## **Electronic Supplementary Information**

## In-Situ Synthesis of Cotton-Derived Ni/C Catalysts with Controllable Structure and Enhanced Catalytic Performance

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**Table S1.** Elemental compositions of CF and CCF

Sample	C (%)	H (%)	O (%)
CF	42.2	6.2	51.6
CCF	81.5	3.5	15



Figure S1. The digital photographs during the synthesis process of Ni/C catalysts.



Figure S2. (a) Typical SEM and (b) magnified SEM images of 1-Ni/C.



Figure S3. (a) Typical SEM and (b) magnified SEM images of 2-Ni/C.



**Figure S4.** (a) Typical SEM image; (b) magnified SEM image; (c) TEM image and (d) size distributions of Ni particles of 4-Ni/C.



Figure S5. XRD patterns of 3-Ni/C catalysts calcined at different temperature.

Table S2. Catalytic performance of various Ni/C catalysts for the selective hydrogenation of o-CNB

Catalyst	Conversion (%)	Selectivity (%)	TOF (h <sup>-1</sup> ) <sup>a</sup>
CCF	11.2	95.1	_
1-Ni/C	21.4	91.4	12.6
2-Ni/C	65.6	84.0	5.1
3-Ni/C	100	86.6	6.3
4-Ni/C	91.7	91.7	4.9

Reaction conditions: 0.50 g *o*-CNB, 0.05 g catalyst, 50 mL ethanol, 140 °C, 0.5 MPa H<sub>2</sub>, time 5 h. <sup>a</sup> Turnover frequency (TOF) =  $(M_{CNB} \times \text{Con} \times \text{Sec}) / (M_{Ni} \times t)$ ;  $M_{CNB}$ : the total amount of CNB (mol); Con: conversion of CNB (%); Sec: selectivity of CAN (%);  $M_{Ni}$ : the Ni amount in catalysts (mol); t: the reaction time (h).

Calcination Temperature (°C)	Conversion (%)	Selectivity (%)	TOF (h <sup>-1</sup> ) <sup>a</sup>
200	8.5	96.5	0.6
300	99.8	78.3	5.7
400	100	86.6	6.3
500	63.6	88.3	4.1
600	25.1	92.7	1.7

**Table S3.** Catalytic performance of 3-Ni/C with different calcination temperatures for the selective hydrogenation of *o*-CNB

Reaction conditions: 0.50 g *o*-CNB, 0.05 g catalyst, 50 mL ethanol, 140 °C, 0.5 MPa H<sub>2</sub>, time 5 h. <sup>a</sup> Turnover frequency (TOF) =  $(M_{CNB} \times \text{Con} \times \text{Sec}) / (M_{Ni} \times t)$ ;  $M_{CNB}$ : the total amount of CNB (mol); Con: conversion of CNB (%); Sec: selectivity of CAN (%);  $M_{Ni}$ : the Ni amount in catalysts (mol); t: the reaction time (h).



**Figure S6.** The digital photographs of reaction solutions before and after the hydrogenation of *o*-CNB.



**Figure S7.** Cycling performance of 3-Ni/C for the selective hydrogenation of *o*-CNB. Reaction conditions: 0.50 g *o*-CNB, 0.05 g 3-Ni/C, 50 mL ethanol, 140 °C, 1.0 MPa H<sub>2</sub>, time 3 h. Inset shows the separation of the 3-Ni/C with a magnet after the reaction.

Cycle Number	Conversion (%)	Selectivity (%)
1	100	93.9
2	99.6	90.3
3	93.6	90.4
4	74.7	90.9
5	75.7	91.0
6	71.5	90.7

Table S4. Cycling performance of 3-Ni/C for the selective hydrogenation of o-CNB

Reaction conditions: 0.50 g o-CNB, 0.05 g catalyst, 50 mL ethanol, 140 °C, 1 MPa H<sub>2</sub>, time 3 h.



Figure S8. TEM image of the 3-Ni/C after 6 cycles.