Supplementary Information for

Iron(III) oxyhydroxide and oxide monoliths with controlled multiscale porosity: synthesis and their adsorption performance

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gure S1. The molecular structure of Congo red.



Figure S2. Time evolution of pH in the reaction solution: $w_{\text{PEO}} = 0$ mg.

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Figure S3. BJH mesopore size distributions obtained from the N₂ adsorption branch of asdried iron(III) oxyhydroxide monoliths prepared with varied amounts of PEO.



Figure S4. Change in diameter of the iron(III) oxyhydroxide and oxide monoliths during gelation, drying, and heat treatment processes.



Figure S5. (A, B) Pore size distributions of iron(III) oxyhydroxide or oxide monoliths heat treated at different temperatures.



Figure S6. (A, B) UV-Vis adsorption spectra of Congo red solutions treated by different amounts and kinds of samples.



Figure S7. Adsorption isotherms for Congo red on the different kinds of samples.



Figure S8. (A, C) SEM images of the crushed macroporous monoliths: $w_{PEO} = 50$ mg. (B, D) SEM images of the crushed non macroporous monoliths: $w_{PEO} = 0$ mg.

Table S1. Zeta potential and corresponding pH of the different samples.

Sample	Zeta potential, V/mV	pН
as-dried (50): amorphous FeOOH	26	4.0
as-dried (0): amorphous FeOOH	27	4.4
sample heated 250 °C : -	-3.0	5.0
sample heated 300 °C: crystalline α -Fe ₂ O ₃	-6.8	5.5
sample heated 350 °C : crystalline α -Fe ₂ O ₃	-18	5.5