Electronic Supplementary Information (ESI)

for

One-pot synthesis of magnetic particles-embedded porous carbon composites from metal-organic frameworks and their sorption properties

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General Methods

Solvents and all other chemicals were obtained from commercial sources and were used as received unless otherwise noted. All scanning electron microscopy (SEM) images were obtained using JEOL JSM-7001F field-emission SEM, and energy dispersive X-ray (EDX) spectra were obtained using a Hitachi SU 1510 SEM equipped with a Horiba EMAX Energy E-250 EDS system. All transmission electron microscopy (TEM) images were acquired on a FEI Tecnai G2 F30 ST at 300 kV. Elemental mapping images were obtained using an STEM attachment (Korea Basic Science Institute, Seoul, Korea). X-ray diffraction studies were conducted using a Rigaku Ultima IV equipped with a graphite-monochromated $Cu_{k\alpha}$ radiation source (40 kV, 40 mA). The X-ray photoelectron spectroscopy (XPS) measurements were performed on a Thermo Scientific K-Alpha KA1066 spectrometer using a monochromatic Al Kα X-ray source (*hv* = 1486.6 eV). Raman spectroscopy data were collected using a HORIBA Jobin Yvon LabRAM ARAMIS confocal Raman microscope at room temperature with a 0.5 mW YAG laser at 532 nm. UV-vis absorption spectra were obtained on a Shimadzu UV-1650PC spectrophotometer using quartz cells (10 x 4 mm light path). The adsorption isotherms of N_2 (77 K) were measured in the gaseous state using a BELSORP Max volumetric adsorption equipment. All gas-adsorption isotherms were measured after pretreatment under a dynamic vacuum at RT.

Synthesis of micro-sized hexagonal rods of Fe-MIL-88A

A precursor solution was prepared by mixing fumaric acid (42 mg, 0.36 mmol) and $Fe(NO_3)_3 \cdot 9H_2O$ (160 mg, 0.40 mmol) in 8 mL of *N,N*-dimethylformamide (DMF). The resulting mixture was placed in an oil bath (110 °C) for 30 min. The resulting Fe-MIL-88A was isolated by cooling the reaction mixture to room temperature, and subsequently washed with DMF and methanol *via* centrifugation-redispersion cycles.

Synthesis of nano-sized hexagonal rods of Fe-MIL-88B

A precursor solution was prepared by mixing 1,4-benzenedicarboxylic acid (30 mg, 0.18 mmol) and $Fe(NO_3)_3 \cdot 9H_2O$ (64 mg, 0.16 mmol) in 4 mL of *N,N*-dimethylformamide (DMF).[1] The precursor solution was then added to 4 mL of CH_3CN. The resulting mixture was placed in an oil bath (110 °C) for 40 min. The resulting Fe-MIL-88B was isolated by cooling the reaction mixture to room temperature, and subsequently washed with DMF and methanol *via* centrifugation-redispersion cycles.

Preparation of A-series and B-series of porous magnetic carbon composites

Fe-MIL-88A (140 mg) or Fe-MIL-88B (120 mg) was placed in a tube furnace and calcined up to various targeted-temperatures (600 ~ 1000 °C) under a nitrogen gas flow at a heating rate of 5 °C min^{-1}. After reaching the targeted-temperature, the resulting porous magnetic carbon composites were cooled to room temperature. The isolated amounts of A-600 ~ A-1000 were 52.5, 38.1, 37.0, 36.6 and 31.3 mg, respectively. The isolated amounts of B-600 ~ B-1000 were 54.2, 42.9, 42.5, 39.2 and 30.4 mg, respectively. The weight percentages of carbon within porous magnetic carbon composites were obtained from EDX data. The weight percentages of carbon within porous magnetic carbon composites were 22.05, 21.25, 20.02, 19.95 and 33.35 % for A-600 ~ A-1000, respectively, and 43.52, 43.27, 42.77, 41.09 and 39.09 % for B-600 ~ B-1000, respectively.

Adsorption of Methylene Blue (MB) from aqueous solution using porous magnetic carbons

5 mg of the A-800 or B-800 were added into 20 mL of MB aqueous solution (5 mg L^{-1}) and stirred to form the dispersed solution. UV-vis spectra were measured at various times after removing the MB adsorbed A-800 or B-800.

