

Supporting Information

Synthesis of FeOOH-Loaded Aminated Polyacrylonitrile Fiber for Simultaneous Removal of Phenylphosphonic Acid and Phosphate from Aqueous Solution

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S1. Materials and reagents

HCl, HNO₃ and H₂SO₄ were purchased from Xilong Scientific Co., Ltd. Ammonium molybdate, *p*-chloronitrobenzene, ascorbic acid and Na₂S₂O₈, KH₂PO₄ were purchased from Shanghai Aladdin Biochemical Technology Co., Ltd. Antimonyl potassium tartrate was obtained by Saan Chemical Technology Co., Ltd. Methyl alcohol. Methyl alcohol Shanghai Macklin Biochemical Co., Ltd. Na₂CO₃, NaHCO₃, NaOH and K₂CO₃ were supplied from Shanghai SuYi Chemical Reagent Co., Ltd. K₂SO₄, KCl and NaCl were provided by Sinopharm Group Chemical Reagent Co., Ltd. Methanol is chromatography-grade pure. The water used in this experiment is deionized water and chemicals used in this study are analytical grade unless otherwise specified.

S2. Characterization

The surface morphology and element distribution of the fibers were determined by scanning electron microscope (S-4800, Hitachi) and X-ray energy dispersive spectrometer (EDS, X-Max N 150). The changes of functional groups and elemental content (C, N and O) on the fiber surface were measured by Fourier Infrared Spectrometer (Nicolette is50, USA) and elemental analyzer (Vario EL Cube, Germany), respectively. The test method of Fourier Infrared chromatography is to cut fiber into pieces, mix the fiber with potassium bromide, and press them into thin sheets, then measure it in the wavenumber range of 4000-400 cm⁻¹. The internal crystal structure of different fibers is performed by the D/MAX-2500 X-ray diffraction (XRD) (the Confucian Co., Ltd.) within the range of 10°-90° at a speed of 4 ° per minute. Thermal stability of the fibers was tested by a STA409F5TGA/DSC simultaneous thermal analyzer (Netzsch company, Germany) from room temperature to 800 °C at a rate of 10 °C min⁻¹ in nitrogen atmosphere. The surface structure and elemental composition of the fibers were characterized by X-ray photoelectron spectroscopy (XPS, PERKIN ELMZR, USA), Full-spectrum scan flux energy is 150 eV in 1 eV steps; narrow-spectrum scan flux energy is 50 eV in 0.1 eV steps. The species of free radicals in the reaction system were determined by Electron paramagnetic resonance spectrometer (EPR, Bruker EMXplus, Germany) at the 5th min from the beginning of the reaction.

S3.Steps and methods for the determination of PPOA and phosphate

PPOA: After the degradation experiment, the fibers were separated from the solution with tweezers, 1 mL of sample solution was filtered by 0.22 μm filter membrane, and 1 mL of methanol was added immediately for quenching. The remaining PPOA content was then determined by HPLC. The set conditions of the HPLC are: methanol as a mobile

phase, methanol to water ratio of 4:1, flow rate of 0.8, wavelength of 220 nm, the use of BaseLine C18 liquid chromatography column.

Phosphate: After the adsorption test, the fibers were separated from the solution with forceps, and 1 mL of the sample solution was taken into a volumetric flask and the remaining phosphate content was determined in a spectrophotometer using the molybdenum blue colorimetric method. The wavelength of the spectrophotometer was used at 700 nm.

S4. Reusability experiments

S4.1 The step of degradation cycle by PAN_{AF}-FeOOH

To investigate the cycling performance of PAN_{AF}-FeOOH. After 30 mg PAN_{AF}-FeOOH was added to a 100 mL glass vial containing 5 mg L⁻¹ of PPOA and 0.5 mmol L⁻¹ PDS at 25 °C for the reaction. When the reaction is over, the material was recovered using forceps and washed repeatedly with deionized water, then the fiber undergoes the next repeated reaction. The above process was repeated successively to test the reusability ability of PAN_{AF}-FeOOH.

