Supporting Information

Polymer-grafted magnetic microspheres for enhanced removal of methylene blue from aqueous solutions

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Summary

Total pages: 13 (S1-S13)

Text S1. Synthesis of Fe_3O_4 nanoparticles

13.5 g of $FeCl_3 \cdot 6H_2O$ (0.05 mol) and 5 g $FeCl_2 \cdot 4H_2O$ (0.025 mol) were dissolved in water under N_2 atmosphere. Then, the mixture of the ferric compounds was dropwise added into excessive NaOH aqueous solution under N_2 purging and vigorous stirring. The mixture was further stirred for 1h at 80 °C after which the product was collected by magnetic separation and repeatedly washed with water. Finally, Fe_3O_4 nanoparticles were obtained.

Text S2. Detailed parameters of solid state ^{13}C NMR spectra acquisition.

The ^{13}C cross polarization magic angle spinning (CP/MAS) NMR spectra were recorded using a 4 mm standard bore MAS probe head with a Larmor frequency of 100 62 MHz. The dried and finely powdered samples were packed in ZrO_2 rotors with a Kel-F cap at a spinning rate of 8 kHz. The experiments were conducted at a contact time of 2 ms. A total of 2000 scans were recorded with 6 s recycle delay for each sample. All ^{13}C CP/MAS chemical shifts are referenced to the resonances of adamantane ($C_{10}H_{16}$) standard (δCH_2=38.4 ppm).

Table S1 Some ^{13}C NMR chemical shifts and assignments for GMs.

δ/ppm	Assignment
27.5	$-CH_3$ (AMPS)
36.3	$-CH_2-$(AMPS-AA, $-CH_2-CH-$)
41.6	-CH- (AMPS-AA, $-CH_2-CH-$)
52.6	-C- (AMPS, $-C(CH_3)_2-$)
60.9	$-CH_2-$ (AMPS, $-CH_2-SO_3H$)
175.8	-C=O (AMPS-AA, -CONH- and -COOH)

Table S2 Comparison of adsorption capacities with various adsorbents for MB.

Adsorbent	pH	Temperature (K)	Dosage	Initial Concentration (mg/L)	Adsorption capacity (mg/g)	Reference
Glutamic acid modified chitosan magnetic composite microspheres (CS-Glu-MCMs)	7.0	293.15	30 mg/30 mL	10-1400	182.5	[1]
N-benzyl -O-carboxymethylchitosan (OC-BzM) magnetic nanoparticles	7.0	333.15	25 mg/20 mL	50-300	223.58	[2]
Magnetic chitosan lignosulfonate grafted with graphene oxide (MCLS/GO)	10.0	293.15	10 mg/20 mL	50-250	253.53	[3]
Chitosan/PAA multilayer-coated magnetic microspheres (Na-(CS/PAA)n/MPC)	7.0	298.15	10 mg/10 mL	100-450	308.6	[4]
Magnetic polydopamine–chitosan nanoparticles (PDA/CS/Fe_3O_4)	6.5	303.15	10 mg/50 mL	5-200	201 .08	[5]
Hydroxy-functionalized ionic liquids (ILs) modified with magnetic chitosan/ grapheneoxide (MG-ILs-OH)	12.0	303	100 mg/100 mL	40-140	243.31	[6]
Polymer-grafted magnetic composite microspheres (GMMs)	9.0	298.15	50 mg/50 mL	100-1200	925.9	This work