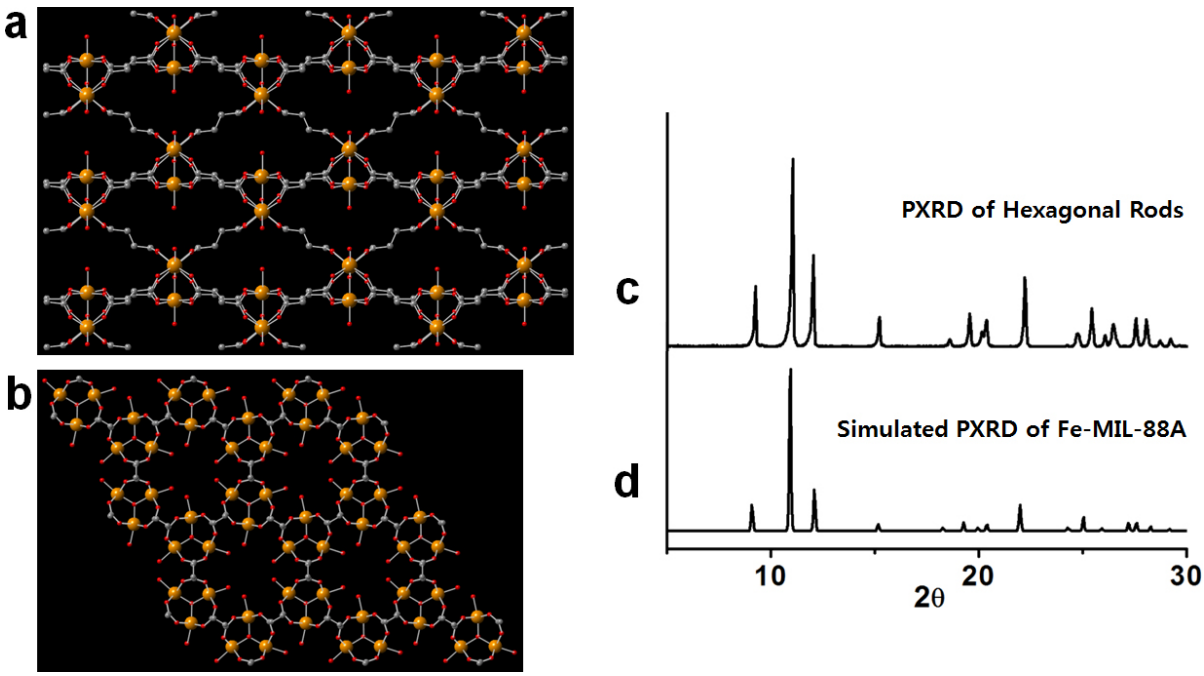

Fig. S1 Ball-and-stick representations of Fe-MIL-88A.[2] (a) A view of the *bc* plane. (b) A view of the *ab* plane. Gray, red and orange represent C, O and Fe, respectively. Hydrogen atoms and guest molecules are omitted for clarity. (c) PXRD pattern of the resulting hexagonal rods and (d) the simulated PXRD pattern of the reported Fe-MIL-88A.

