

Supplementary Information

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3 **Characterization of NiFe-LDHs**

4 X-ray diffraction (XRD) measurements were conducted to explore the crystallinity
5 and phase composition of as-prepared samples on a Rigaku D/MAX-2550
6 diffractometer using Cu-K α radiation ($\lambda = 0.15418$ nm). The morphology and size of
7 3D NiFe-LDHs were examined by field emission scanning electron microscope
8 (FESEM/EDS, ZEISS Ultra Plus) using an accelerating voltage of 15 kV. X-ray
9 photoelectron spectroscopic (XPS) measurement of 3D NiFe-LDHs was performed by
10 VG Multilab 2000 to examine the elemental composition and chemical state. The FT-
11 IR measurements were using the AVATAR 360 spectrometer (Nicolet instrument
12 Corporation, America) in the region 4000-500 cm^{-1} . All binding energies were
13 calibrated with the saturated hydrocarbon C 1s peak at 284.5 eV. The specific surface
14 area was tested by the Brunauer-Emmett-Teller (BET) model. The active species were
15 certified by using an Electron Spin Resonance (ESR, Bruker A200). LC/MS/MS
16 (Agilent1100HPLC-tandem an Agilent 6410 Triple Quadruple mass spec-trometer
17 (MS/MS)) was employed to detect the degradation intermediates derived from TC.
18 Electrochemical tests were performed at an electrochemical workstation (CHI 660E,
19 CH Instruments Inc, Shanghai, China) using Na₂SO₄ (0.1 M) as an electrolyte and a
20 three-electrode system (working electrode: CC/NiFe-LDHs material; opposite
21 electrode: carbon rod, reference electrode: Hg/HgO electrode).

22 **H₂O₂ decomposition**

23 The iodometric method was used to test H₂O₂. Fluorescence reagents (0.1
24 M potassium hydrogen phthalate and 0.4 M potassium iodide aqueous solution)
25 were typically complexed with the reaction system in a specific volume. The
26 H₂O₂ concentration was determined during each reaction phase by measuring th
27 e absorbance at 220 nm with a UV-Vis spectrophotometer. The H₂O₂ breakdo
28 wn tests were carried out in an ·O₂⁻ saturated, visible light-irradiated environme
29 nt. In the Quartz tube, 30 mg of catalyst was suspended in 50 mL of H₂O₂ a

30 queous solution (10 mM). The apparatus was then exposed to visible light for
31 90 minutes before measuring the remaining H₂O₂ concentration.

32 **Cyclic experiment**

33 Using xenon lamp to simulate sunlight as a light source, add 30 mg catalyst into a
34 beaker containing 50 mL 30 mg/L tetracycline solution, place the beaker in a magnetic
35 stirrer and stir for 30 min to reach adsorption equilibrium, turn on the light source, add
36 the corresponding amount of H₂O₂ to start the degradation experiment and time. The 3
37 mL samples were taken from the beaker at a specific time point, filtered by 0.22 μ m
38 filter membrane, and the absorbance was measured by ultraviolet spectrophotometer.
39 After the reaction, the corresponding volume of tetracycline was added to the beaker
40 and mixed evenly, so that the concentration of the TC in the beaker was maintained at
41 30 mg/L, and then H₂O₂ was added to the solution to start the degradation experiment,
42 which was repeated for six times. Each group of experiments was carried out three
43 times to ensure the accuracy of experimental data.

44 **Methods for analysis of Ni and Fe content**

45 In this study, the contents of Ni and Fe in the reaction solution and on NiFe-LDHs
46 were measured by Inductively Coupled Plasma Optical Emission Spectrometer, ICP-
47 OES 167nm-785nm/725 Agilent, USA) determination The solid catalyst was dissolved
48 with aqua regia, the pH was adjusted to 5-7 after complete dissolution, and the volume
49 was finally fixed to 50 mL for analysis. The accuracy and precision were controlled by
50 blank and parallel samples. The relative deviation of Ni and Fe in parallel samples was
51 within 16%~3.4%, and the results of blank samples were lower than the lower limit of
52 detection.