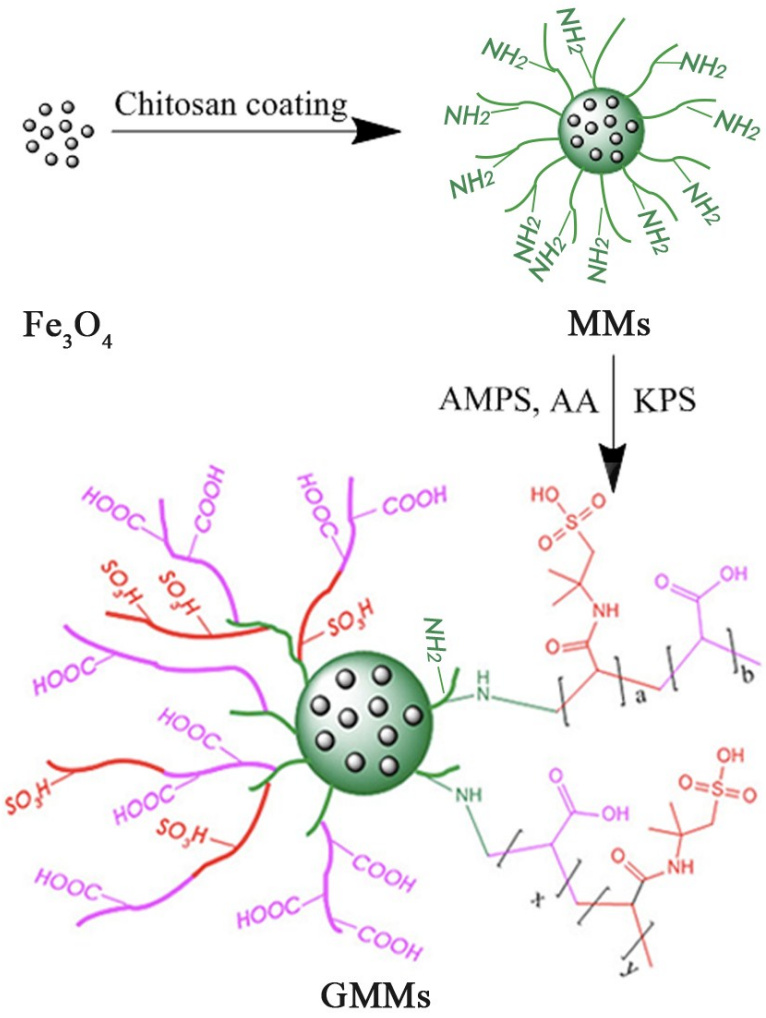

Fig. S1 Synthesis route for MMs and GMMs.