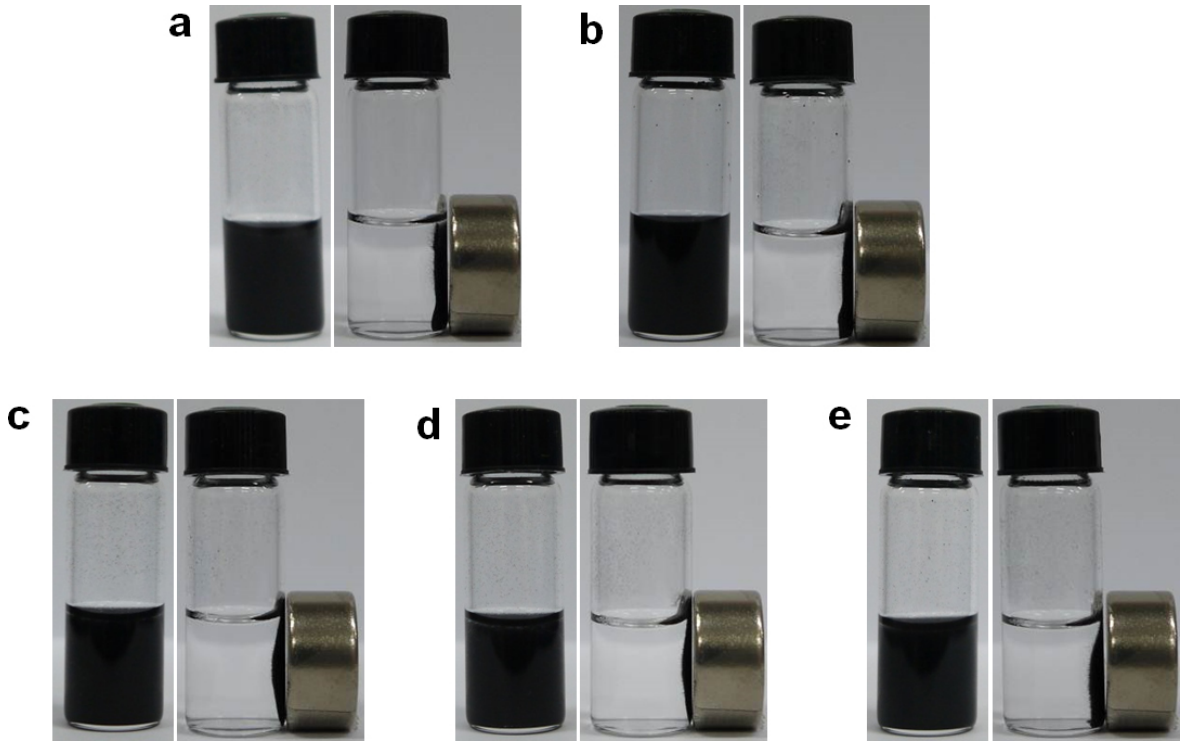

Fig. S2 Photographs of (a) A-600, (b) A-700, (c) A-800, (d) A-900 and (e) A-1000 dispersed in water and magnetically separated.

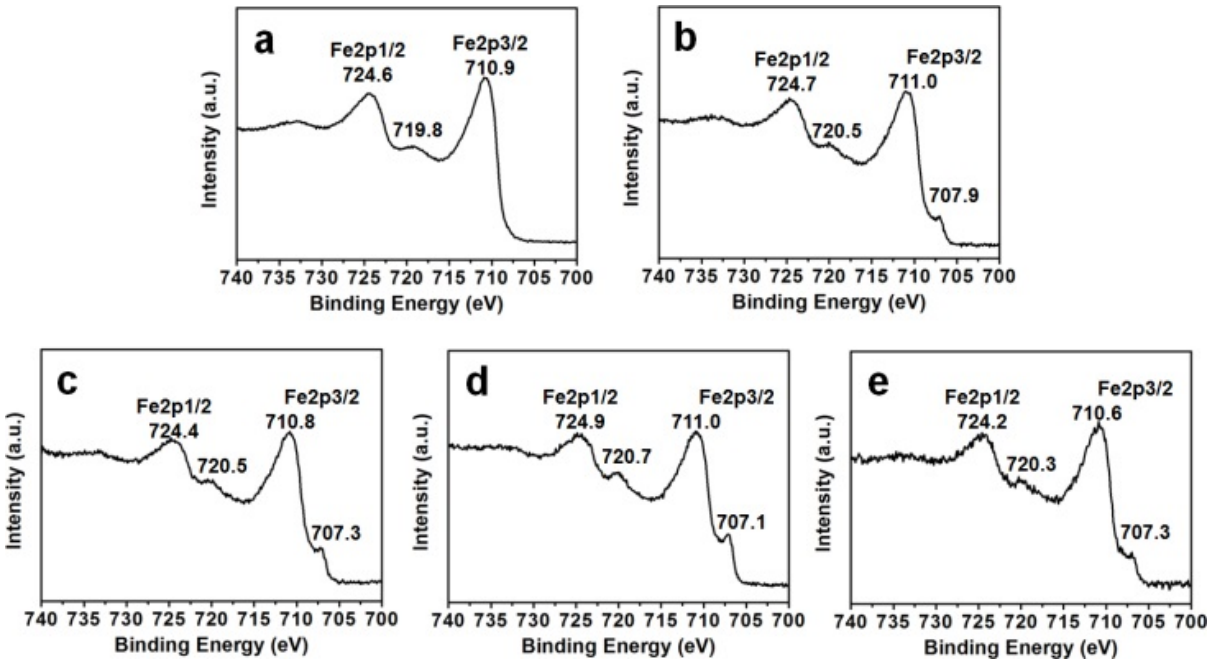

Fig. S3 XPS spectra of (a) A-600, (b) A-700, (c) A-800, (d) A-900 and (e) A-1000.

