Supporting information for:

Copper–Terephthalic Starch-Regulated Acid pH/Hydrogen Peroxide as Simultaneous-Responsive Fluorescence Probes for Lysosomes Imaging Jian Chen,<sup>a</sup> Yubing Si,\*<sup>a</sup> Yibiao Liu,<sup>a</sup> Saisai Wang,<sup>a</sup> Shijie Wang,<sup>a</sup> Ying Zhang,<sup>a</sup> Baocheng Yang,<sup>a</sup> Zuling Zhang,<sup>b</sup> Shouren Zhang\*<sup>a</sup> <sup>a</sup>Henan Key Laboratory of Nanocomposite and Applications, Institute of Nanostructured Functional Materials, Huanghe Science and Technology College, Zhengzhou, Henan 450006, China <sup>b</sup>Henan Provincial Chemi-Industries Research Station Co., Ltd, Zhengzhou, Henan 450000, China **Corresponding Authors** Shouren Zhang E-mail: <a href="mailto:shourenzhang@infm.hhstu.edu.cn">shourenzhang@infm.hhstu.edu.cn</a> Yubing Si E-mail: yubingsi@infm.hhstu.edu.cn Supporting Information Figures



Figure S1. EDX spectrum of CuBDC-2.



Figure S2. (a)  $N_2$  adsorption and desorption isotherm and (b) BJH pore distribution of the CuBDC-1 and CuBDC-2.



Figure S3. SEM images of CuBDC synthesized using different copper source with or

without soluble starch.



**Figure S4.** (a) UV-Vis spectra of supernatants withdrawn from the solution containing CuBDC-1 or CuBDC-2 at pH~4 or pH~7 after 4 h of dispersion. (b) Release of Cu<sup>2+</sup> ions from CuBDC-2 in solutions at pH 4, 5, 6 and 7, respectively. (c) Fluorescence spectra of solution containing CuBDC-2 and H<sub>2</sub>O<sub>2</sub> (1.5 mM) at pH 7 or pH 4 with or without extra Cu<sup>2+</sup> added. (d) Zeta potential of CuBDC-1 and CuBDC-2 dispersed in solution at pH 7 or pH 4.



Figure S5. Fluorescence spectra of CuBDC-2 treated with different concentrations of

H<sub>2</sub>O<sub>2</sub> at pH~4 (a), pH~5 (b), pH~6 (c), pH~7 (d) for 4 h.



Figure S6. (a) Fluorescence intensity change of mixture of Cu<sup>2+</sup> and TA treated with

different concentrations of  $H_2O_2$  and different pH values for 4 h. F indicated the fluorescence intensity of solution containing mixture of  $Cu^{2+}$  and TA and different concentrations of  $H_2O_2$  at different pH values for 4 h,  $F_0$  indicated the fluorescence intensity of distilled water containing mixture of  $Cu^{2+}$  and TA. (b) Relationship between the fluorescence intensity change and  $H_2O_2$  concentration in solutions containing mixture of  $Cu^{2+}$  and TA at pH~4 for 4 h. F indicated the fluorescence intensity of solution containing mixture of  $Cu^{2+}$  and TA and different concentrations of  $H_2O_2$  at pH~4 for 4 h,  $F_0$  indicated the fluorescence intensity of distilled water containing mixture of  $Cu^{2+}$  and TA. (c) Relationship between the fluorescence intensity change and pH values in solution containing mixture of  $Cu^{2+}$  and TA and 1 mM  $H_2O_2$  for 4 h. F indicated the fluorescence intensity of solution containing mixture of  $Cu^{2+}$  and 1 mM  $H_2O_2$  at different pH values for 4 h,  $F_0$  indicated the fluorescence intensity of solution containing mixture of  $Cu^{2+}$  and TA and 1 mM  $H_2O_2$  for 4 h. F indicated the fluorescence intensity of solution containing mixture of  $Cu^{2+}$  and 1 mM  $H_2O_2$  at different pH values for 4 h,  $F_0$  indicated the fluorescence intensity of distilled water containing mixture of  $Cu^{2+}$  and TA.



**Figure S7.** Fluorescence intensity of CuBDC-2 incubated with physiologically important metal ions and bio-molecules including  $Fe^{3+}$ ,  $Cu^{2+}$ ,  $Mn^{2+}$ ,  $Zn^{2+}$ ,  $Ca^{2+}$ ,  $Mg^{2+}$ ,  $Co^{2+}$ , glucose and L-lysine at pH~4 or pH~7.



**Figure S8.** Fluorescence spectra of CuBDC-2 treated with 1mM  $H_2O_2$  at pH~4 (a), pH~5 (b), pH~6 (c) or at pH~7 (d) over 48 h.



**Figure S9.** (a) Cell viability of HeLa cells treated with CuBDC-2 at 2.5, 7.5, 12.5, 17.5, 25  $\mu$ g/mL for 24 h or 48 h. (b) Growth inhibition of HeLa cells by different concentrations of CuBDC-2. IC50 was determined by sigmoidal curve fitting.



Figure S10. Fluorescence images of HeLa cells after co-incubation with CuBDC-2 for 4

h and then stained with ROS fluorescence probe DCFH-DA.



**Figure S11.** (a) Cell internalization of  $Cu^{2+}$  after incubated with CuBDC-2 for 6 h, 12 h or 24 h. (b) Cell internalization of  $Cu^{2+}$  after pretreated with different endocytosis inhibitors and then incubated with CuBDC-2 for 6 h.



**Figure S12.** Fluorescence intensity of HeLa cells incubated with CuBDC-2 for 24 h and then treated with buffer solution at different pH values (pH~4, pH~5, pH~6 or pH~7), containing  $H_2O_2$  (0.1 mM or 1mM) and nigericin. I indicated the fluorescence intensity of cells incubated with CuBDC-2 and treated with specific pH value and  $H_2O_2$  concentration,  $I_0$  indicated the fluorescence intensity of cells only incubated with CuBDC-2.