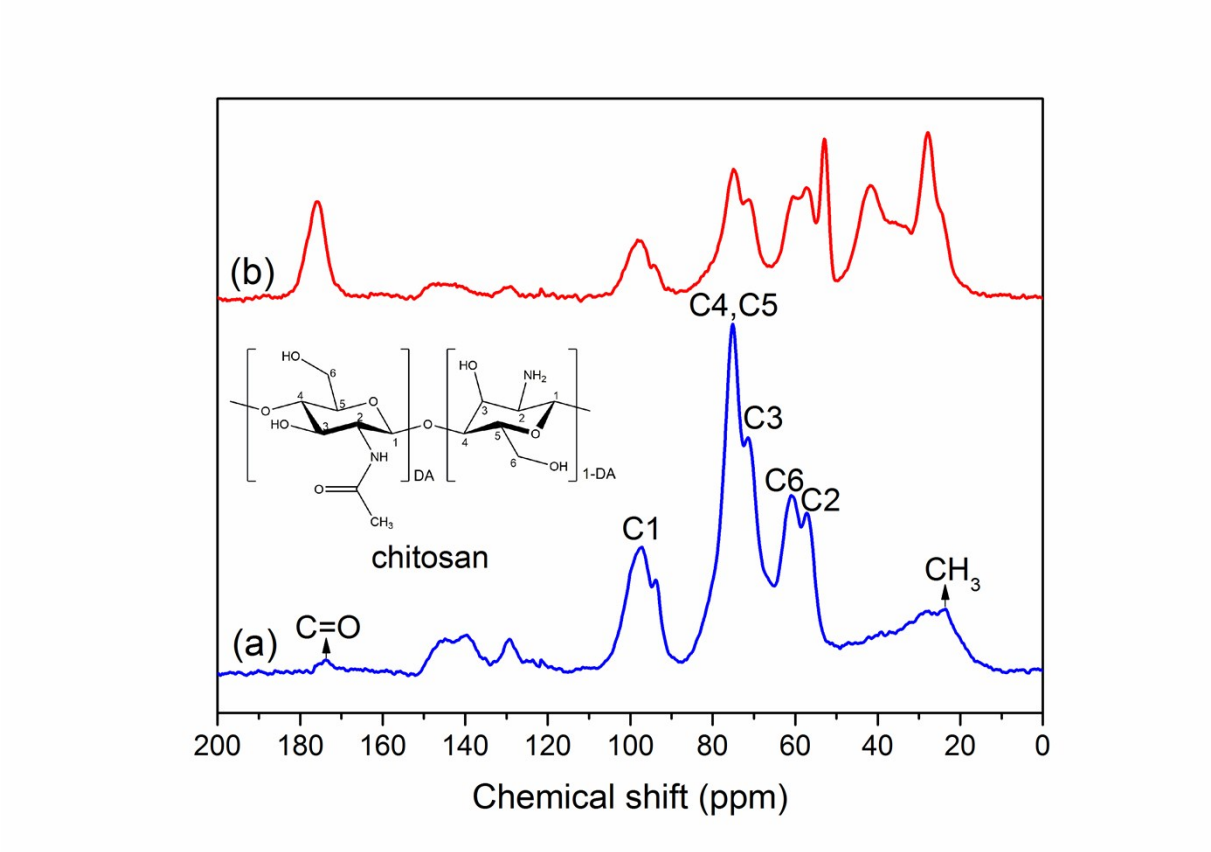

Fig. S2 Solid state ^{13}C NMR spectra of Ms (a), GMs (b).

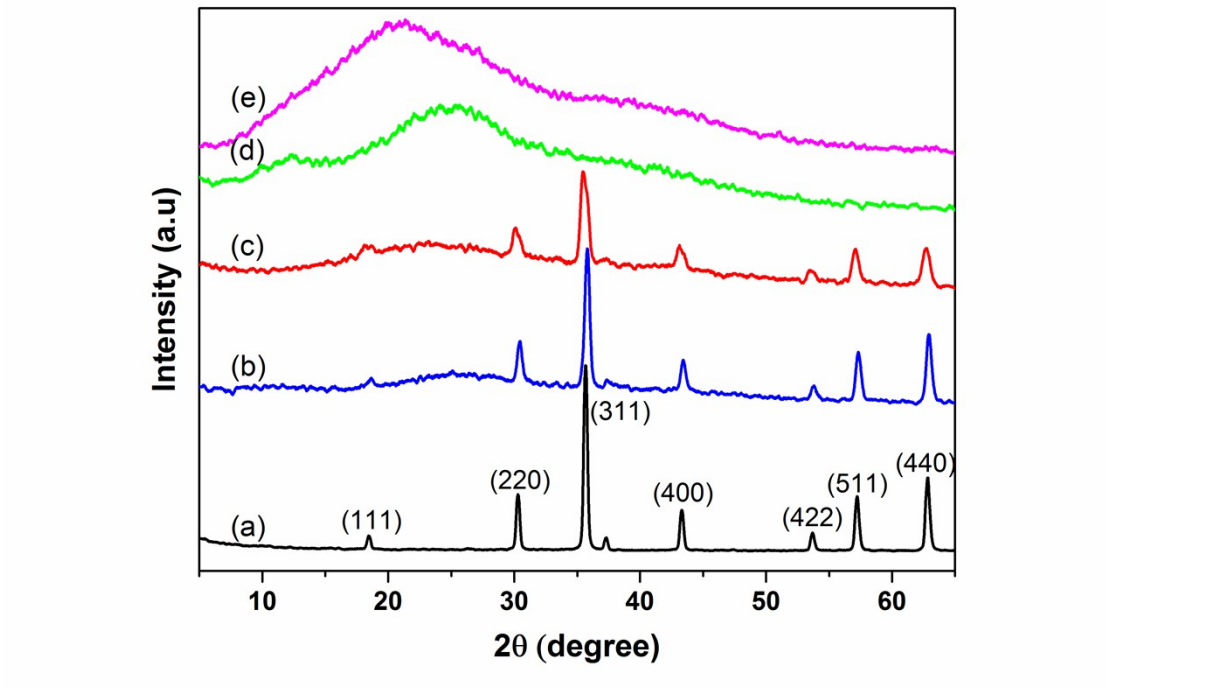

Fig. S3 XRD patterns of Fe_3O_4 (a), MMs (b), GMMs (c), Ms (d) and GMs (e).

