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| | Element | Weight | Atomic |
|---|---------|--------|--------|
| Sample | | % | % |
| Fe ^{II} Fe ^{III} LDHNS | С | 6.22 | 12.03 |
| $[\mathrm{Fe_4}^{2+}\mathrm{Fe_2}^{3+}(\mathrm{OH}^{-})_{12}]^{2+}$ | Ο | 46.72 | 67.89 |
| $[SO_4^{2^-} \cdot 2H_2O]^{2^-}$ | S | 1.58 | 1.15 |
| | Fe | 45.47 | 18.93 |
| | Total | 100 | 100 |

Table S1 The elemental quantity of the element normalized.

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Fig. S4 (A) X-ray Photoelectron Spectroscopy (XPS) survey spectra of Fe^{II}Fe^{III} LDHNS. The high resolution XPS spectra of (B) Fe (2p), (C) O
(1s) and (D) S (2p) for Fe^{II}Fe^{III} LDHNS



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Fig. S5 (A) and (B) Effect of pH for TMB and ABTS, (C) and (D) effects of reaction time for

34 TMB and ABTS on the catalytic activity of $Fe^{II}Fe^{III}$ LDHNS.



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Fig. S6 Effects of $Fe^{II}Fe^{III}$ LDHNS concentration on the catalytic oxidation of TMB. Experiments were carried out using 50 µL of difference concentration $Fe^{II}Fe^{III}$ LDHNS, 100 µL of H₂O₂ (10 µM), 200 µL of 1 mM TMB (in ethanol) and 400 µL of 0.1 M pH 4.0 acetate buffer solution. The solution mixture was kept to react at ambient temperature (25 ± 1 °C) for 30 min





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Fig. S7 Effects of $Fe^{II}Fe^{III}$ LDHNS concentration on the catalytic oxidation of ABTS. Experiments were carried out using 50 µL of difference concentration $Fe^{II}Fe^{III}$ LDHNS, 100 µL of H₂O₂ (20 µM), 100 µL of ABTS (20 mM) and 500 µL of deionized water. The solution

47 mixture was kept to react at ambient temperature $(25 \pm 1 \text{ °C})$ for 30 min.

- Table S2 Comparison of maximum reaction rate (V_{max}) obtained from Fe^{II}Fe^{III} LDHNS for 48
- TMB and ABTS substrate. 49
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| Catalyst ^a | $V_{max} (10^{-8} \text{ M s}^{-1})$ | | Defense | Catalant ^a | V _{max} (10 ⁻⁸ M s ⁻¹) | | Defense |
|--|--------------------------------------|-------------------------------|-----------|---|--|-------------------------------|-----------|
| | TMB | H ₂ O ₂ | Kelerence | Catalyst | ABTS | H ₂ O ₂ | Reference |
| HRP ^a | 10.0 | 8.71 | 20 | HRP | - | - | 62 |
| Fe ₃ O ₄ NPs | 3.44 | 9.78 | 20 | Fe ₃ O ₄ -NH ₂ -SH | 60.0 | 51.0 | 63 |
| NiFe-LDHNS | - | - | 32 | NiCo-LDHs | 3.29 | 3.24 | 21 |
| Co-Al ELDH | - | - | 42 | Fe ₃ O ₄ @Au–Cys– | 10.05 | 2.03 | 23 |
| | | | | FA NPs | 10.95 | | |
| DNA/CuAl-LDHs | 4.09 | 2.30 | 43 | NiFe ₂ O ₄ | 17.48 | 14.11 | 30 |
| C-dot/NiAl-LDHs | 5.52 | 7.89 | 44 | Prussian blue NPs | - | - | 39 |
| Fe ^{II} Fe ^{III} LDHNS | 1.70 | 9.37 | This work | Fe ^{II} Fe ^{III} LDHNS | 3.46 | 7.19 | This work |

^aCatalyst: HRP (horseradish peroxidase), NPs (nanoparticles), ELDHs (exfoliated layered double hydroxides), 51 52 LDHs (layered double hydroxides), LDHNS (layered double hydroxide nanosheets).



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Fig. S8 The long-term stability of Fe^{II}Fe^{III} LDHNS for colorimetric detection of H₂O₂ by 56 using TMB (A) and ABTS (B) substrate for 21 days. 57