

Electronic Supporting Information (ESI)

Reline-assisted synthesis of fcc-hcp Ni/Ni(OH)₂ nanocatalyst for effective reductive hydrogenation of 4-nitrophenol

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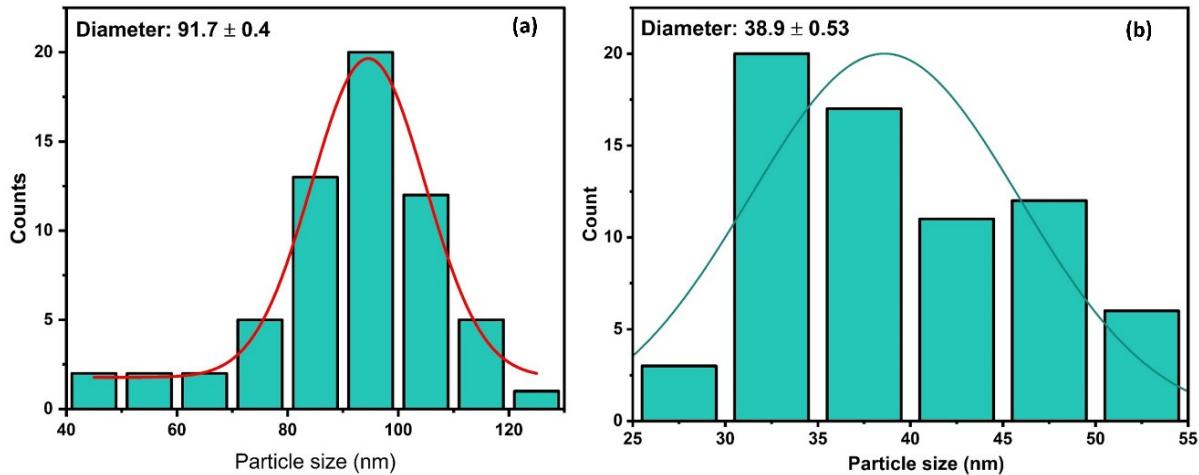
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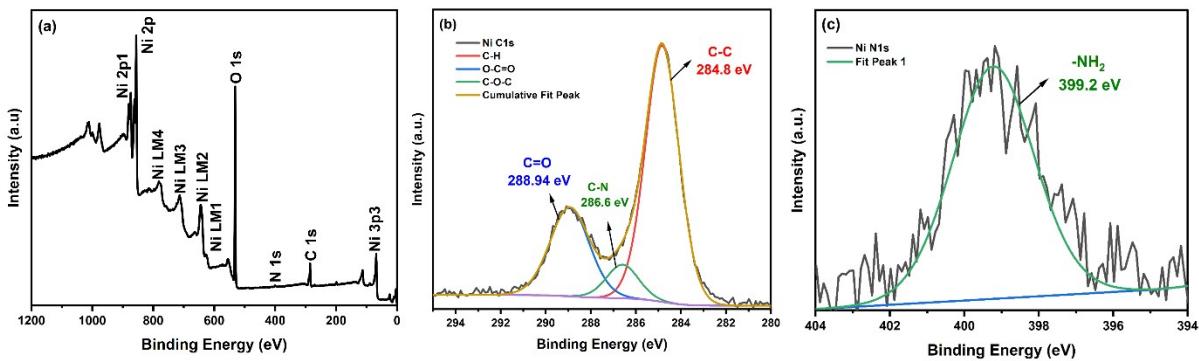
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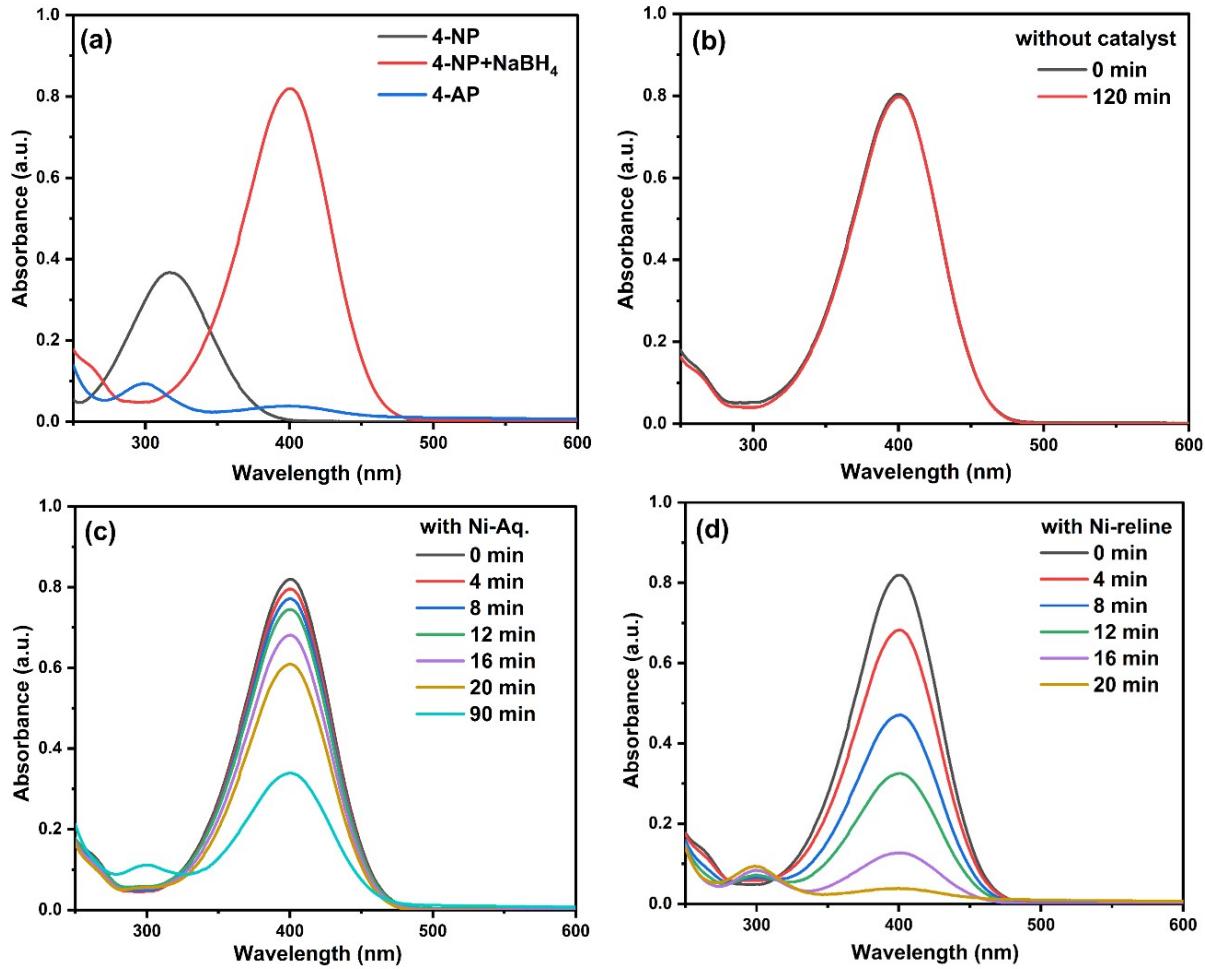
20 Fig. S1. Average particle size histogram (a) Ni-aq. and (b) Ni-reline