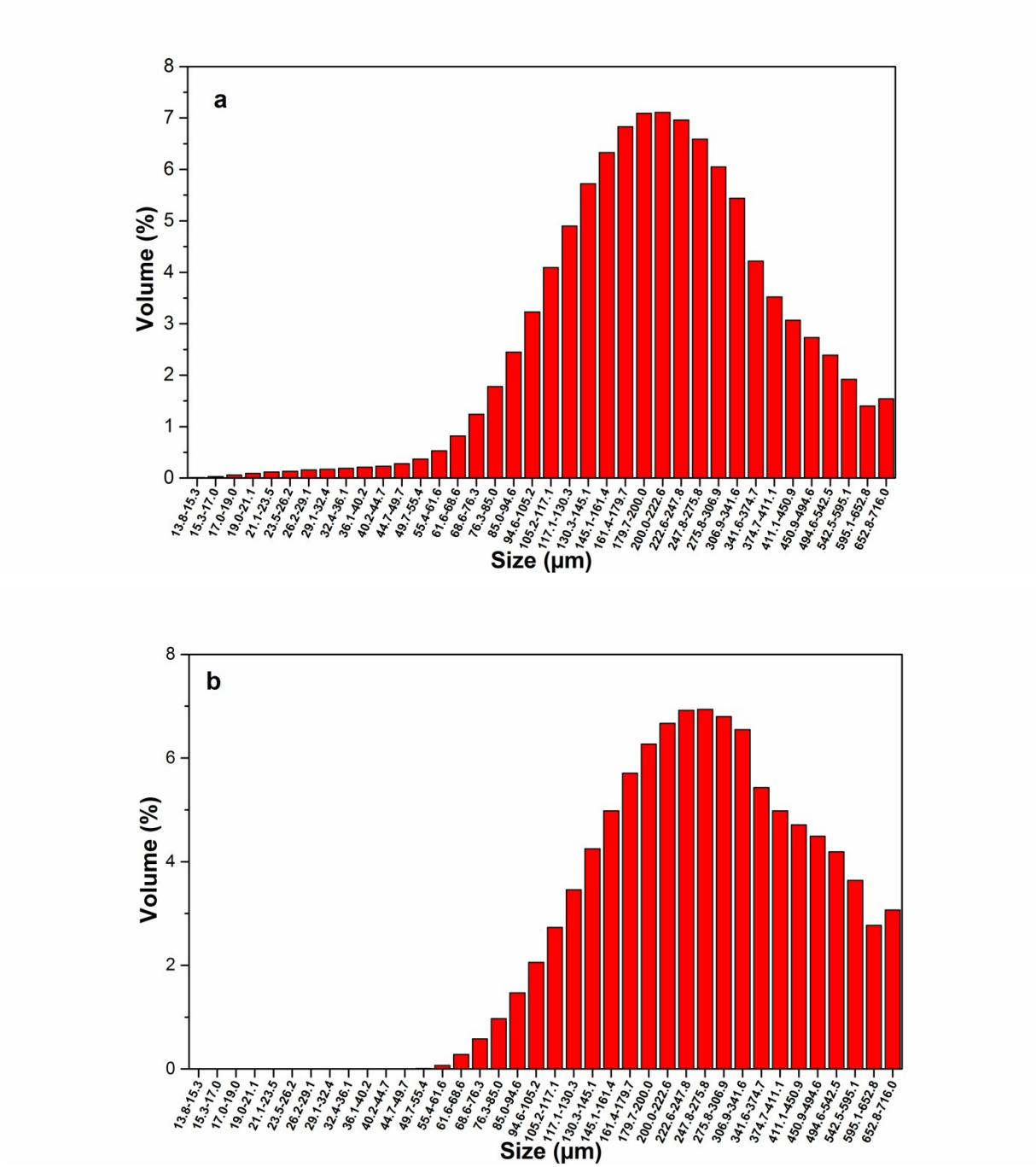

Fig. S4 Size distribution of MMs (a) and GMMs (b) based on static light scattering (SLS).