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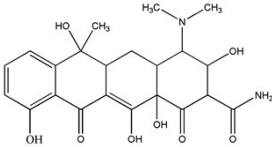
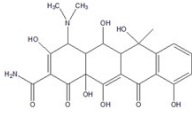
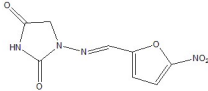
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Table S1 Molecular information of TC, OTC and FT

antibiotic	Formula	Structure	Molecular weight	UV, λ_{\max} (nm)
TC	$C_{22}H_{24}N_2O_8$		444.45	365
OTC	$C_{22}H_{24}O_9N_2$		460.434	355
FT	$C_8H_6N_4O_5$		238.16	367

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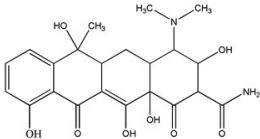
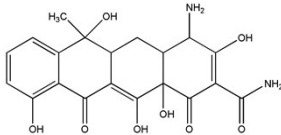
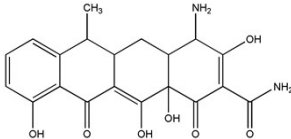
Table S2 Water sample data of FAW

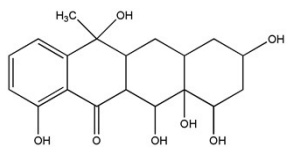
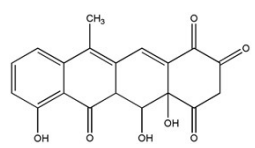
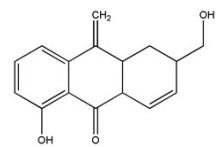
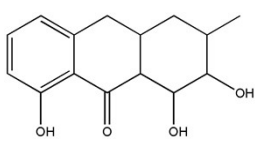
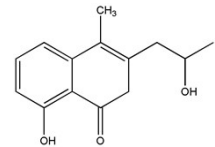
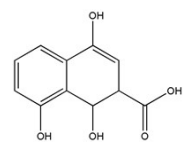
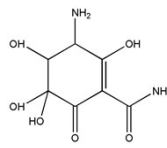
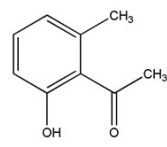
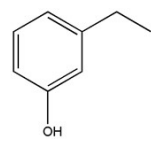
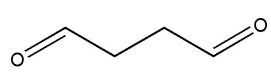
Sewage type	Source of sewage	Sewage flow rate	Major pollutant	COD
Electrophoretic wastewater	Automobile coating technology	14 m ³ /h	Polymer resins, emulsified oils, surfactants, PO ₄ ³⁻ , Pb ²⁺	2300 mg/L

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Table S3 Intermediate products of TC-HCl degradation

m/z	Molecular formula	Structural formula
445	$C_{22}H_{24}O_8N_2$	
415	$C_{21}H_{22}O_8N_2$	
401	$C_{21}H_{22}O_8N_2$	

366	$C_{20}H_{20}O_8N_2$	
353	$C_{20}H_{19}O_7N_2$	
274	$C_{20}H_{21}O_7N$	
262	$C_{18}H_{21}O_6N$	
246	$C_{17}H_{19}O_5$	
221	$C_9H_{10}O_2$	
218	$C_5H_{10}O_3$	
135	$C_3H_4O_3$	
123	$C_3H_4O_2$	
85	C_3H_4O	

69 **Table S4** Content changes of nickel ion and iron ion before and after the reaction

Samples	Relative ratio			
	Ni 2p		Fe 2p	
	Ni ²⁺	Ni ³⁺	Fe ²⁺	Fe ³⁺
Fresh NiFe-LDHs	24.9%	75.1%	50.1%	49.9%
Used NiFe-LDHs	37.85%	62.15%	31.99%	68.01%

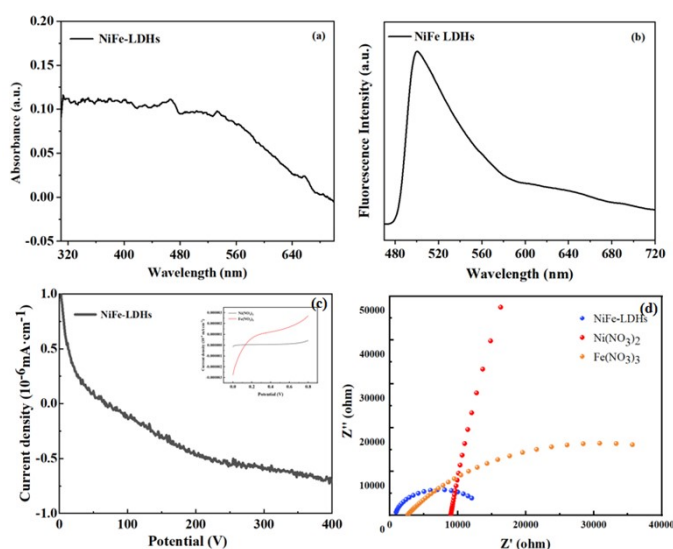
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71 **Table S5** Repeated use of metal ion leaching by catalysts