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23 Fig. S2. Survey XPS spectrum of Ni-reline, (b) C 1s XPS spectrum and (c) N 1s XPS spectrum.

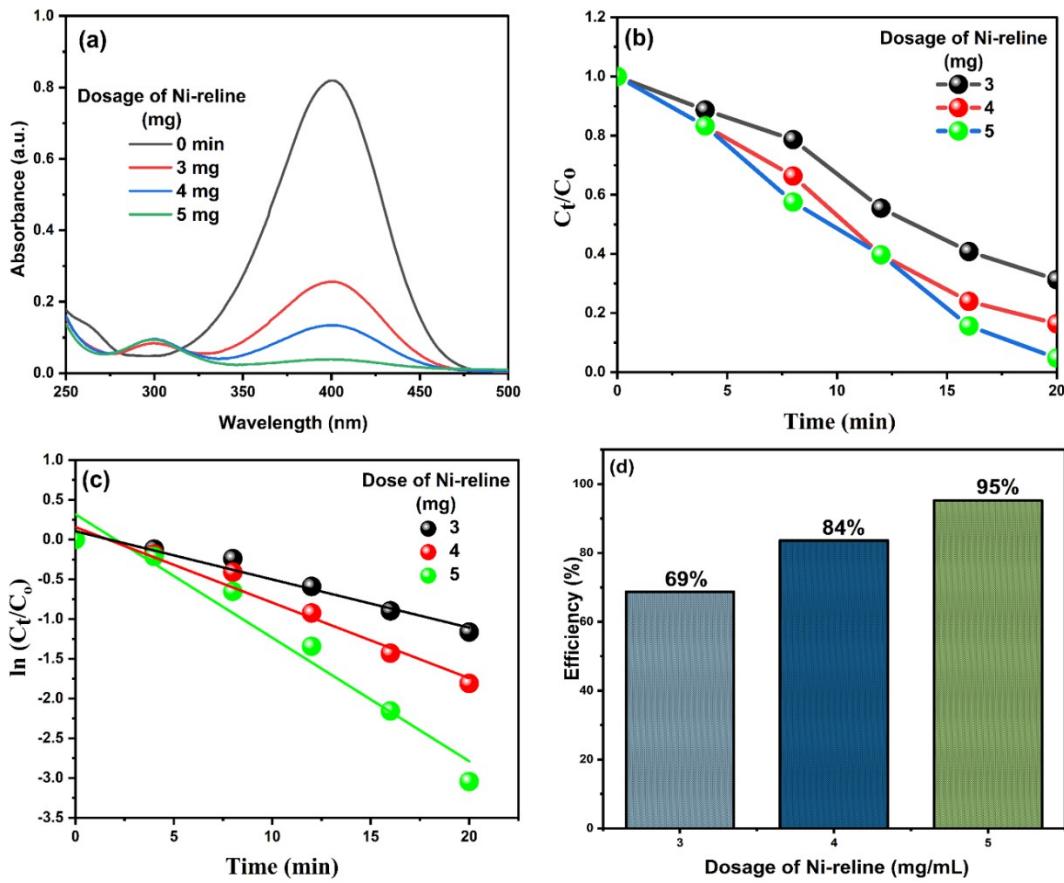
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26 Fig. S3. UV-Vis absorption spectra of (a) 4-NP, 4-NP+NaBH₄, 4-AP (b) hydrogenation of 4-NP
 27 without catalyst (c-d) catalytic hydrogenation of 4-NP with Ni-Aq. and Ni-reline.