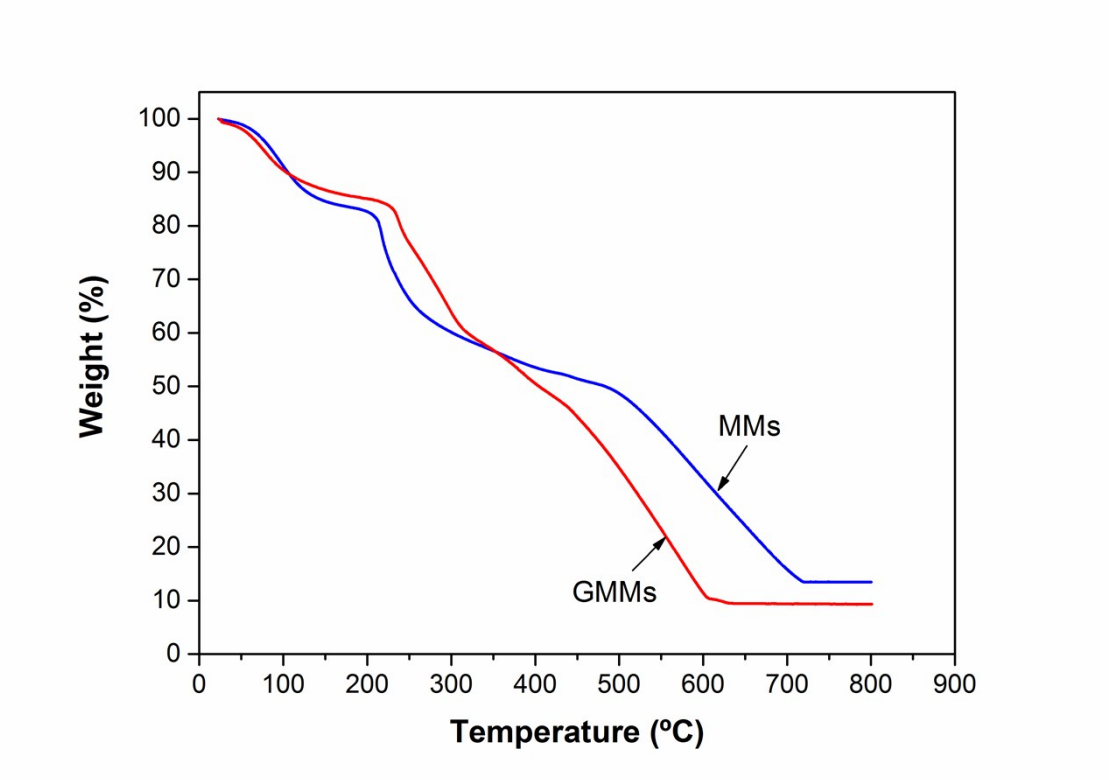

Fig. S5 TG curves of MMs and GMMs.