Samples	Ion leaching	
	Ni	Fe
Fresh NiFe-LDHs	6.55	10.21
Used NiFe-LDHs	3.92	3.14

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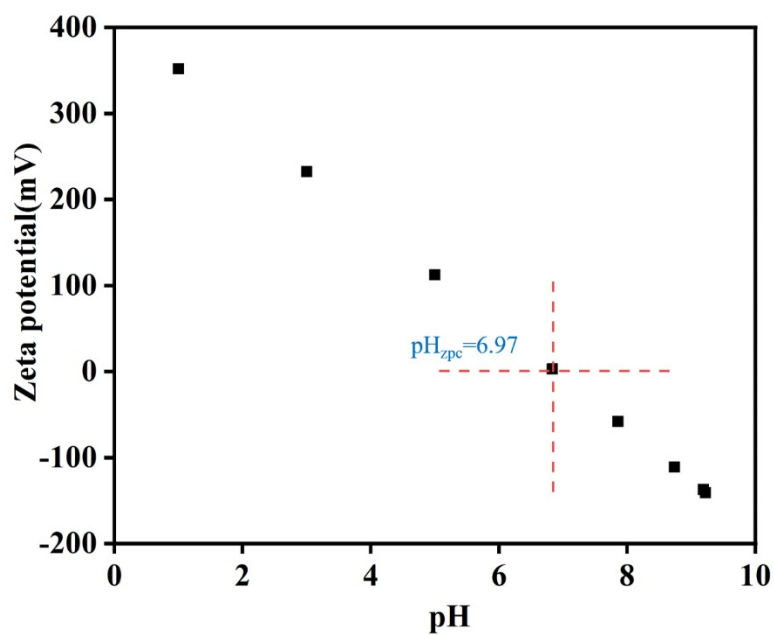


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Fig. S1 (a-d) UV-vis, PL, LSV and EIS of NiFe-LDHs

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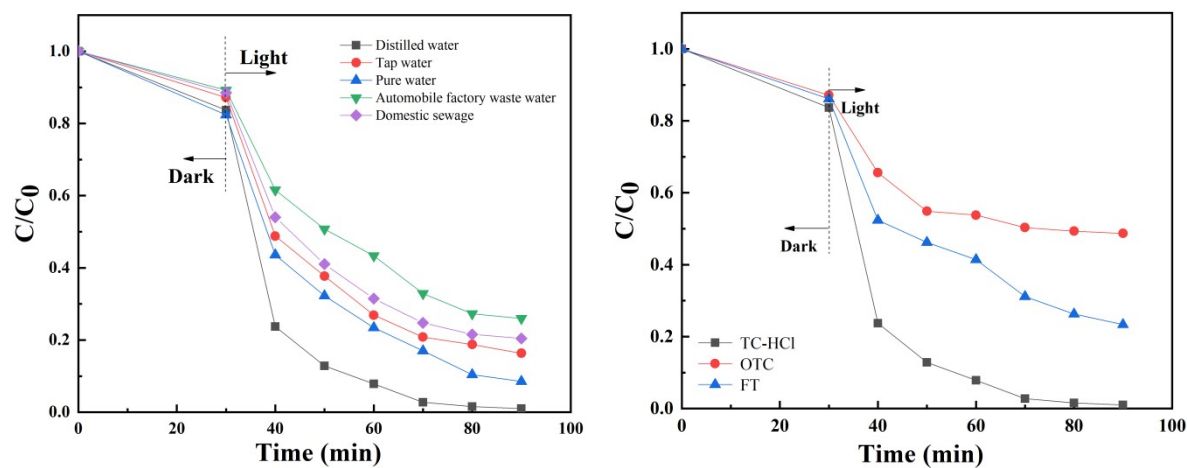
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Fig. S2 Isoelectric point of NiFe-LDHs

