

## Supporting Information

### Effect of reduction temperature on structure and catalytic performance of mesoporous Ni-Fe-Al<sub>2</sub>O<sub>3</sub> in oxidative dehydrogenation of ethane

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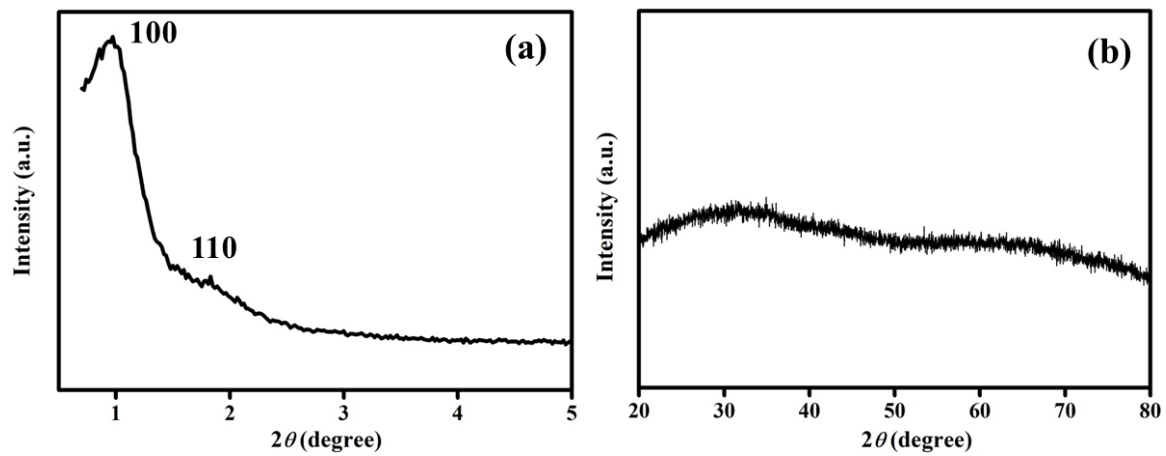
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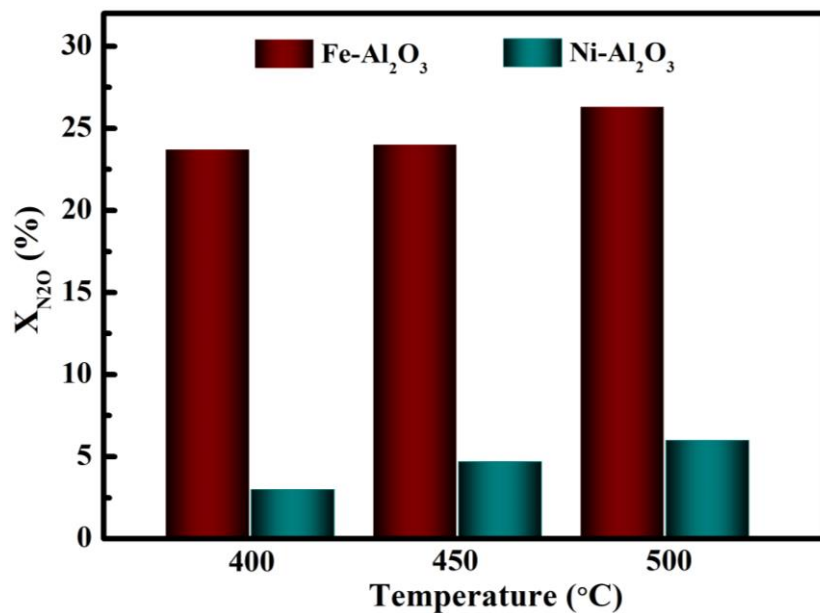
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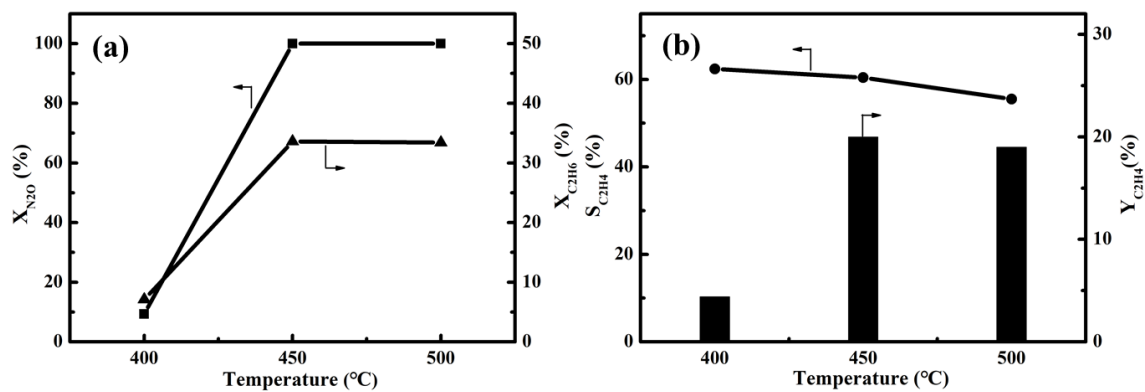
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**Figure S1.** (a) Small- and (b) wide-angle XRD patterns of fresh Ni-Fe-Al<sub>2</sub>O<sub>3</sub> catalyst.



**Figure S2.** N<sub>2</sub>O splitting as a function of reaction temperature over mesoporous Ni-Al<sub>2</sub>O<sub>3</sub> with 10% molar fraction of Ni, and Fe-Al<sub>2</sub>O<sub>3</sub> with 10% molar fraction of Fe. Reaction conditions: Feed gas flow rate = 25 mL min<sup>-1</sup>, N<sub>2</sub>O concentration = 10 vol%, He as balance gas.