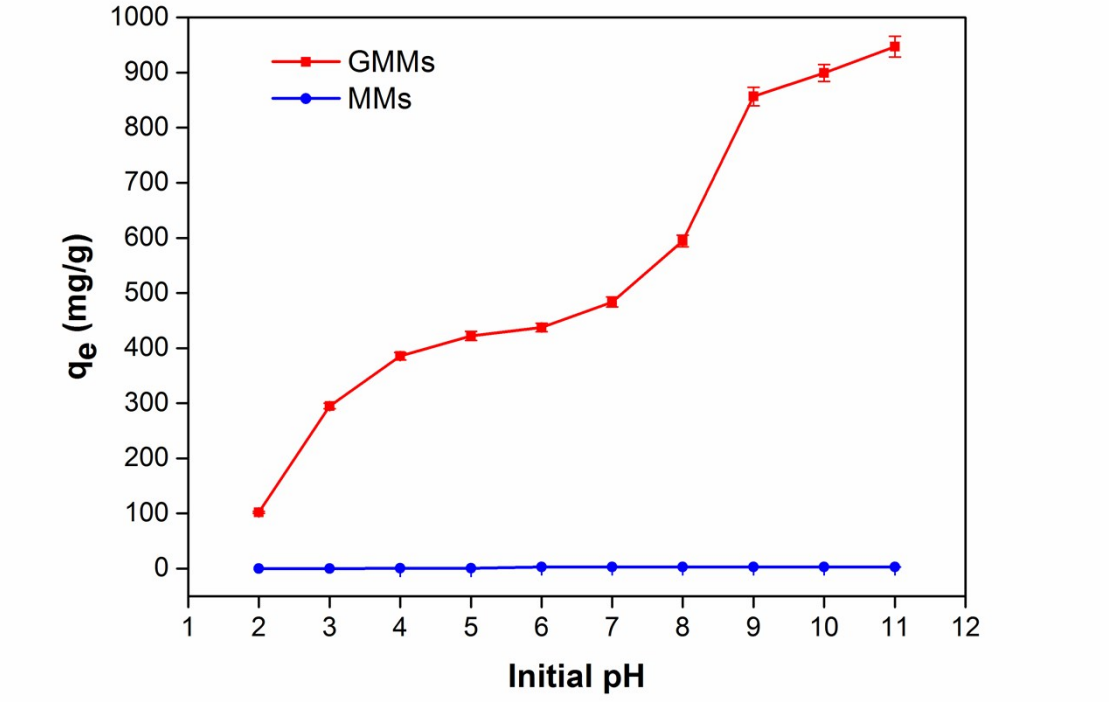

Fig. S6 Effects of initial solution pH on adsorption of MB by MMs and GMMs at 298.15 K and initial concentration 1000 mg/L.

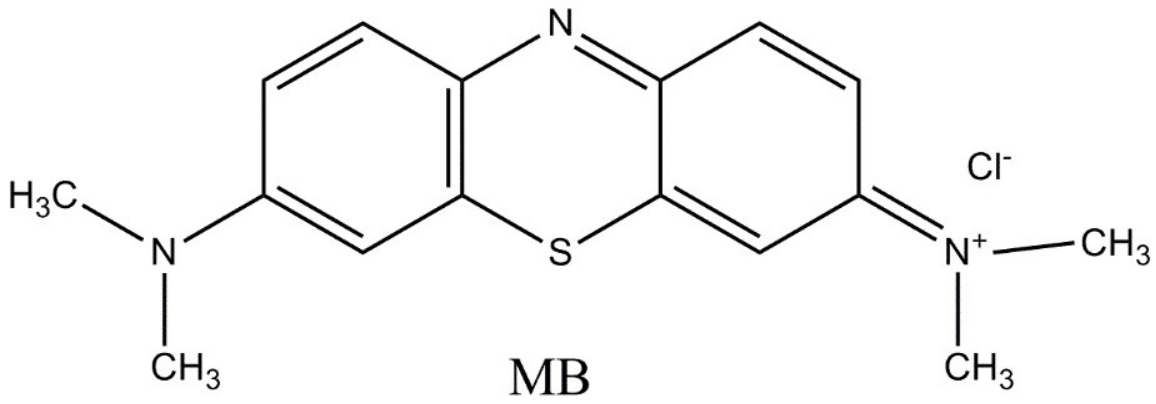

Fig. S7 Chemical structure of MB.

