Supporting Information

Hollow Nanocapsules of NiFe Hydroxides to Enable Doxorubicin

Delivery and Combinational Tumour Therapy

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Figure S1. SEM image of SiO_2 nanoparticles as the template during the synthesis.



Figure S2. (a) TEM and (b) STEM image of $SiO_2@NiFe(OH)_x$ nanoparticles.



Figure S3. Elemental mapping of SiO₂@NiFe(OH)_x nanoparticles.



Figure S4. STEM images with (a) high and (b) low magnification of H-NiFe(OH)_x.



Figure S5. (a) XRD pattern and (b) FTIR spectrum of H-NiFe(OH)_x nanocapsules.



Figure S6. XPS (a) survey and (b) Ni 2p spectrum of H-NiFe(OH)_x.



Figure S7. Size distribution of H-NiFe(OH)_x during surface modification.



Figure S8. (a) Size distribution and (b) polymer disperisity index(PDI) of H-NiFe(OH)_x in water, normal saline (0.9% NaCl) and cell culture medium (1640) at 0 ,1,3 day. And (c) corresponding digital photographs.



Figure S9. FTIR spectrum of H-NiFe LDH, DOX and DOX/H-NiFe(OH)_x.



Figure S10. Elemental mapping images of H-NiFe(OH)_x (scale bar 50 nm).



Figure S11. TEM images of H-NiFe(OH)_x nanocapsules in PBS with (a) pH 7.4 and (b) pH 5.5 for varied periods of time (scale bar 500 nm).



Figure S12. Fluorescence images of 4T1 cells stained with calcein-AM (green, live cells) and propidium iodide (red, dead cells) after treatments with various drugs. Scale bar: $200 \mu m$.



Figure S13. The fluorescence intensity of 4T1 cells incubated with DOX and DOX/H-NiFe(OH)_x quantified by flow cytometry.



Figure S14. Bright filed images of 4T1 tumor cells incubated with DCFH-DA probe incubated with different drugs (Scale bar: $200 \ \mu m$).



Figure S15. Digital photographs of tumours collected from mice after 15-day treatments.



Figure S16. Ni content distribution (per unit tumor mass) after DOX/H-NiFe(OH)_x injection at different time points.



Figure S17. Acute and chronic toxicity assays of DOX/ H-NiFe(OH)_x after 30-day intravenous administrations.