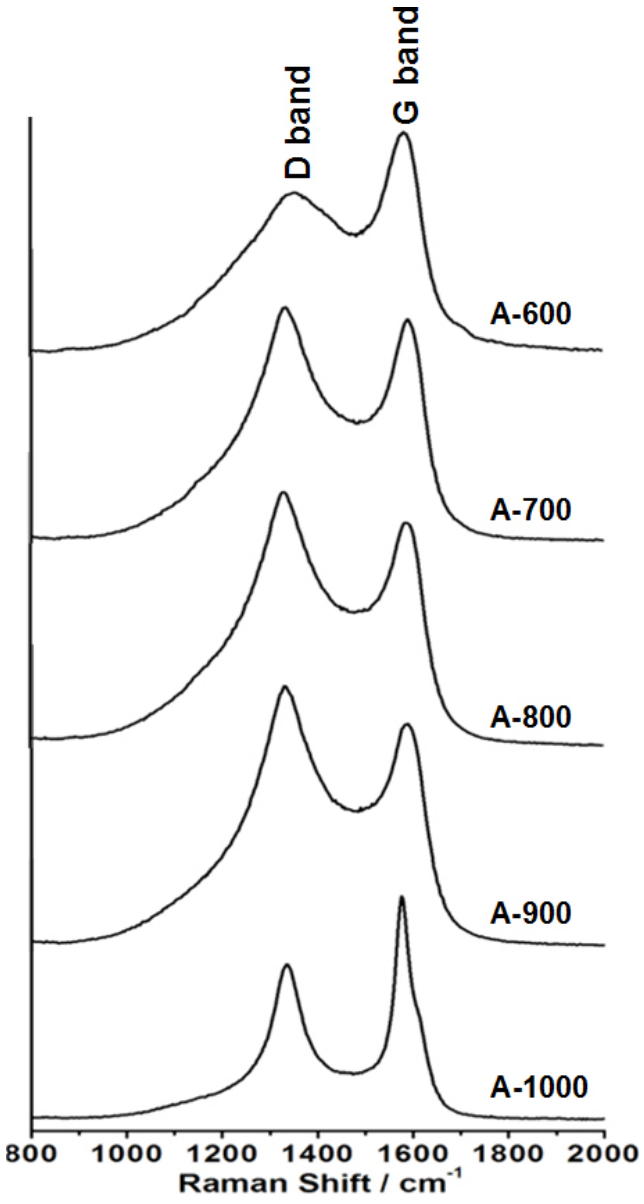

Fig. S4 Raman spectra of A-600 ~ A-1000 from top to bottom.

Table S1 EDX data for A-series hybrid composites.

Element	C (wt%)[a]	O (wt%)[a]	Fe (wt%)[a]
A-600	22.05	14.40	63.35
A-700	21.25	5.21	73.55
A-800	20.02	4.25	75.73
A-900	19.95	3.86	76.20
A-1000	33.35	0.92	65.73

[a]Weight percents (wt%) are obtained from EDX data.

Table S2 EDX data for B-series hybrid composites.

Element	C (wt%)[a]	O (wt%)[a]	Fe (wt%)[a]
B-600	43.52	16.91	39.57
B-700	43.27	12.41	44.32
B-800	42.77	5.24	51.99
B-900	41.09	4.91	54.00
B-1000	39.09	2.40	58.51

[a]Weight percents (wt%) are obtained from EDX data.

