Supporting information

Metallized hollow-COF nanobowls with dual-mode ROS generation for cancer sonodynamic therapy

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Fig. S1 N_2 adsorption-desorption isotherms of (a) HCOF and (b) HHC.



Fig. S2 Normalized at different Hemin concentration for $\lambda = 397$ nm.



Fig. S3 (a) SEM images of FeHHC; (b) SEM images of CuHHC; (c) SEM images of MnHHC; (d) XRD pattern of FeHHC, CuHHC, MnHHC and Hemin; (e) UV-vis absorption spectra of FeHHC, CuHHC, MnHHC and Hemin; (f) The energy bandgap of FeHHC, CuHHC, MnHHC and HHC. (Scale bar: $1 \mu m$)



Fig. S4 Zeta potentials of COF, HCOF, HHC, FeHHC, CuHHC and MnHHC.



Fig. S5 UV-vis spectra of supernatants of FeHHC collected at different time points under different pH conditions.



Fig. S6 ESR spectra of FeHHC, CuHHC and MnHHC trapped by TEMP under US irradiation for 5 min.



Fig. S7 Detection of ${}^{1}O_{2}$ production of Hemin, HHC, CuHHC and MnHHC with US via degradation of ABDA.



Fig. S8 (a) Zeta potentials of FeHHC and FeHHCA; (b) Hydrated particle sizes of COF, HCOF, FeHHC and FeHHCA.



Fig. S9 Hydrated particle size of HCOF freshly prepared and after one week in PBS7.4.



Fig. S10 Cellular uptake of FeHHCA/RhB by 4T1 cells at the dose of 200 μ g mL⁻¹ after different incubation times. scale bar: 200 μ m.



Fig. S11 Hemolysis of different concentrations of FeHHCA.



Fig. S12 Distribution of Fe in major organs and tumor after intratumoral injection at different times.



Fig. S13 H&E staining images of major organs after different treatment groups. Scale bar: 100 μ m.



Fig. S14 *In vivo* toxicology results in mice treated with FeHHCA (day 0 and day 7 after injection). (a) White blood cell level (WBC); (b) Lymphocytes level (LYM); (c) Monocytes level (MON); (d) Mean hematocrit volume (MCV); (e) Hemoglobin (HGB); (f) Procalcitonin (PCT); (g) Urea nitrogen (UREA); (h) Albumin/Globulin (A/G); (i) Neutrophils level (GRA).