S4.2 The step of adsorption cycle by PAN_{AF}-FeOOH

After weighing 50 mg of dried PAN_{AF}-FeOOH into 20 mL of KH₂PO₄ solution with an initial phosphate concentration of 2 mg P L⁻¹ and stirring for 1 h, the phosphate concentration in the solution was measured by removing the fibers with forceps. The fiber was rinsed repeatedly with deionized water to clean the phosphate adhering to the surface, then the fiber transferred to 20 mL of sodium hydroxide solution with an initial concentration of 0.05 mol L⁻¹ for desorption for 1 h. The phosphate concentration in the solution was measured. Then the next adsorption experiment was conducted directly after the fiber is washed.

S5. Synthesis and optimization of PAN_{AF}-FeOOH

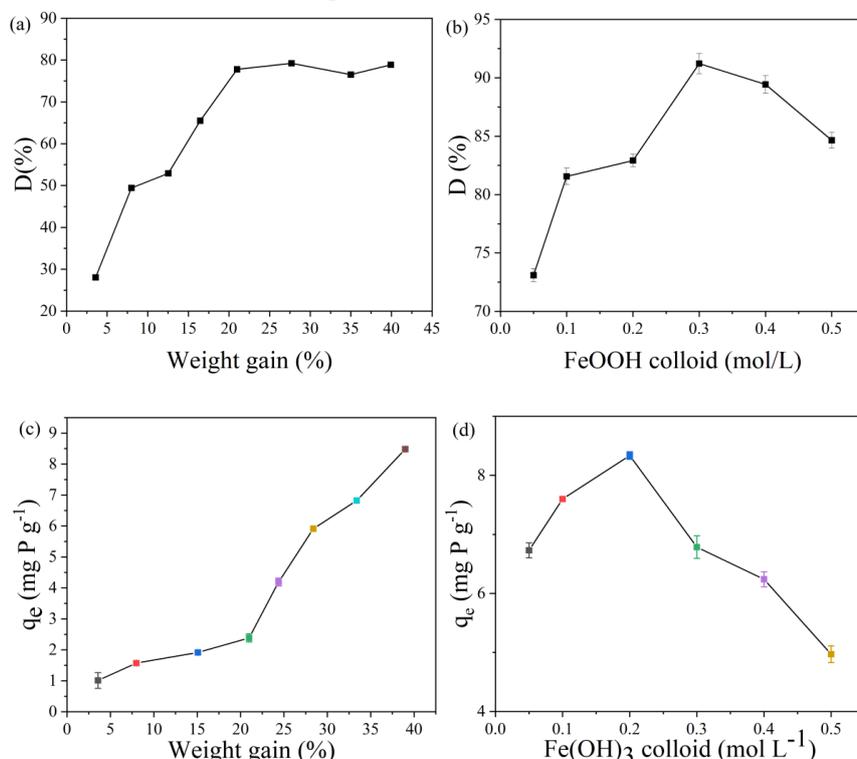


Figure S1. (a) The degradation effect of PPOA by 0.3 mol/L Fe(OH)₃ combined with aminated fibers with different weight gain; (b) The degradation effect of PPOA by aminated fibers with weight gain

of 38% combined with different concentrations of Fe(OH)₃ colloid; (c) Phosphate adsorption capacity of 0.2 mol/L Fe(OH)₃ combined with aminated fibers with different weight gain; (d) Phosphate adsorption capacity of aminated fibers with weight gain of 38% combined with different concentrations of Fe(OH)₃ colloid.

S6. Adsorption kinetics

Table S1. Kinetic parameters of phosphate adsorption by PANAF-FeOOH.

