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Electronic Supplementary Information

Carbon coated Ni/Al_2O_3 as high-efficiency catalysts for hydrogenation of furfural to furfuryl alcohol

Meng Ge,[a,b] Xin Ling, [a,b] Yanyan Liu,[a,b*] Weiwei Lin [a,b*]

[a]Anhui Laboratory of Functional Coordinated Complexes for Materials Chemistry and Application, Anhui Polytechnic University, Wuhu, 241000, China.
E-mail: linweiwei@ahpu.edu.cn.
[b]School of chemical and environmental Engineering, Anhui Polytechnic University, Wuhu, 241000, China. E-mail: liuyanyan@ahpu.edu.cn.

Chemicals

$Ni(NO_3)_2·6H_2O$ (AR), $Al(NO_3)_3)·9H_2O$ (AR), ethanol (AR, 97.0%), glucos (AR), furfural (AR, 99.0%), furfuryl alcohol (AR, 98%), tetrahydrofurfuryl alcohol (AR, 98%), 2-methylfuran (AR, 98%), n-butanol (AR, 99.5%), isopropanol (AR), methylbenzene (AR), n-hexane (AR) and dioxane (AR) were purchased from Sinopharm Group Chemical Reagent Co. Ltd. They were all used directly.

Catalysts characterization

The crystal structure of the samples was analyzed by SHIMADZU LabXRD-6100 X-ray diffractometer with a Cu-K_α source (λ = 0.154 nm). The test was conducted with a tube voltage of 40 kV and a tube current of 30 mA, with a scanning range set from 5° to 70° and a scanning rate of 4°/min. The carbon content of the catalysts was analyzed by thermogravimetric analysis (TGA) using a NETZSCH TG 209 F3 thermogravimetric analyzer. The test was performed under an air atmosphere with a temperature range of 40-700 °C and a heating rat of 10 °C per minute. The microscopic morphology of the catalyst was observed by a Hitachi FESEM SU8600 field emission scanning electron microscope. Transmission electron microscopy (TEM) was carried out using a FEI Tecnai F20 instrument at an acceleration voltage of 200 kV. For he TEM sample preparation, the catalyst was ultrasonically dispersed in ethanol for 10 min and the resulting suspension was drop-cast onto a carbon-film-coated copper grid. High-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) was conducted on the Thermal fisher Spectra 300 with an acceleration voltage of 300 kV. X-ray photoelectron spectroscopy (XPS) analysis was conducted using a Thermo Fisher Escalab 250Xi spectrometer with an Al-Kα X-ray source. The working voltage and current were 14.8 kV and 4.5 mA, respectively. Ar^+ ion etching was carried out before the test and the C 1s peak (284.8 eV) of adventitious carbon was used as the reference. The specific surface area, pore volume and average pore diameter were determined by Brunauer-Emmett-Teller (BET) adsorption

measurements on a Micromeritics TriStar II 3020 analyzer. ICP-OES measurements were performed on an Agilent 720ES spectrometer with a radio frequency (RF) power of 1200 W and a nebulizer flow rate of 0.75 L/min.

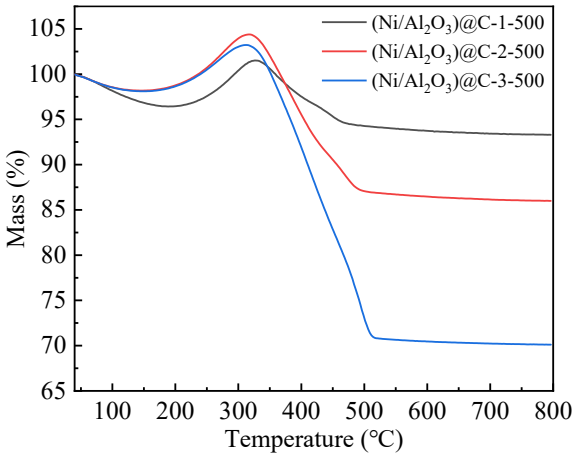


Fig. S1 TGA curve of the carbon coated Ni/Al_2O_3 catalysts.

