## **Electronic Supplementary Information (ESI)**

High-quality elliptical iron glycolate nanosheets: selective synthesis and chemical conversion into Fe<sub>x</sub>O<sub>y</sub> nanorings, porous nanosheets, and nanochains with enhanced visible-light photocatalytic activity

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**Fig. S1** XPS spectra of the typical precursor samples. (a) Survey spectrum and deconvolution of (b) C1s peak, (c) Fe 2p3/2 peak and (d) O1s peak.

The surface chemistry composition of the precursors was analyzed with XPS, as shown in Fig. S1. The binding energies obtained in the XPS analysis are standardized for specimen charging using C1s as the reference at about 285 eV. Three elements (i.e., Fe, O, and C) were detected in the survey XPS spectrum (Fig. S1a). The C1s spectrum (Fig. S1b) was best fitted with three peaks at 287.3, 286.1, and 285.19 eV, which correspond to C=O, C-O, and C-C, respectively.<sup>1</sup> The existence of dual iron oxidation states (Fe<sup>2+</sup> and Fe<sup>3+</sup>) led to the broadness of the Fe 2p3/2 peak. The Fe 2p3/2 spectrum was fitted with six peaks, as shown in Fig. S1c. In this study, 0 to 2 peaks referred to Fe<sup>2+</sup> and 3 to 6 peaks pertained to Fe<sup>3+</sup>. The O1s spectrum was best fitted with three peaks at 529.0, 530.3, and 531.2 eV (Fig. S1d). The lowest binding energy of the O1s peak at 529.0 eV corresponded to the lattice oxygen in the Fe<sub>3</sub>O<sub>4</sub> phase (Fig. S1d), which further confirmed the presence of Fe<sub>3</sub>O<sub>4</sub>. The peak located at 530.3 eV was associated with hydroxyl species,<sup>2</sup> and the remaining higher binding energy peak at 531.2 eV was associated with the crystallization of water oxygen. The

presence of O<sup>2-</sup>, OH<sup>-</sup>, and H<sub>2</sub>O species is commonly observed in iron glycolate. The relative atomic ratio ( $\alpha$ ) can be calculated with the equation:  $\frac{n_i}{n_j} = \frac{I_i}{I_j} \times \sqrt{\frac{E_{k_i}}{E_{k_i}}}, \text{ where } n_i \text{ is }$ 

the number of surface atoms,  $I_i$  is the peak area, and  $E_{k_i}$  is the photoelectron kinetic energy. The Fe<sup>3+</sup>/Fe<sup>2+</sup> relative atomic ratio was approximately 3.3, and the relative atomic ratio of the lattice oxygen to the crystallization water ( $\alpha$ ) was 3.0. Based on the above data, we may reasonably conclude that the chemical formula of the iron glycolates in this study can be expressed as  $Fe_{33}^{IIII}Fe(C_2H_3O_3)_{III,9}g4H_2O$ .



**Fig. S2** SEM images of the precursor samples formed at 200 °C for 6 h under various  $Fe^{3+}$  concentrations of (a) 0.125, (b) 0.250, and (c) 0.375 M; under  $Fe^{3+}$  concentration of 0.125 M and at 200 °C for different times of (d) 3, (e) 12, and (f) 24 h; at (g) 150 °C, (h) 180 °C, and (i) 220 °C for 12 h with  $Fe^{3+}$  concentration of 0.125 M.

Comparative experiments were carried out to investigate the influences of Fe<sup>3+</sup> concentration, reaction temperature ( $T_r$ ), and reaction time (t) on the morphology and phase structure of the products. When temperature and reaction time remained unchanged (at 200 °C for 6 h), increasing Fe<sup>3+</sup> concentration favored the growth of nanosheets. All the products were bright yellow powders of iron glycolate; the

powders took on elliptically sheet-like configurations with the length of the long axis increasing from 150–230 nm at  $[Fe^{3+}] = 0.125$  M to 350–400 nm at  $[Fe^{3+}] = 0.375$  M (Figs. S2a to S2c). If Fe<sup>3+</sup> concentration and temperature remained constant, the products were nonmagnetic bright yellow powders at 3-12 h and magnetic black powders at 24 h. Figs. S2d to S2e show that the bright yellow powders were nanosheets, whose long axis length ranged from 50-80 nm at 3 h to 150-230 nm at 12 h; the black Fe<sub>3</sub>O<sub>4</sub> powders consisted of uniform hollow microspheres with diameter of 0.8–1.0  $\mu$ m (Fig. S2f). T<sub>r</sub> is another important factor used to tailor the morphology of the precursors. At 150-200 °C, bright yellow, nonmagnetic iron glycolate powders were easily obtained, with long axis length increasing from 50 nm at 150 °C to 150-230 nm at 200 °C (Figs. S2g and S2e), whereas the black magnetic  $Fe_3O_4$  powders were formed at 220 °C and composed of 0.8–1.0  $\mu m$  hollow microspheres (Fig. S2i). In addition, using urea and hexamethylenetetramine as base could also synthesize sheet-like precursors. Our data demonstrated that short reaction time, low temperature, and low concentration facilitated the formation of small IGNSs; long reaction time and high temperature contributed immensely to the growth of Fe<sub>3</sub>O<sub>4</sub> hollow microspheres.