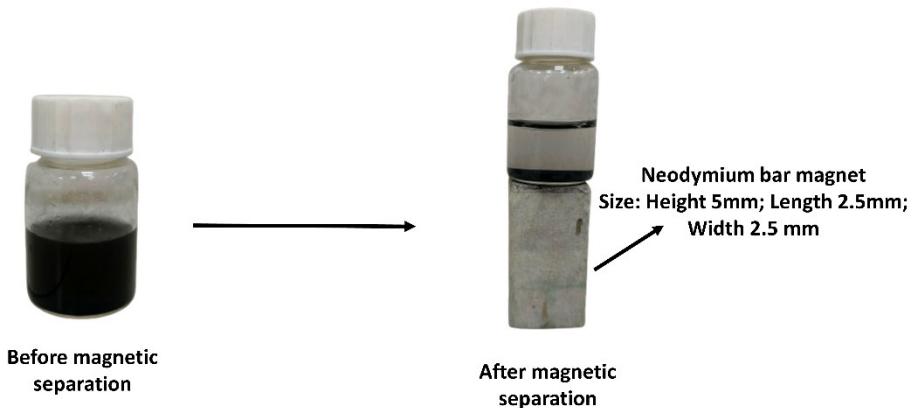
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30 Fig. S4. Comparative analysis of (a) Time-dependent UV-vis spectral changes of the catalytic
 31 hydrogenation of 4-NP to 4-AP using NaBH_4 at different amounts of Ni-reline (b) C_t/C_0 vs time
 32 (c) Efficiency after 20 minutes.

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35 Fig. S5. Magnetic separation of $\text{Ni}/\text{Ni(OH)}_2$ nanocomposite.

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37 Table S1. Chemical composition in Atomic % of the Ni-reline derived from XPS analysis

| Sample | Atomic % of each element | | | |
|-----------|--------------------------|-------|-------|------|
| | Ni | C | O | N |
| Ni-reline | 23.56 | 20.44 | 53.98 | 2.02 |

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39 Table S2. A summary of the correlation coefficient of $\ln(C_t/C_0)$ vs. time plot, rate constant, and
40 activity parameter for the reduction of 4-NP of the synthesized nanocatalysts

| Catalyst | <i>k</i> (min ⁻¹) | R ² | Efficiency (%) |
|-----------|-------------------------------|----------------|----------------|
| Ni-Aq. | 0.0121 | 0.94 | 26 |
| Ni-reline | 0.1336 | 0.97 | 95 |

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42 Table S3. Summary of the correlation coefficient of $\ln(C_t/C_0)$ vs. time plot, rate constant, and
43 activity parameter for reducing 4-NP of different dose of Ni-reline

| Dose of catalyst (mg) | <i>k</i> (min ⁻¹) | R ² | Efficiency (%) |
|-----------------------|-------------------------------|----------------|----------------|
| 3 | 0.0607 | 0.96 | 69 |
| 4 | 0.0901 | 0.97 | 84 |
| 5 | 0.1559 | 0.96 | 95 |

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45 Table S4. Summary of the catalytic performance of Ni-reline at various pH within fixed time
46 duration

| Time | Efficiency (%) | | | | |
|------|----------------|------|------|------|-------|
| | pH 3 | pH 5 | pH 7 | pH 9 | pH 11 |
| 0 | 0 | 0 | 0 | 0 | 0 |
| 4 | 57.9 | 42.5 | 20.9 | 16.5 | 1.3 |
| 8 | 84.9 | 76.1 | 42.5 | 21.4 | 1.7 |

| | | | | | |
|-----------|------|------|------|------|-----|
| 12 | 96.7 | 90 | 62.5 | 27.8 | 2.2 |
| 16 | 98 | 96.9 | 84.4 | 35.3 | 3.7 |
| 20 | 98.2 | 97.8 | 95.2 | 41.9 | 4.0 |

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