T (K)	Pseudo-first-order kinetic model			Pseudo-second-order kinetic model		
	K ₁ (min ⁻¹)	q _e (mg P g ⁻¹)	R ²	K ₂ (g mg ⁻¹ min ⁻¹)	q _e (mg P g ⁻¹)	R ²
288	0.485	7.91	0.907	0.094	8.38	0.997
298	0.575	8.27	0.872	0.105	8.76	0.983
308	0.607	8.79	0.850	0.108	9.28	0.992

S7. Adsorption thermodynamics

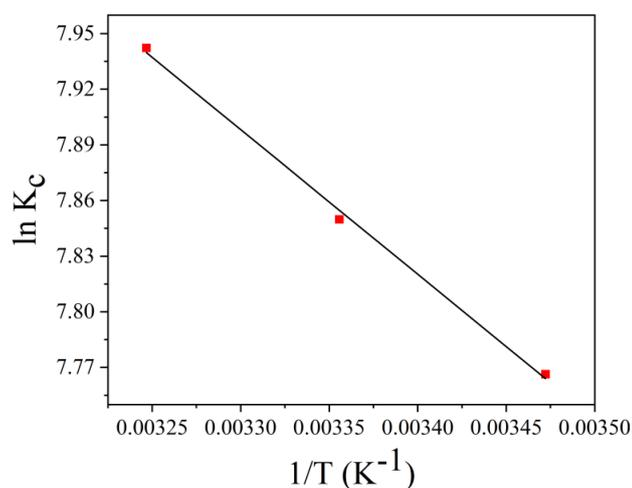


Figure S2. Fitting diagram of thermodynamics of phosphate.

Table S2. Thermodynamic parameters of phosphate adsorption by PANAF-FeOOH.

T (K)	G° (kJ mol ⁻¹)	ΔH° (kJ mol ⁻¹)	ΔS° (J mol ⁻¹ K ⁻¹)
288	-18.596	6.482	87.056
298	-19.448		
308	-20.338		

S8. Adsorption isotherm

Table S3. Isotherm parameters of phosphate adsorption by PANAF-FeOOH.

Langmuir			Freundlich		
q _e (mg P g ⁻¹)	KL (L mg ⁻¹)	R ²	K _F [mg/g·(L/mg) ^{1/n}]	1/n	R ²
9.918	0.281	0.990	2.954	0.330	0.921

S9. Continuous flow experiment

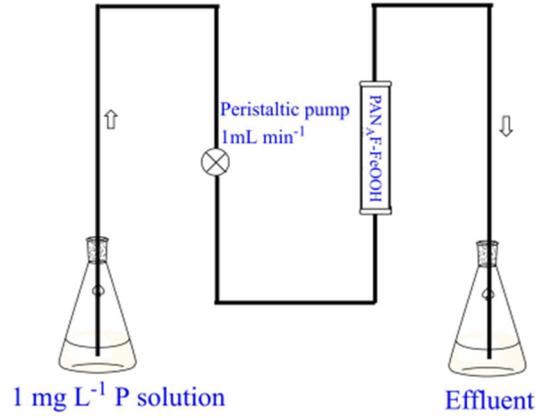


Figure S3. Simple diagram of continuous flow process device.

