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# Supplementary Information

## 3D NiCo-LDH hollow nanocages for rapid and efficient removal of tetracycline hydrochloride from water

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### Text S1

### Synthesis of NiCo-LDH nanosheets

$NiCl_2 \cdot 6H_2O$ (0.006 mol) and $Co(NO_3)_2 \cdot 6H_2O$ (0.003 mol) with Ni/Co radio of 2: 1 were mixed into 100 mL deionized water. NaOH solution (0.5 mol/L) was added dropwise into the mixture under vigorous stirring and adjusted pH to 10. The obtained sample was aged for 24 h. Prepared solid product was collected by centrifugation, washed with ultrapure water several times, and vacuum dried at 60℃ for 24 h.

### Text S2

### Evaluation of the antibacterial activity

A 50 μL aliquot of an Escherichia coli (E. coli) suspension ($1\times10^8$ CFU $mL^{-1}$) was spread evenly onto LB agar plates using the glass bead spreading method and allowed to absorb completely. Sterile circular filter paper discs (diameter ~1 cm) were immersed for 10 min in the supernatant of TC solution collected at different degradation time

points. The supernatant-loaded discs were then placed centrally on the E. coli-seeded LB agar plates. Following incubation at 37°C for 12 h, the zones of inhibition were measured.

**Text S3**

**Analysis of TC intermediates**

The mobile phase was acetonitrile/0.1% formic acid (22/78, v/v) at a flow rate of 0.25 mL/min; the column temperature was 25°C. TC-HCl and intermediates were estimated in the positive ion mode using ESI under the following conditions: m/z range, 50-650; DL temperature, 250°C; ion source temperature, 350°C; nebulization gas flow rate, 10 L/min; pressure, 45 psi.