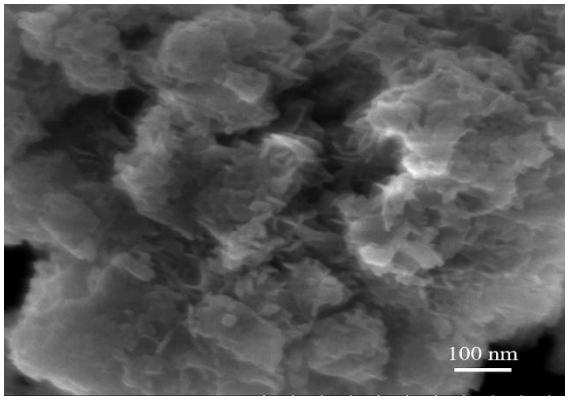


Fig. S2 SEM image of the Ni_2Al-LDH

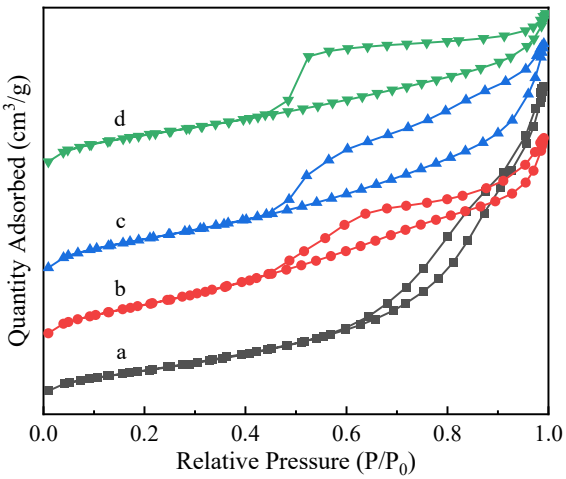


Fig. S3 N_2 adsorption-desorption isotherms of Ni/Al_2O_3 (a), (Ni/Al_2O_3)@C-1-500 (b), (Ni/Al_2O_3)@C-500 (c), (Ni/Al_2O_3)@C-500 (d).

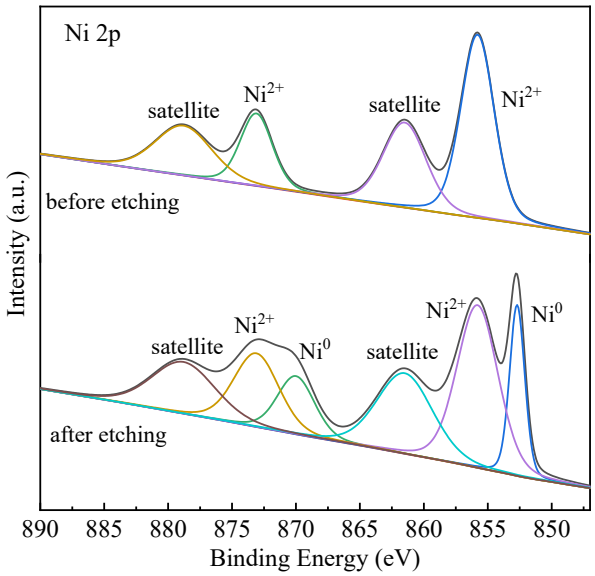


Fig. S4 XPS spectrum of Ni 2p in Ni/Al_2O_3.

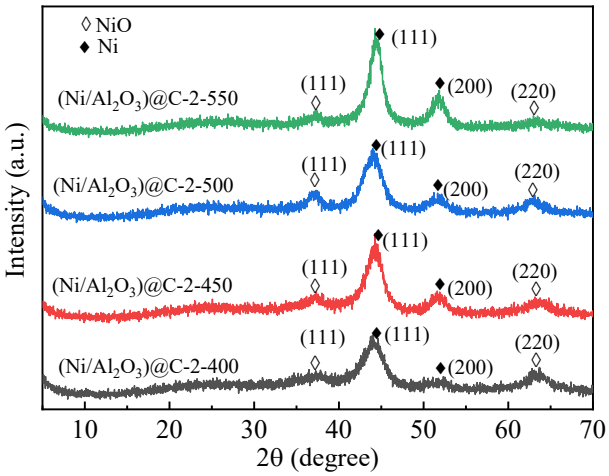


Fig. S5 XRD patterns of (Ni/Al_2O_3)@C-2 at different calcination temperatures.