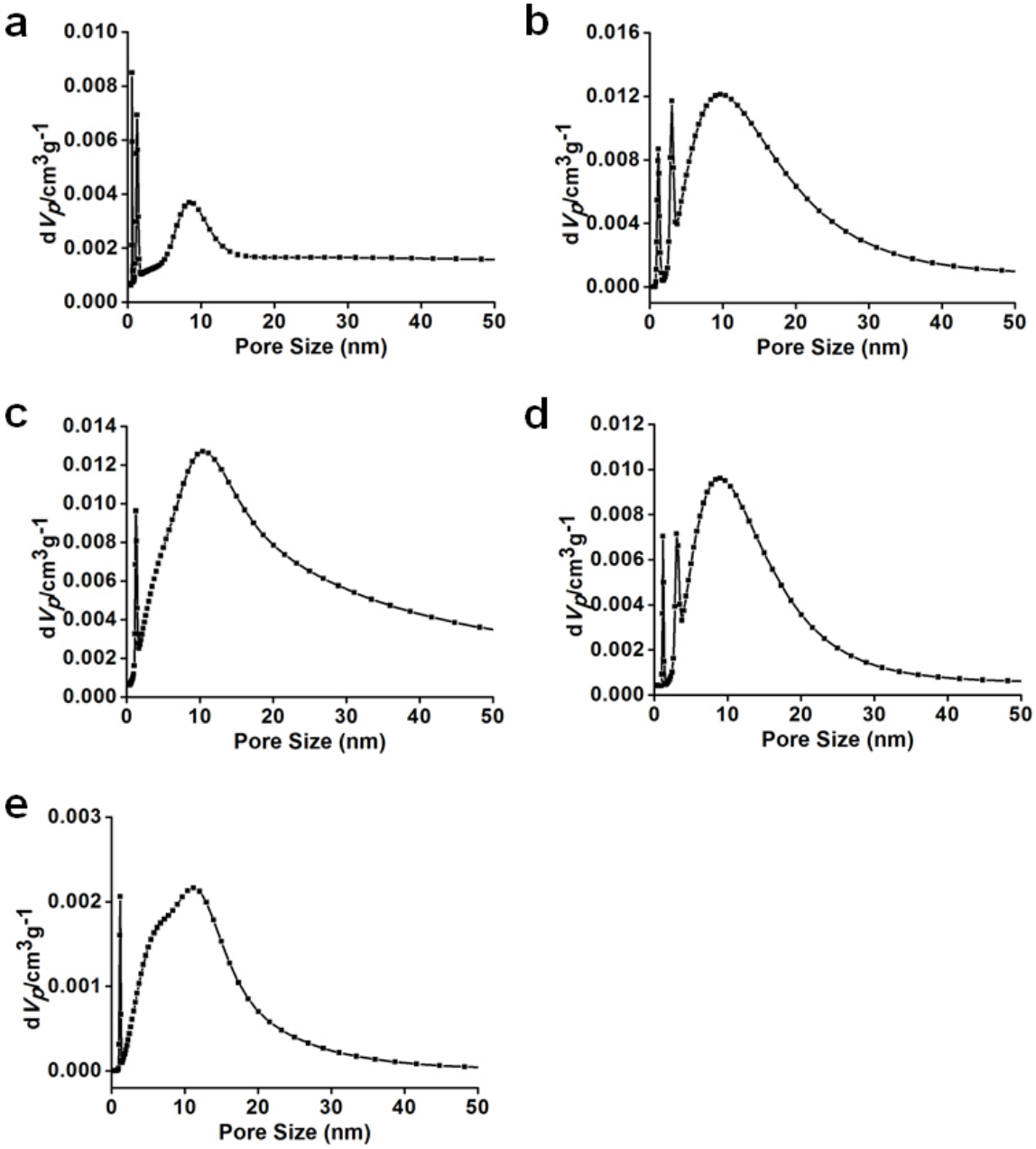

Fig. S5 Pore size distributions of (a) A-600, (b) A-700, (c) A-800, (d) A-900 and (e) A-1000 calculated using the NLDFT (non local density function theory) method.

