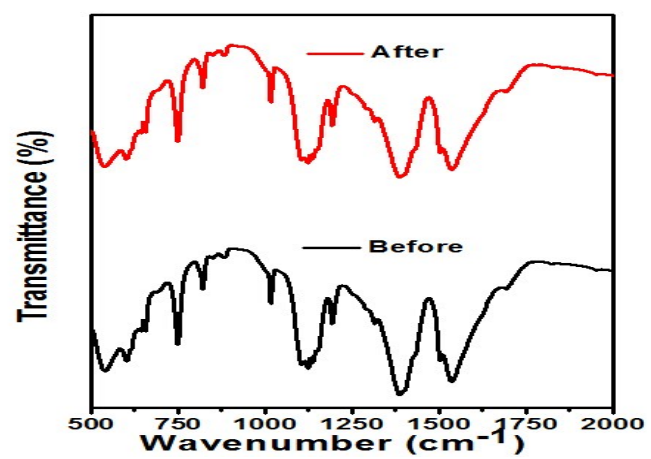


Fig. S10: FTIR spectra of FM-1 before use and after use.