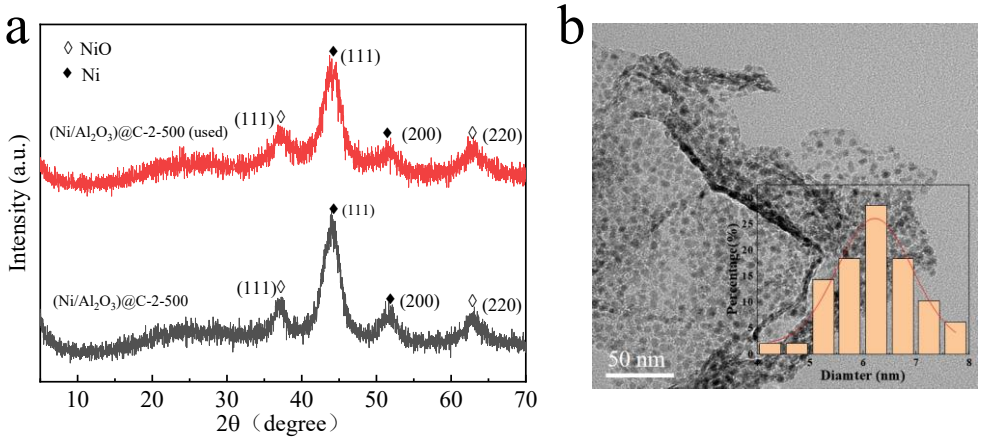
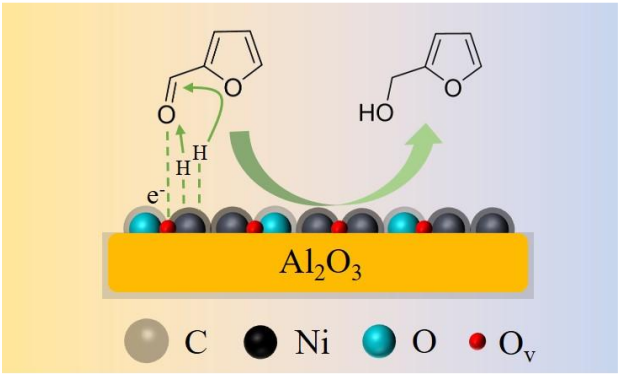


Fig. S6 (a) XRD patterns of catalyst (Ni/Al_2O_3)@C-2-500 before and after usage; (b) TEM image and particle size distributions of (Ni/Al_2O_3)@C-2-500 after usage.



Scheme S1 Proposed reaction mechanism of FF to FA over the (Ni/Al_2O_3)@C-2-500 catalyst.

Table. S1 Relative amount of Ni species as quantified by XPS spectra of Ni $2p_{3/2}$ in different catalysts.

Entry	Catalysts	Relative amount of Ni species (%)	
		Ni^0	Ni^{2+}
1	Ni/Al_2O_3	0	100
2	(Ni/Al_2O_3)@C-1-500	25.6	74.4
3	(Ni/Al_2O_3)@C-2-500	27.1	72.9
4	(Ni/Al_2O_3)@C-3-500	50.5	49.5

Table. S2 Proportions of O species as quantified by XPS spectra of O 1s in different catalysts.

Entry	Catalysts	Relative amount of O species (%)		
		O_L	O_V	O_{ads}
1	Ni/Al_2O_3	51.1	24.8	24.1
2	(Ni/Al_2O_3)@C-1-500	29.2	33.9	36.9
3	(Ni/Al_2O_3)@C-2-500	20.0	38.0	42.0
4	(Ni/Al_2O_3)@C-3-500	3.2	26.0	70.8

Table. S3 Comparison of the catalytic hydrogenation of FF to FA among (Ni/Al_2O_3)@C-2-500 and other Ni-based catalysts.

Catalysts	$m_{FF}/m_{Cat.}$	t/h	T/°C	Solvent	P/MPa	Con./%	Sel./%	Ref.
Ni/Al_2O_3	1.0	1	100	Isopropanol	2	98.5	31.3	[1]
Ni/TiO_2	2.0	3	130	Dioxane	3	68.7	52.4	[2]
5%Ni/AC	4.0	3	130	Isopropanol	2	21.5	71.5	[3]
Ni/C-500	5.8	0.5	80	Ethanol	1	91.5	58.0	[4]
Ni/MgO	5.8	2	200	Isopropanol	2	97.7	94.7	[5]
$NiFe_2O_4$	3.3	4	180	Isopropanol	0	95.0	90.0	[6]
(Ni/Al_2O_3)@C-2-500	10.0	0.5	120	Water	1	96.4	89.4	This work

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