## Investigation of a Halloysite-Based Fluorescence Probe with a Highly Selective and Sensitive "Turn-On" Response upon Hydrogen Peroxide

----- Supporting Information

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Figure S2. <sup>13</sup>C NMR spectra of PF1



Figure S3. <sup>1</sup>H NMR spectrum of PA



Figure S4. <sup>13</sup>C NMR spectrum of PA



Figure S5. the FTIR spectrum of PA



Figure S6. XPS spectra of HNTs and HNTs-PA



**Figure S7**. Fluorescence turn-on response of 0.1 mg/mL HNTs-PA. Date were acquired at 25 °C in 2 mL H<sub>2</sub>O, with excitation at  $\lambda$ =470 nm and emission was collected between 480-700 nm. Time points represent 0-60 min after the addition of different times H<sub>2</sub>O<sub>2</sub> (different times to the mole number of PA). (a) [H<sub>2</sub>O<sub>2</sub>] = 5 × 10<sup>-5</sup> M (the addition amount of H<sub>2</sub>O<sub>2</sub> is 1 × 10<sup>-7</sup> mol); (b) [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-4</sup> M; (c) [H<sub>2</sub>O<sub>2</sub>] =  $5 \times 10^{-4}$  M; (d) [H<sub>2</sub>O<sub>2</sub>] =  $2.5 \times 10^{-3}$  M; (e) [H<sub>2</sub>O<sub>2</sub>] =  $2.5 \times 10^{-2}$  M. The turn-on response of each probe was completed at these time points.



**Figure S8.** Fluorescence "turn-on" response of 0.1 mg/mL HNTs-PA. Date were acquired at 25 °C in 2 mL H<sub>2</sub>O, with excitation at  $\lambda$ =470 nm and emission was collected between 480-700 nm for HNTs-PA. Time points represent 60 min after the addition of different times H<sub>2</sub>O<sub>2</sub> (1 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 5 × 10<sup>-5</sup> M; 5 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-4</sup> M; 10 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 5 × 10<sup>-4</sup> M; 50 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-3</sup> M; 500 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-3</sup> M; 500 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-3</sup> M; 500 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-3</sup> M; 500 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-3</sup> M; 500 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-3</sup> M; 500 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-4</sup> M; 500 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-4</sup> M; 500 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-4</sup> M; 500 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-4</sup> M; 500 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-4</sup> M; 500 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-4</sup> M; 500 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-4</sup> M; 500 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-4</sup> M; 500 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-4</sup> M; 500 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-4</sup> M; 500 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-4</sup> M; 500 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-4</sup> M; 500 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-4</sup> M; 500 euqal represents [H<sub>2</sub>O<sub>2</sub>] = 2.5 × 10<sup>-5</sup> M].



**Figure S9.** Fluorescence "turn-on" response of 0.1 mg/mL HNTs-PA upon different substances. Date were obtained at 25 °C in 2 mL H<sub>2</sub>O, with excitation at  $\lambda$ =470 nm. The PL intensity represents the fluorescence intensity at the maximum emission wavelength.



Figure S10. The UV-vis spectra of HNTs, PA and HNTs-PA



Figure S11. The energy dispersive spectra (EDS) results of HNTs-PA



**Figure S12**. Confocal fluorescence images of living Hela cells after treating with 0.5 mg/mL HNTs-PA for 1 (a and f), 2 (b and g), 4 (c and h), 8 (d and i) and 12 h (e and k) at 37 °C. The excitation wavelength was fixed at 488 nm and fluorescent signals were collected from 500 to 600 nm.



**Figure S13**. Plot of the fluorescence intensity of HNTs-PA solution *vs* the concentration of  $H_2O_2$  (fluorescence intensity was recorded at 20 min after the addition of  $H_2O_2$ ). Results of the linear regression for fluorescence intensity and concentration of  $H_2O_2$  show that the linear correlation coefficient ( $R^2$ ) is greater than 0.99 which indicates that the fluorescence intensity of HNTs-PA solution and concentration of  $H_2O_2$  shows a good linearity relationship in the range from 5 ×10<sup>-5</sup> to 5 ×10<sup>-3</sup> M.



Figure S14. Cell viability of Hela cells at different concentrations of HNTs-PA. The  $IC_{50}$  value was calculated as 1.11 mg/mL