## Supporting information for

Efficient photosensitization of terbium ions enabled by hydrolysis of siloxy groups in ligands with specific side-chains

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**Figure S1. a:** <sup>1</sup>H NMR spectrum of compound **1** in CDCl<sub>3</sub>; **b:** <sup>1</sup>H NMR spectrum was observed when 6.7 mg of **1** was dissolved in 0.7 ml of CD<sub>3</sub>OD at 50 °C, to which was added 0.7 ml of HCl (pH = 1.0) in CD<sub>3</sub>OD and stirred for 6 hours. No peaks from compound **1** were detected; **c:** <sup>1</sup>H NMR spectrum of solid of hydrolyzed products precipitated from the reaction of **1** in CDCl<sub>3</sub>.



**Figure S2.** We manually mixed free compound **1** (monomer) and hydrolyzed oligomer in CHCl<sub>3</sub>, then eluted by CHCl<sub>3</sub> through polystyrene packed column in GPC (left), and oligomer only (right).



**Figure S3.** IR spectra of hydrolytic compound **1** before (top) and after addition of terbium ions (1/Tb = 1/1) (lower).



Figure S4. Phosphorescence spectra of compound 1 with Tb ions (1/Tb = 1/1,  $5 \times 10^{-6}$  M, CHCl<sub>3</sub>/EtOH = 99/1) before (red) and after hydrolysis (black). Ex = 280 nm. Inset: Emission of compound 1 ( $5 \times 10^{-6}$  M, CHCl<sub>3</sub>) before (red) and after hydrolysis (black).



Figure S5. Absorption spectra of compound 1 ( $5 \times 10^{-6}$  M, CHCl<sub>3</sub>) before (red) and

after hydrolysis (black).



Figure S6. DLS data of compound 1 ( $5 \times 10^{-6}$  M, CHCl<sub>3</sub>) before (full circle) and after

hydrolysis (empty circle).







**Figure S7.** AFM of compounds **2** (upper) and **3** (lower, 10<sup>-3</sup> M, CHCl<sub>3</sub>) before (a,c) and after hydrolysis (b,d).







**Figure S8. a**: Emission spectra of compound **1** ( $10^{-5}$  to  $10^{-3}$  M) with Tb ions (Tb was fixed as  $10^{-5}$  M, CHCl<sub>3</sub>/EtOH = 99/1). Ex = 280 nm. **b**: Emission spectra of compound **2** ( $10^{-5}$  to  $10^{-3}$  M) with Tb ions (Tb was fixed as  $10^{-5}$  M, CHCl<sub>3</sub>/EtOH = 99/1). Ex = 280 nm. **c**: Emission spectra of compound **3** ( $10^{-5}$  to  $10^{-3}$  M) with Tb ions (Tb was fixed as  $10^{-5}$  M, CHCl<sub>3</sub>/EtOH = 99/1). Ex = 280 nm.