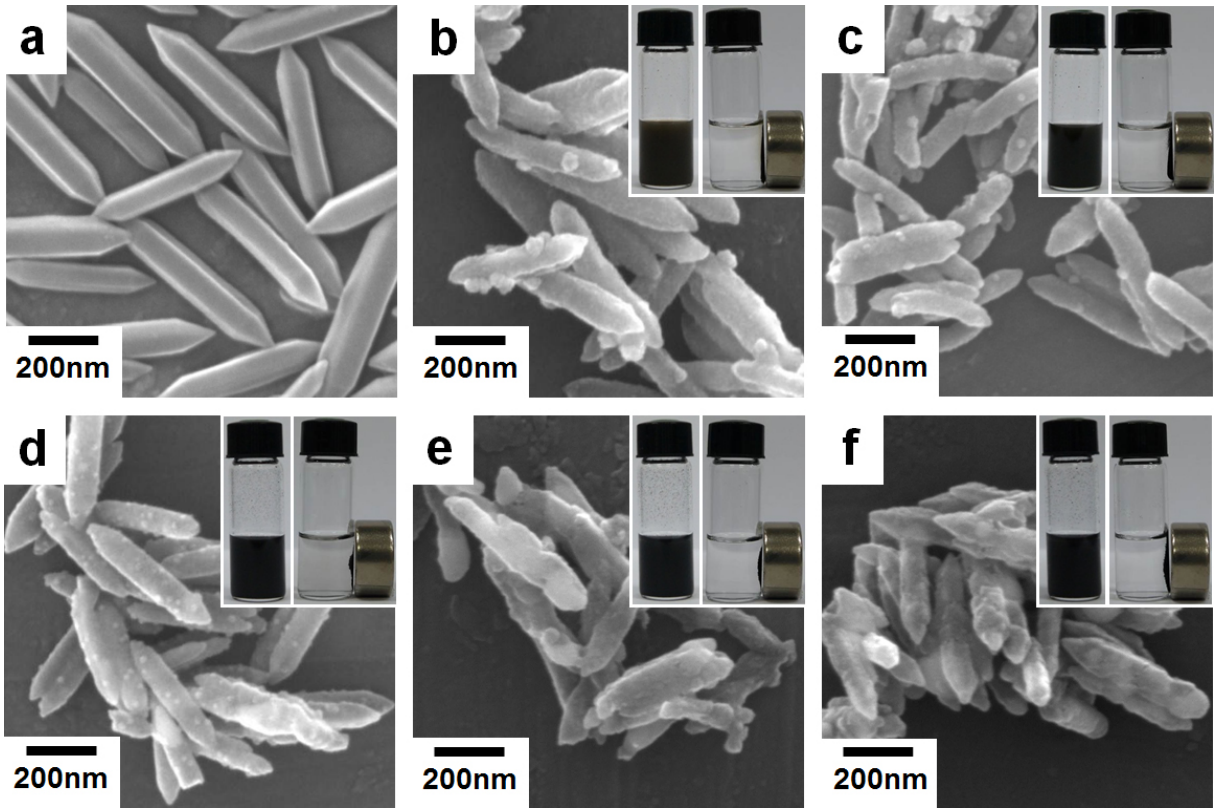

Fig. S6 SEM images of (a) Fe-MIL-88B, (b) B-600, (c) B-700, (d) B-800, (e) B-900 and (f) B-1000. The insets shows products dispersed in water and magnetically separated.

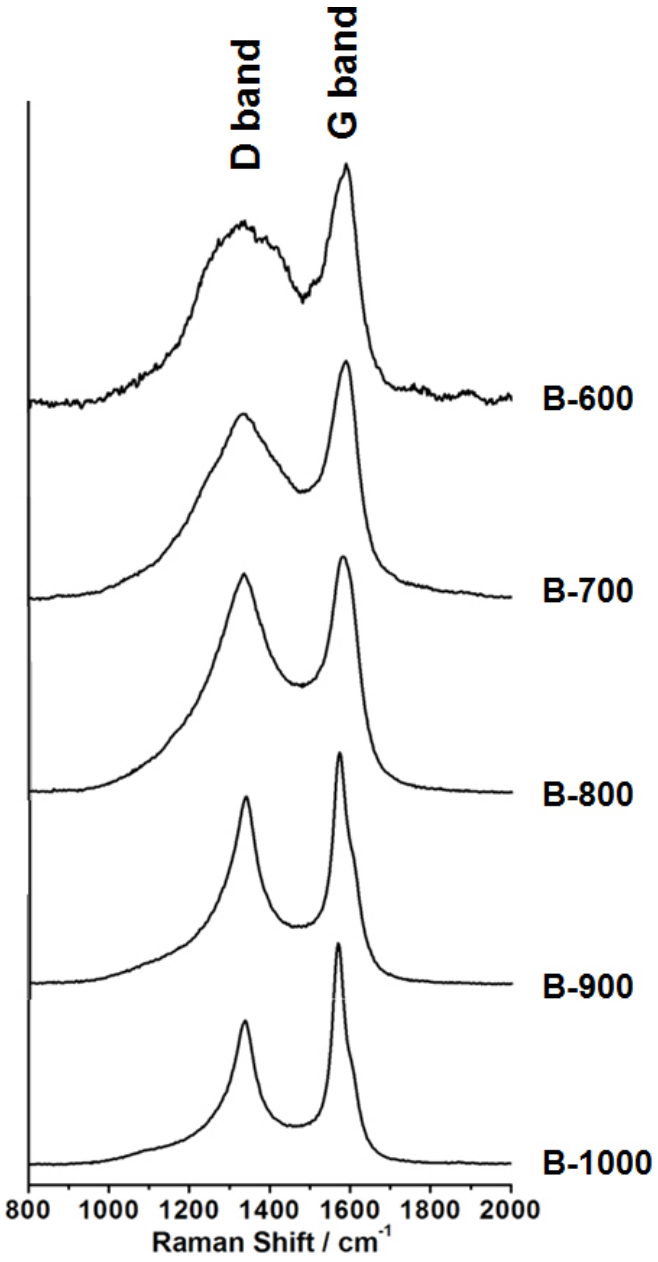

Fig. S7 Raman spectra of B-600 ~ B-1000 from top to bottom.