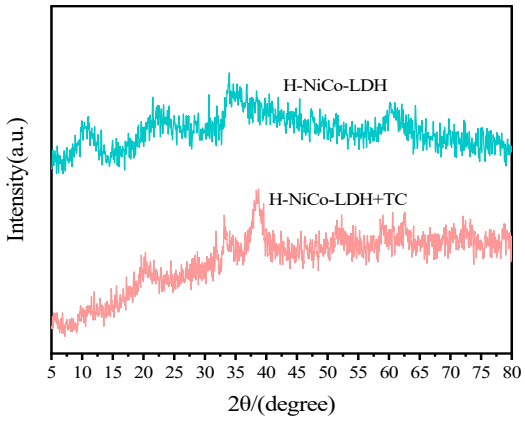



Fig. s1 XRD of H-NiCo-LDH+TC and H-NiCo-LDH

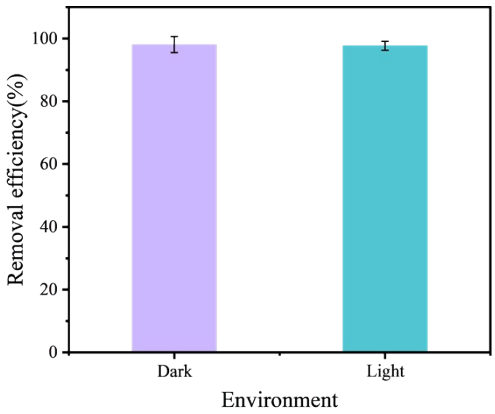



Fig. s2 Effect of light or dark removal TC

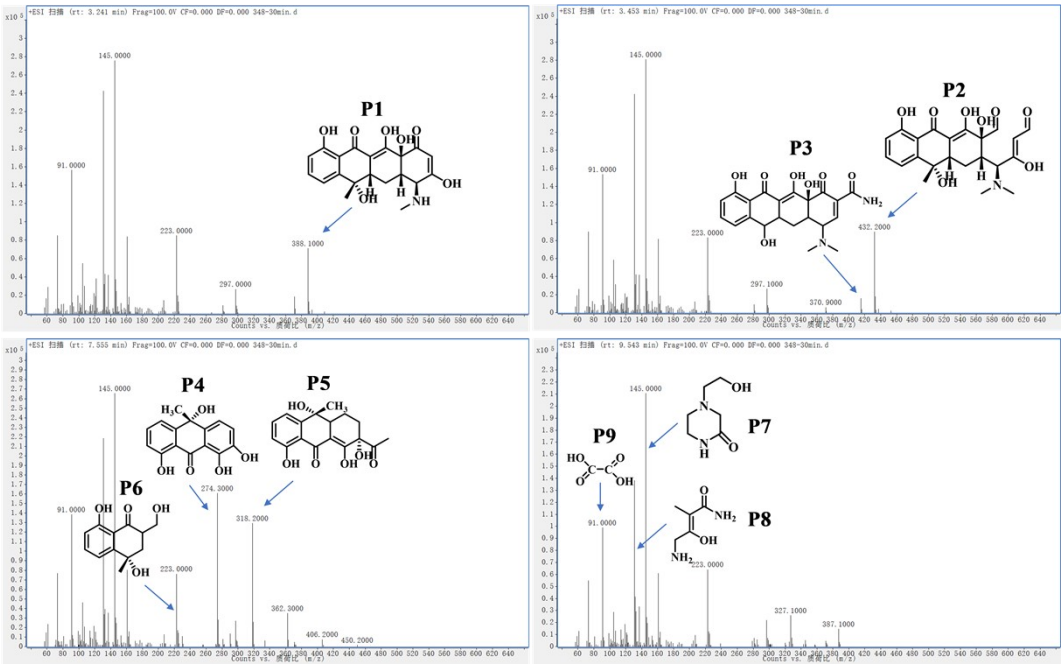



Fig. s3 Mass spectrum of the intermediate product after 5 min of TC degradation

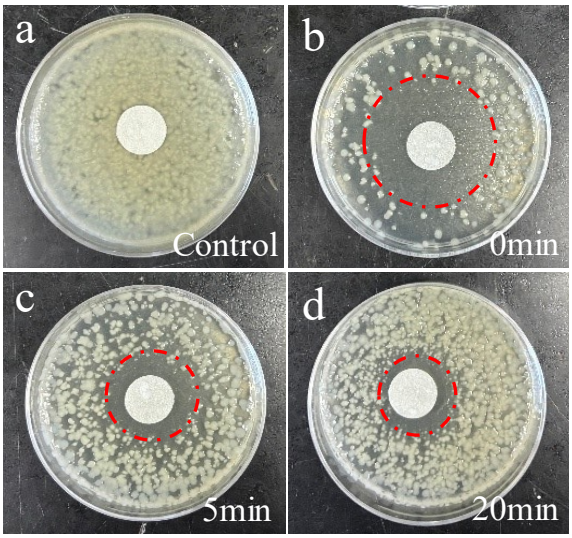
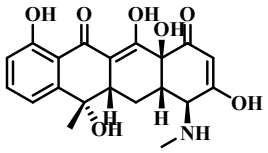
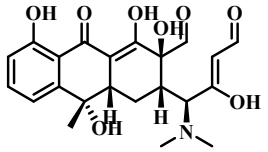
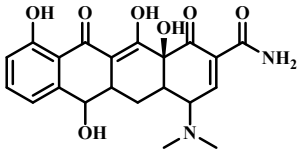
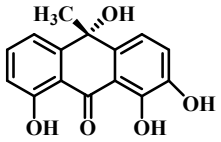
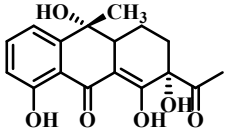
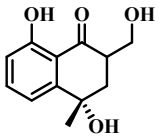
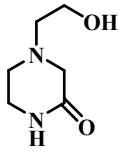
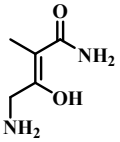
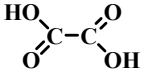



Fig.s4 Antibacterial properties of the supernatant at different times

Table s1. Structural formulas of various possible intermediates in the TC degradation of H-NiCo-LDH system identified by LC-MS

| Retention Time/min | Molecular formula | m/z $[M+H]^+$ | Possible structure | Compound |
|---|---|---|---|---|

| | | | | |
|---|---|---|---|---|
| 3.241 | $C_{20}H_{21}NO_7$ | 388 |  | P1 |
| | $C_{22}H_{25}NO_8$ | 432 |  | P2 |
| 3.453 | $C_{21}H_{22}N_2O_7$ | 415 |  | P3 |
| | $C_{15}H_{12}O_5$ | 274 |  | P4 |
| 7.555 | $C_{17}H_{18}O_6$ | 318 |  | P5 |
| | $C_{12}H_{14}O_4$ | 223 |  | P6 |
| | $C_6H_{12}N_2O_2$ | 145 |  | P7 |
| 9.543 | $C_5H_{10}N_2O_2$ | 131 |  | P8 |
| | $C_2H_2O_4$ | 91 |  | P9 |