## **Supporting Information**

## Luminescent Protein-Rare Earth Fluoride Nanoflowers

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**Figure S1.** Nitrogen adsorption/desorption isotherms of trypsin-Na<sub>5</sub>Yb<sub>9</sub> $F_{32}$  hybrid nanoflowers (a), and the corresponding BJH pore sizes distribution plot of trypsin-Na<sub>5</sub>Yb<sub>9</sub> $F_{32}$  (b).



**Figure S2.** XRD patterns of hybrid nanomaterials prepared at different incubation time: 5 min, 30 min, 3 hrs, 5 hrs, 12 hrs and 36 hrs. Standard XRD pattern of  $Na_5Yb_9F_{32}$  (JCPDS No. 27-1426) was shown beneath the plots of hybrid nanomaterials.



**Figure S3**. SEM images of the trypsin-Na<sub>5</sub>Yb<sub>9</sub>F<sub>32</sub> hybrid nanoflowers treated by calcination (a), and glutaraldehyde/ EDTA (b). Calcination was performed at 350 °C for 6 hrs. The trypsin-Na<sub>5</sub>Yb<sub>9</sub>F<sub>32</sub> hybrid nanoflowers were first treated with glutaraldehyde (0.8 wt %) and then EDTA (1 wt %).