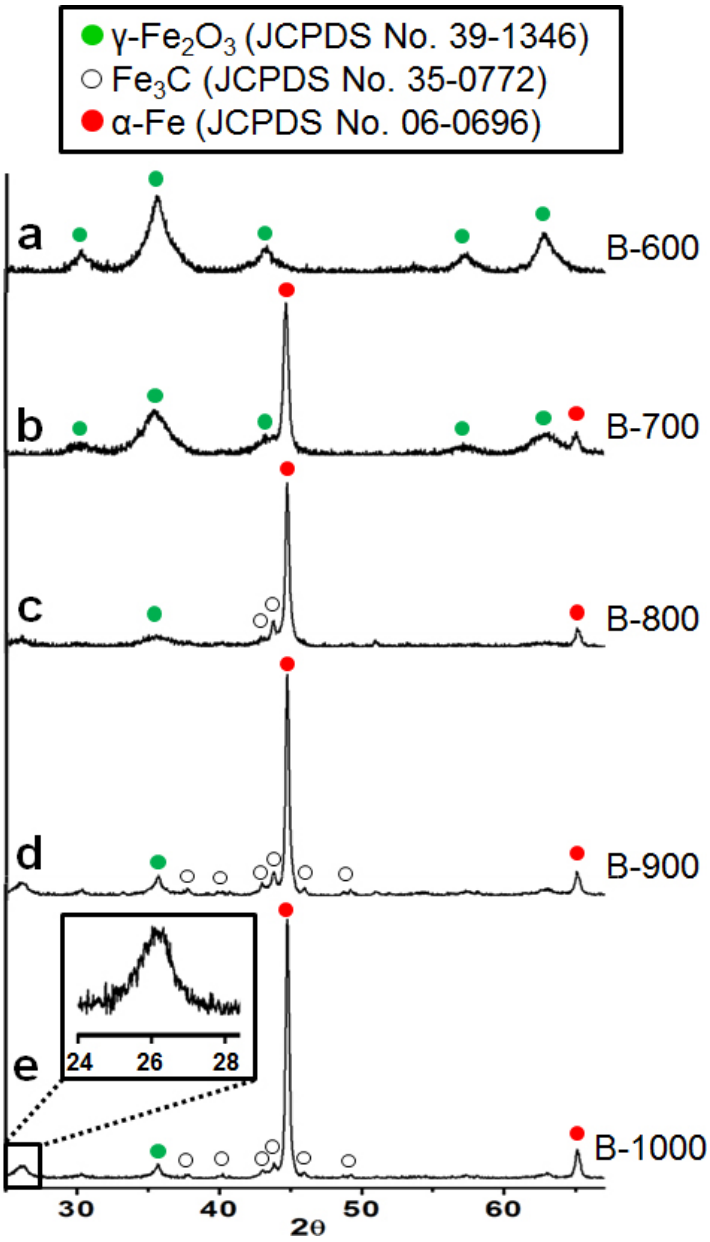

Fig. S8 PXRD patterns of (a) B-600, (b) B-700, (c) B-800, (d) B-900 and (e) B-1000. γ-Fe_2O_3 (JCPDS No. 39-1346), Fe_3C (JCPDS No. 35-0772) and α-Fe (JCPDS No. 06-0696). Magnified peak around 26° is shown in inset of (e).

