Electronic Supplementary Information (ESI)

Multifunctional triple-porous Fe₃O₄@SiO₂ superparamagnetic microspheres for

potential hyperthermia and controlled drug release

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Fig. S1 SEM image for *m*-Fe₃O₄@CTAB/SiO₂ microspheres.

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Fig. S2 Zeta potential distribution of the initial prepared *m*-Fe₃O₄@d*m*-SiO₂ microspheres in aqueous solution.



Fig. S3 Zeta potential distribution of the *m*-Fe₃O₄@*m*-SiO₂ microspheres in aqueous solution after being stored for 30 days.



Fig. S4 Zeta potential distribution of the *m*-Fe₃O₄@d*m*-SiO₂ microspheres in PBS buffer solution at pH 7.4.



Fig. S5 Mean hydrodynamic diameter (D_h) changes of *m*-Fe₃O₄@*dm*-SiO₂ microspheres before and after incubation with human blood plasma



Fig. S6 Drug loading profile in mespoprous *m*-Fe₃O₄@*dm*-SiO₂ microspheres



Fig. S7 TEM image of porous Fe₃O₄ microspheres



Fig. S8 N₂ adsorption-desorption isotherms and pore size distribution (the inset) of the synthesized porous

 Fe_3O_4 microspheres. The BET surface area is 26.5 m²/g.



Fig. S9 TEM image of *m*-Fe₃O₄@*dm*-SiO₂ microspheres after 3 h of hot water etching



Fig. S10 TEM image of *m*-Fe₃O₄@*m*-SiO₂ microspheres prepared with CTAB templating and without PVP protecting. Only perpendicular aligned mesochannels can be observed in SiO₂ shells.



Fig. S11 TEM image of *m*-Fe₃O₄@*m*-SiO₂ microspheres prepared with PVP protecting and without CTAB templating. Only randomly distributed pores can be observed in SiO₂ shells.



Fig. S12 N₂ adsorption-desorption isotherms of the *m*-Fe₃O₄@*m*-SiO₂ microspheres prepared with CTAB templating and without PVP protecting. The BET surface area is 304.5 m²/g



Fig. S13 N₂ adsorption-desorption isotherms of the *m*-Fe₃O₄@*m*-SiO₂ microspheres prepared with PVP protecting and without CTAB templating. The BET surface area is 86.9 m²/g.



Fig. S14 Images of SGC-7901 cells after being cultured at different concentrations of *m*-Fe₃O₄@*dm*-SiO₂/5-FU for 24 h: (a) 0 μg/mL, (b) 32 μg/mL, (c) 200μg/mL