## **Supporting Information**

## A "win-win" TiO<sub>2:</sub>Yb,Ho,F nanoplatform for NIR light-induced synergistic therapy and imaging

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FigureS1. (A) FTIR spectra of  $\beta$ -CD, CA, cit-CD; (B) Upconversion luminescence spectra of TiO<sub>2</sub>:Yb,Ho,F after modifications, excited under the same test condition by 980 nm laser; (C)  ${}^{1}O_{2}$  generation by TiO<sub>2</sub>:Yb,Ho,F under 808nm laser(0.8 W/cm<sup>2</sup>), different powers within 10min irradiation; (D) XPS measured the content of TiO2:Yb,Ho,F



Figure S2. (A) Evaluation of OH• generation by TiO<sub>2</sub>:Yb,Ho,F under 808nm laser(0.8 W/cm<sup>2</sup>, different irradiation times) through the decay of MB absorption at  $\lambda = 664$  nm; (B) the OH• comparison between TiO<sub>2</sub>:Yb,Ho,F and undoped TiO<sub>2</sub> at the same conditions.

**Combination Index:** The combination index (CI) was calculated using the following equation:  $CI=D_1/D_{m1}+D_2/D_{m2}$ , where  $D_1$  and  $D_2$  are concentrations of DTX combination with TiO<sub>2</sub>:Yb,Ho,F- $\beta$ -CD @HA under irradiation at IC<sub>50</sub>, respectively, while  $D_{m1}$  and  $D_{m2}$  are the concentrations of the drugs dosed individually to achieve that same drug effect level. CI values lower than 1, indicating the synergistic effects.

According to the cell cytotoxicity (Figure 4B and 4C.b) and SPSS IC<sub>50</sub>.

 $CI=D_1/D_{m1}+D_2/D_{m2}=1.26/7.89+9.36/65.23=0.303$ , CI values was lower than 1, indicating the good synergistic effects.(IC<sub>50</sub> of synergistic therapy group, free DTX group, only PDT group were 1.26ug mL<sup>-1</sup>, 7.89 ug mL<sup>-1</sup>, 65.23 ug mL<sup>-1</sup>.)

S3. Combination index (CI) of TiO<sub>2</sub>:Yb,Ho,F- $\beta$ -CD/DTX@ HA *vs.* monotherapy on MCF-7 cancer cells.



Figure S4. cell circle arrest with the PI single staining assay by flow cytometry (a) Control cells, (b) TiO<sub>2</sub>:Yb, Ho,F- $\beta$ -CD@HA (c) TiO<sub>2</sub>:Yb,Ho,F- $\beta$ -CD @HA +laser,(d) DTX (e)TiO<sub>2</sub>:Yb, Ho,F- $\beta$ -CD/DTX; (f)TiO<sub>2</sub>: Yb,Ho,F- $\beta$ -CD/DTX@HA; and (g)TiO<sub>2</sub>:Yb,Ho,F - $\beta$ -CD/DTX @HA+ laser Data were presented as mean ± standard deviation.



Figure S5. H&E stained organs with different treatments: saline a),null NPs b); only laser c); null NPs+laser d); DTX e); TiO<sub>2</sub>:Yb,Ho,F- $\beta$ -CD/DTX f); TiO<sub>2</sub>:Yb,Ho,F- $\beta$ -CD/DTX @ HA g); TiO<sub>2</sub>:Yb,Ho,F- $\beta$ -CD/DTX @ HA +laser h). Data were presented as mean ± standard deviation (n=6)