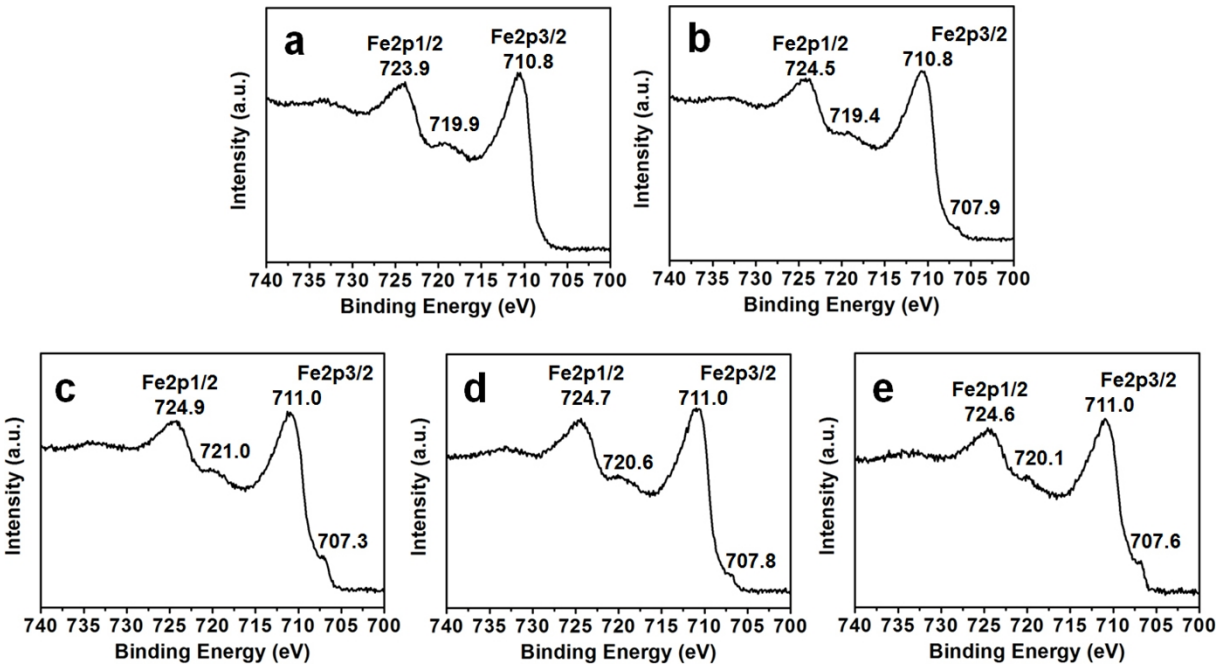

Fig. S9 XPS spectra of (a) B-600, (b) B-700, (c) B-800, (d) B-900 and (e) B-1000.

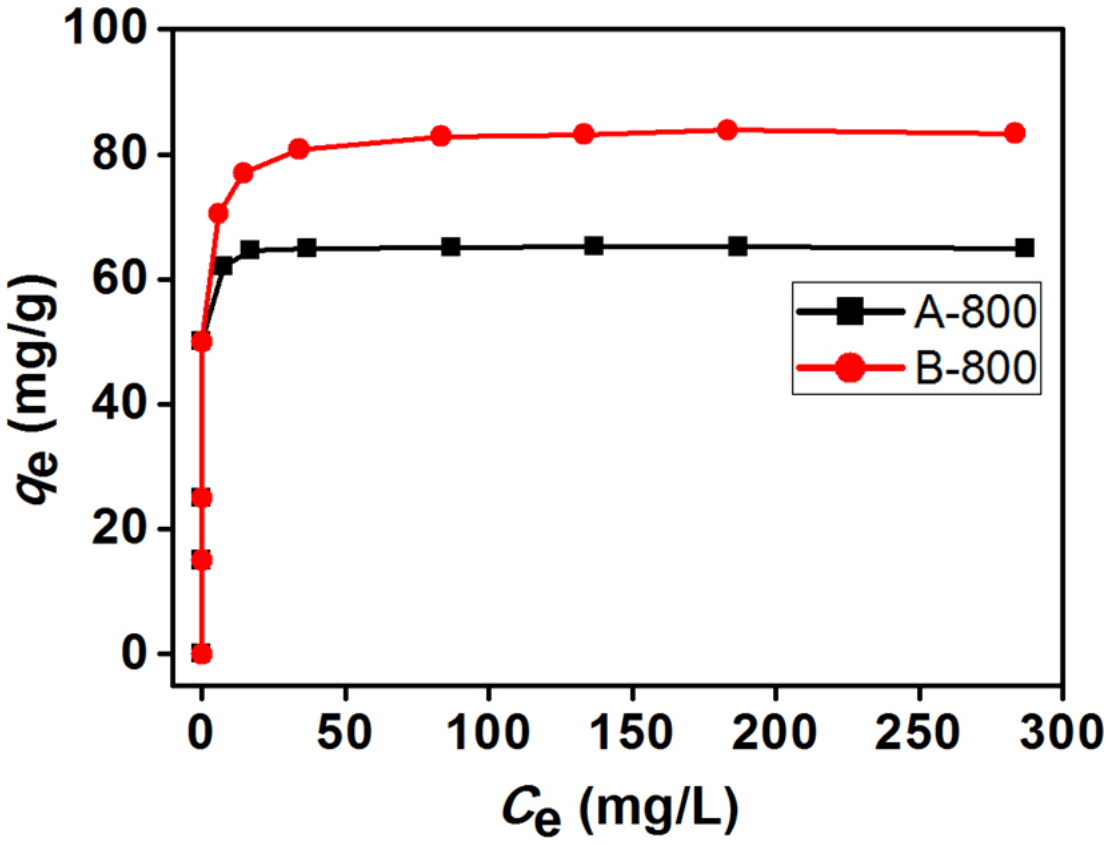

Fig. S10 Adsorption isotherms of methylene blue (MB) on A-800 and B-800.

References

1 W. Cho, S. Park and M. Oh, *Chem. Commun.*, 2011, **47**, 4138-4140.

2 C. Serre, F. Millange, S. Surblé and G. Férey, *Angew. Chem., Int. Ed.*, 2004, **43**, 6286-6289.