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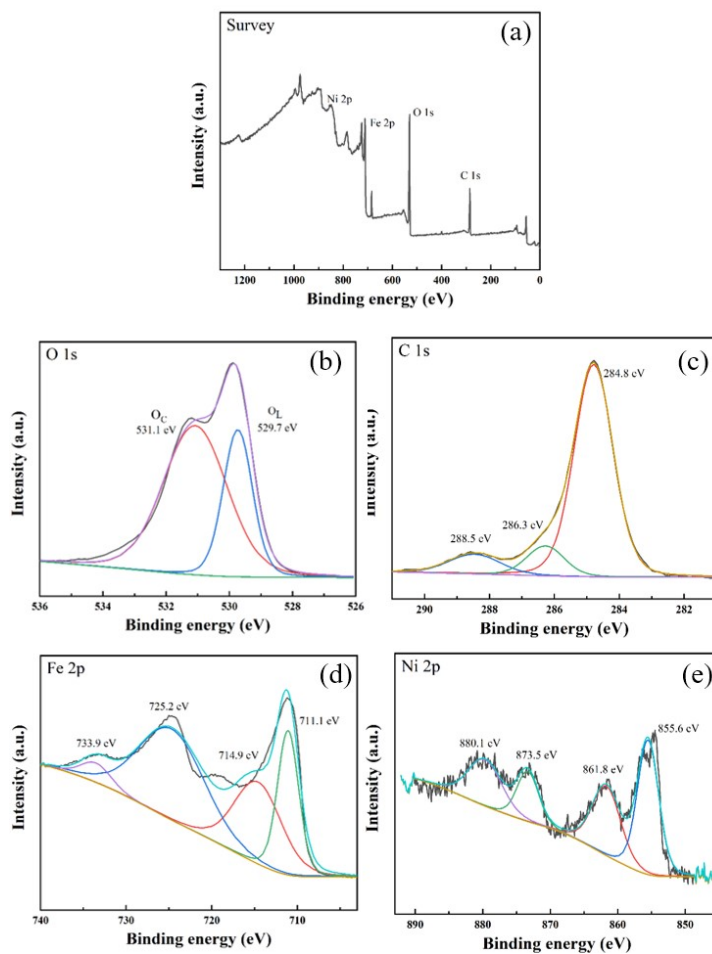
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Fig. S3 Effects of different water quality on degradation rate

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Degradation of other types of antibiotics by NiFe-LDHs

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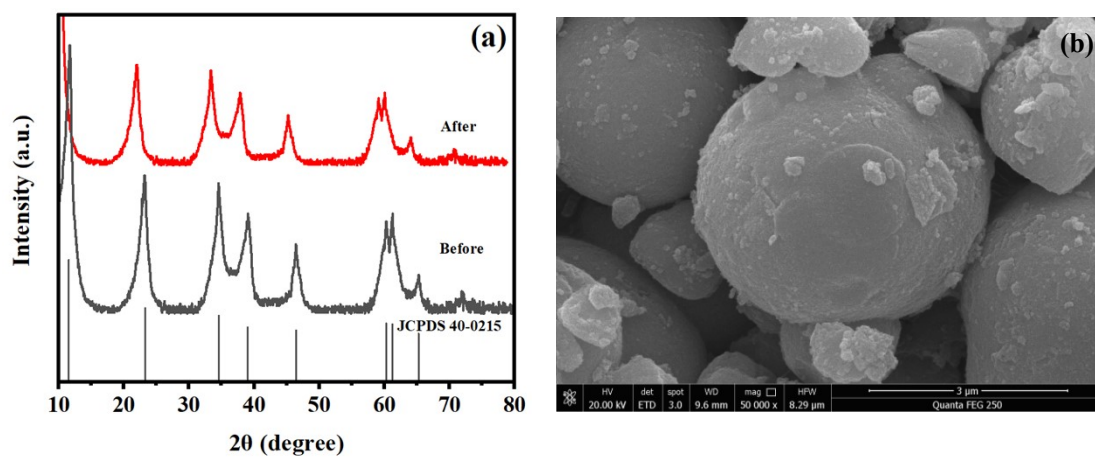


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Fig. S4 XPS diagram of NiFe-LDHs after four cycles of experiment

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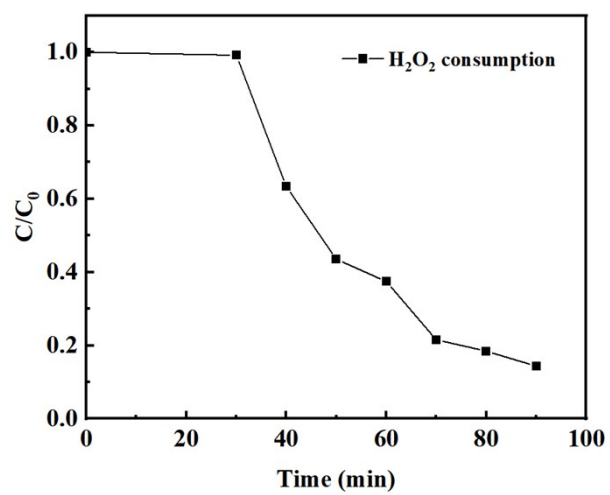


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Fig. S5 (a)XRD and (b)SEM of NiFe-LDHs after six cycles

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Fig. S6 Decomposition of H₂O₂ in different reaction systems.