S10. EPR

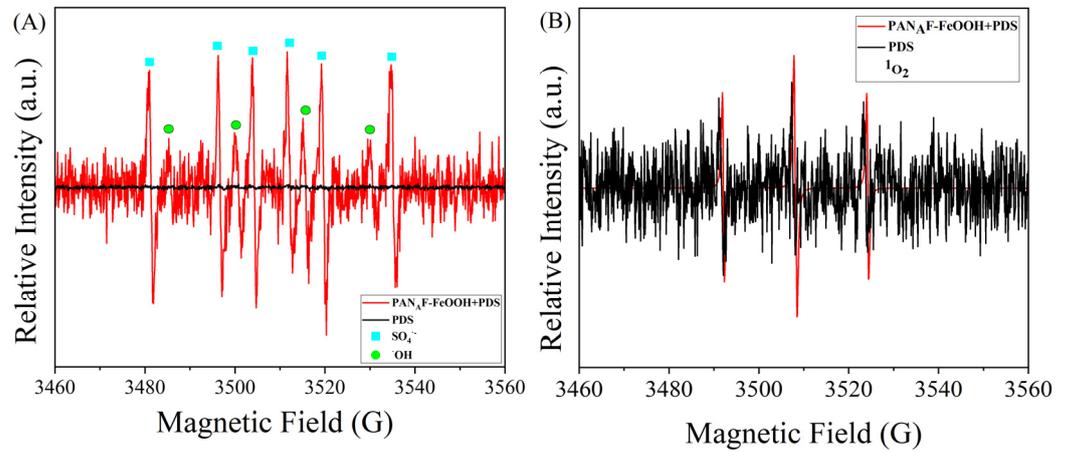


Figure S4. (A) EPR spectrum of $\text{SO}_4^{\bullet-}$ and $\bullet\text{OH}$ of PDS/PDS- $\text{PAN}_A\text{F-FeOOH}$ alytic system; (B) EPR spectrum of $^1\text{O}_2$ of PDS/PDS- $\text{PAN}_A\text{F-FeOOH}$ alytic system.

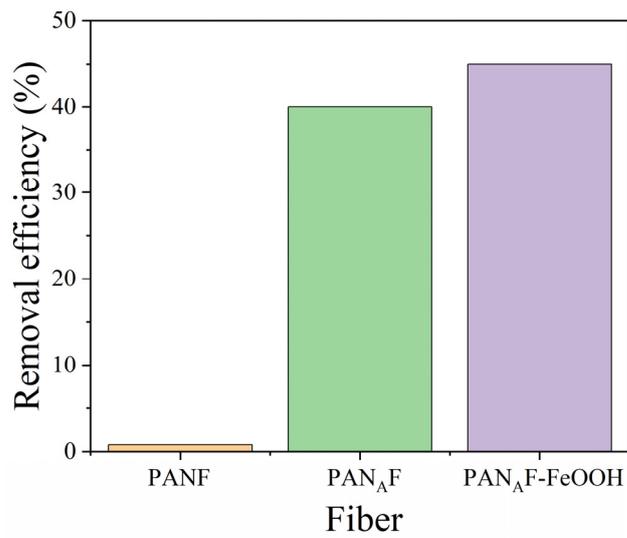


Figure S5. Adsorption capacity of $\text{PAN}_A\text{F-FeOOH}$ for PPOA (30 mg of $\text{PAN}_A\text{F-FeOOH}$ was soaked in 50 mL of PPOA with concentration of 5 mg P L^{-1} for 2 h, $\text{pH}=5$).

S11. XPS

Table S4. The mass and atomic percentage of the functionalized fiber obtained by XPS.

Fiber	C	N	O	Fe	P
PANF	73.78	7.71	18.51	0	0
PAN _A F	79.55	10.14	10.31	0	0
PAN _A F-Fe	46.34	8.65	36.82	8.2	0
PAN _A F-Fe-P	57.67	3.13	30.44	6.01	2.76

S12. Comparison of different adsorbents

Table S5. Comparison of different phosphate adsorbents.

Number	Materials	q_m (mg P g ⁻¹)	Run	Adsorption equilibrium	References
1	FeOOH-GO	5.81	/	7 h	[1]
2	Amorphous FeOOH	98.69	/	4 h	[2]
3	FeOOH@MS	115.5	/	2 h	[3]
4	FeOOH/BF	39.08	3	12 h	[4]
5	FSP-f	7.6	3	48 h	[5]
6	β -FeOOH/GO	45.2	/	120 min	[6]
7	α -FeOOH-600BC	11.39	/	192 h	[7]
8	PAN _A F-FeOOH	9.92	5	30 min	This manuscript

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