**Figure S3.** Catalytic performance of fresh Ni-Fe-Al<sub>2</sub>O<sub>3</sub> for ODH. Reaction conditions: Feed gas flow rate = 25 mL min<sup>-1</sup>, C<sub>2</sub>H<sub>6</sub> concentration = 10 vol%, N<sub>2</sub>O concentration = 10 vol%, He as balance gas.

**Table S1** Percentage of Ni and Fe species determined by XPS method

Sample	Ni 2p <sub>3/2</sub>			Fe 2p <sub>3/2</sub>	
	Sat	Ni <sup>2+</sup>	Ni <sup>0</sup>	Fe <sup>3+</sup>	Fe <sup>2+</sup>
Ni-Fe-Al <sub>2</sub> O <sub>3</sub>	49.4	50.6	0	80.3	19.7
Ni-Fe-Al <sub>2</sub> O <sub>3</sub> -H400	41.4	58.6	0	55.6	44.4
Ni-Fe-Al <sub>2</sub> O <sub>3</sub> -H600	35.7	53.5	10.8	46.8	53.2

“Sat” represents satellite peak.

**Table S2.** Catalytic behaviors of Ni-Fe-Al<sub>2</sub>O<sub>3</sub> catalysts with different Fe doping amount for ODH.

Catalyst	X <sub>N<sub>2</sub>O</sub> (%)	X <sub>C<sub>2</sub>H<sub>6</sub></sub> (%)	S <sub>C<sub>2</sub>H<sub>4</sub></sub> (%)	Y <sub>C<sub>2</sub>H<sub>4</sub></sub> (%)
10Ni-Al <sub>2</sub> O <sub>3</sub>	9.5 (8.2)	7.2 (6.4)	88.9 (89.9)	6.4 (5.7)
10Ni-0.5Fe-Al <sub>2</sub> O <sub>3</sub>	51.2 (78.0)	22.8 (27.6)	74.7 (67.9)	17.0 (18.8)
10Ni-1Fe-Al <sub>2</sub> O <sub>3</sub>	99.1 (95.7)	35.8 (30.6)	59.3 (63.2)	21.2 (19.3)
10Ni-3Fe-Al <sub>2</sub> O <sub>3</sub>	100 (100)	33.5 (31.8)	57.6 (60.4)	19.3 (19.2)

The values in parentheses are the catalytic results after reaction for 3 h.

**Table S3.** Representative results reported in literatures toward low temperature ( $\leq 500$  °C) ODH reaction.

Catalyst	Synthesis	Metal ratio	Feed gas	$Y_{C_2H_4}$	Ref.
Ni-Fe- Al <sub>2</sub> O <sub>3</sub>	EISA	Ni/Fe= 10/3	10% C <sub>2</sub> H <sub>6</sub> + 10% N <sub>2</sub> O+He	18% (400°C)	This work
Ni-Ce/ Al <sub>2</sub> O <sub>3</sub>	Impregnation	Ce/Ni= 0.25	6% C <sub>2</sub> H <sub>6</sub> +6% O <sub>2</sub> +He	7.7% (400°C)	1
Ni-Ce/ Al <sub>2</sub> O <sub>3</sub>	Impregnation	Ni/Ce= 11/3	6% C <sub>2</sub> H <sub>6</sub> +6% O <sub>2</sub> + He	20% (500°C)	2
Ni-Ce/ Al <sub>2</sub> O <sub>3</sub>	Impregnation	Ce/Ni= 0.13	6% C <sub>2</sub> H <sub>6</sub> +6% O <sub>2</sub> +He	23.5% (450°C)	3
Cordierite monoliths coated with Ni-Ce/ $\gamma$ -Al <sub>2</sub> O <sub>3</sub>	washcoating	Ni/Ce= 19/8	6% C <sub>2</sub> H <sub>6</sub> +6% O <sub>2</sub> +He	17% (450°C)	4
Ni-Al mixed oxide	Co-precipitation	80 wt% NiO	C <sub>2</sub> H <sub>6</sub> /N <sub>2</sub> O/He= 1/1/48	18% (420°C) 24% (460°C)	5
NiO/ Al <sub>2</sub> O <sub>3</sub>	Impregnation	–	10% C <sub>2</sub> H <sub>6</sub> +10% O <sub>2</sub> +N <sub>2</sub>	38.6% (450°C)	6
Ni-Fe/ $\gamma$ -Al <sub>2</sub> O <sub>3</sub>	Coprecipitation -deposition	Ni/(Ni+Fe)=0.9	0.5% C <sub>2</sub> H <sub>6</sub> + 0.5% O <sub>2</sub> +N <sub>2</sub>	40.1% (400°C)	7
Fe/Al <sub>2</sub> O <sub>3</sub>	Vapor deposition	3.6 wt% Fe	11.6% C <sub>2</sub> H <sub>6</sub> +5.2% O <sub>2</sub> +75.2% He+ 8% N <sub>2</sub>	2.7% (400°C)	8
NiO-CeO <sub>2</sub>	Evaporation	Ni/Ce= 12	9.1% C <sub>2</sub> H <sub>6</sub> + 3% O <sub>2</sub> +87.9% He	6.1% (275°C)	9
NiO/TiO <sub>2</sub>	Evaporation	50 wt% NiO	C <sub>2</sub> H <sub>6</sub> /O <sub>2</sub> /He= 3/3/24	41% (450°C)	10
Ni-Nb-O/CeZrO	Evaporation	Nb/Ni= 0.176	C <sub>2</sub> H <sub>6</sub> /O <sub>2</sub> =2/1	27.6% (450°C)	11

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