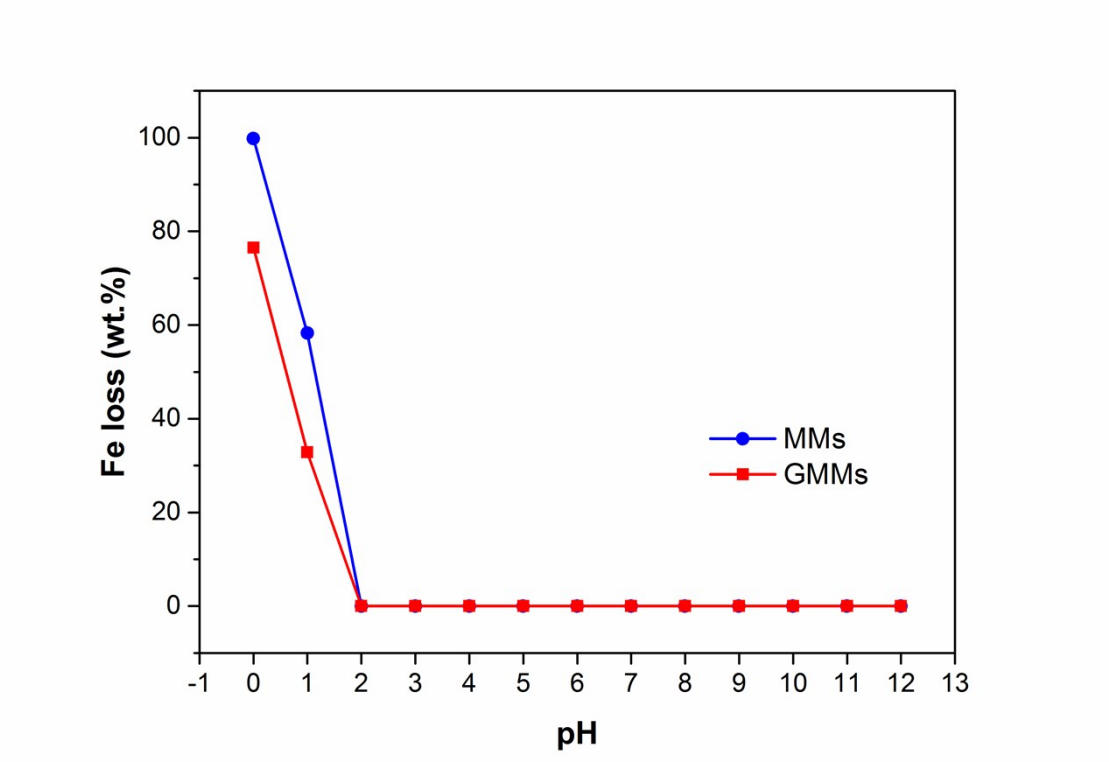

Fig. S8 Leaching of Fe from magnetic microspheres at different pHs.

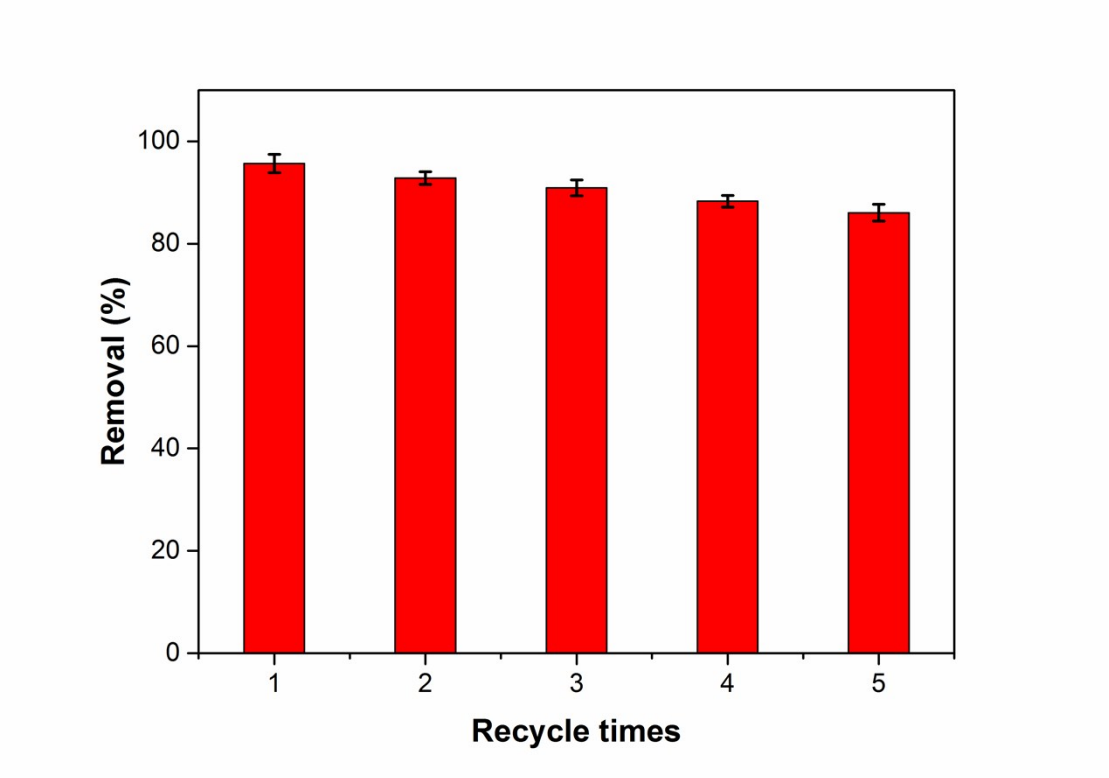

Fig. S9 Adsorption capacity of GMMs for MB in different adsorption-desorption cycles. The initial concentration and initial solution pH for adsorption experiments are 1000 mg/L and 9.0 respectively.

References

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