Fig. S3 TGA and DSC curves of the typical precursor samples sintered under different atmospheres: (a) air and (b)  $N_2$ .

The compositional changes associated with the calcination process were also followed using the thermogravimetric (TG) and differential scanning calorimetric

(DSC) analysis methods. The initial samples were prepared by drying in a vacuum oven at 60 °C for 6 h. Figs. S3a and S3b showed the TG and DSC curves recorded under a flow of nitrogen gas or air, respectively. In air, the TG curves exhibited a twostep pattern for weight loss of 1.5% and 24.31% in the temperature range of 25 to 200 °C and 200 to 400 °C, respectively. The first weight loss could be attributed to the desorption of physically adsorbed water and ethylene glycol molecules, and the second one could be ascribed to the removal of ethylene glycol units and the degradation of organic groups contained in the precursor nanosheets.<sup>3</sup> Under N<sub>2</sub>, TG curves showed a third-step weight loss. A weight loss of 1.12% was observed for the first step, 23.72% for the second step (was caused by the decomposition and removal of the organic constituent in the precursor during heat treatment), and 5.55% for the third step (corresponding to the conversion of  $Fe_3O_4$  to Fe). In the DSC curves of the precursors (Fig. S3), an obvious exothermic peak corresponding to the sharp mass loss can be observed around 314.7 °C for in air and 323.3 °C under N<sub>2</sub>, respectively. The exothermic peak indicated the decomposition temperature of the precursor, which helped us to determine the temperature for the thermal treatment of the precursor. From Fig. S3a and S3b, we can see that the DTA curve in air exhibits a stronger exotherm at lower temperature (314.7 °C) than that under  $N_2$  (323.3 °C). The precursor is possibly a kind of ferrous alkoxide.



Fig. S4 SEM images of the sintered samples formed via sintering the typical precursors under  $N_2$  at (a) 300 °C, (b) 600 °C, and (c) 700 °C for 2h.



Fig. S5 SEM images of the sintered samples formed via sintering the typical precursors under  $N_2$  at 300 °C for (a) 10 min, (b) 30 min, and (c) 1 h.

Sample	RhB concentration (mol·L <sup>-1</sup> )	catalyst concentration (g·L <sup>-1</sup> )	discoloration rate (%) and time (min)	Ref.
γ-Fe <sub>2</sub> O <sub>3</sub> @SiO <sub>2</sub> @Ce-TiO <sub>2</sub>	1.0×10 <sup>-4</sup>	1.0	88/180	4
γ-Fe <sub>2</sub> O <sub>3</sub> /ZnO	1.0×10 <sup>-5</sup>	0.5	91.1/50	5
$\alpha$ -Fe <sub>2</sub> O <sub>3</sub> hollow spheres	1.0×10 <sup>-5</sup>	2.5	57/180	6
γ-Fe <sub>2</sub> O <sub>3</sub> /TiO <sub>2</sub> Janus hollow bowls	1.0×10 <sup>-5</sup>	2.5	75/180	7
$Bi_2Fe_4O_9/\gamma$ - Fe_2O_3heterojunctions	1.0×10 <sup>-5</sup>	1.0	80/120	8
$\alpha$ -Fe <sub>2</sub> O <sub>3</sub> nanocubes		0.2	100/180	9
$\alpha$ -Fe <sub>2</sub> O <sub>3</sub> nanorods	2.0×10 <sup>-5</sup>		100/70	
$\alpha$ -Fe <sub>2</sub> O <sub>3</sub> nanoplates			50/180	
$\alpha$ -Fe <sub>2</sub> O <sub>3</sub> nanorods			100/70	
iron glycolate nanosheets				This
$\gamma$ -Fe <sub>2</sub> O <sub>3</sub> porous nanosheets	2.0×10 <sup>-5</sup>	0.2	100/120	work
$\alpha$ -Fe <sub>2</sub> O <sub>3</sub> / $\gamma$ -Fe <sub>2</sub> O <sub>3</sub> porous nanosheets			100/120	

<b>Table St</b> Summary of photocatary de properties for regos and its composites
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