Eco-Friendly Preparation of Ultrathin Biomass-Derived Ni_3S_2-Doped Carbon Nanosheets for Selective Hydrogenolysis of Lignin Model Compounds in the Absence of Hydrogen

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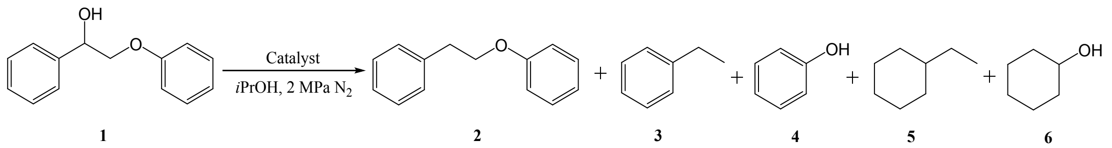
Table S1 ICP result of the fresh and used catalyst after five runs.

	C (%)	Ni (%)	S (%)
Ni_3S_2-CSs-0.2-700	90.8	6.5	2.7
Ni_3S_2-CSs-0.4-700	81.3	13.2	5.5
Ni_3S_2-CSs-0.6-700	71.7	19.2	9.1
Ni_3S_2-CSs-0.8-700	62.9	27.1	10.0
Used Ni_3S_2-CSs-0.4-700	91.5	6.2	2.3

Table S2 cleavage of β-O-4 lignin model compounds in different solvents.



Entry	Cat.	Solvent	Temp. (°C)/ T.(h)	Conv. (%)[b]	Yield(%)[b]				
					2	**3**	**4**	**5**	**6**
1	Ni_3S_2-CSs-0.4-700	ethylene glycol	260/4	42	21	19	17	0	0
2	Ni_3S_2-CSs-0.4-700	formic acid	260/4	30	18	11	9	0	0

[a] Reaction conditions: **1** (100mg), Cat. (20mg), *i*PrOH (10 mL), 2.0 MPa N_2, 260 ℃, 4 h; [b] Conversion and yields were determined by GC/MS with n-dodecane as the internal standard;

Table S3 Molecular weight of original and residual lignin under different reaction condition.

Entry	T/℃	Time/h	M_w (g/mol)	M_n (g/mol)	PDI (M_w/M_n)
1	Alkaline lignin		2092	1306	1.602
2	260	4	1840	1156	1.591
3	280	4	1765	1102	1.575
4	280	8	1610	1035	1.555

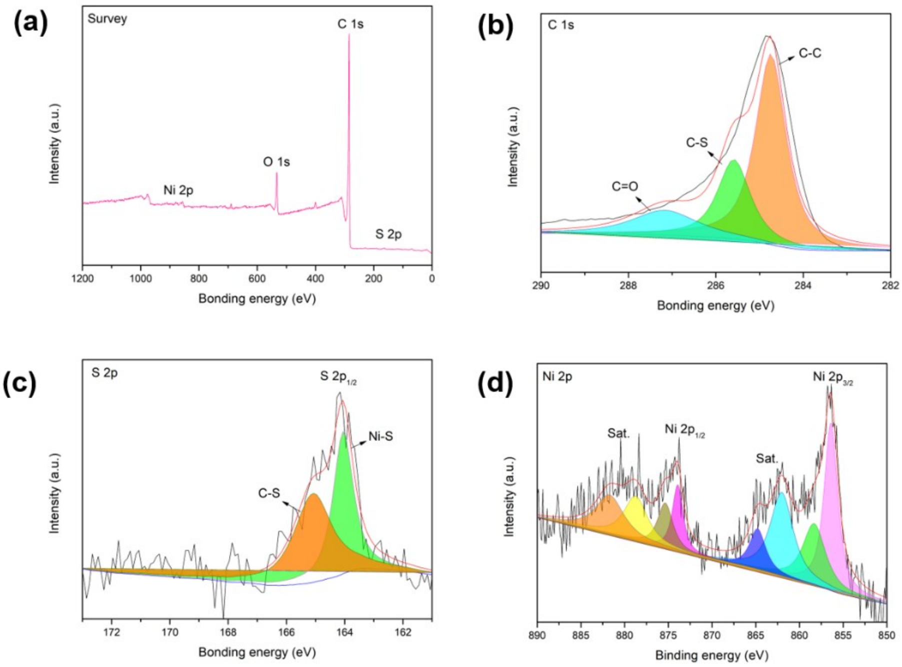


Fig. S1 XPS spectra of used Ni_3S_2-CSs-0.4-700 catalyst.

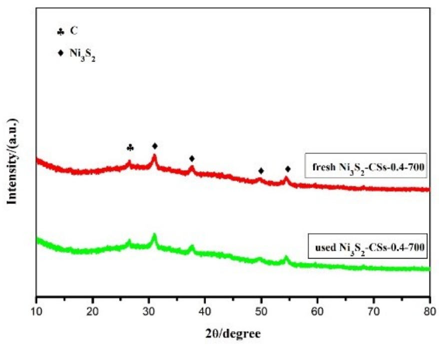


Fig. S2 XRD pattern of fresh and used Ni_3S_2-CSs-0.4-700 catalyst.

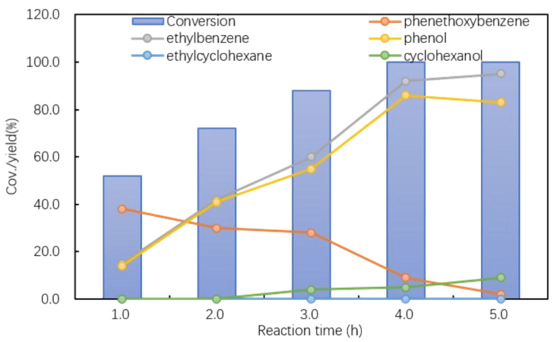
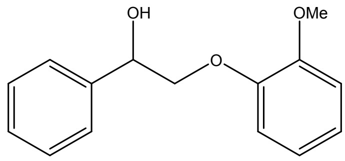
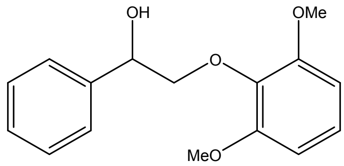


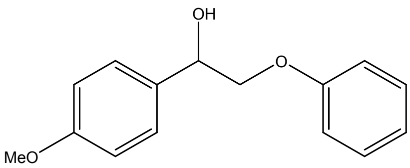
Fig. S3 The time course of the product distributions for the conversion of β-O-4 model compound over Ni_3S_2-CSs-0.4-700.



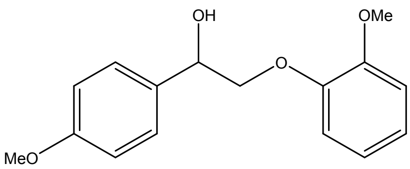
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.27 (m, 5H), 6.98 – 6.85 (m, 4H), 5.10 (dd, *J* = 9.6, 2.8 Hz, 1H), 4.15 (dd, *J* = 10.0, 2.8 Hz, 1H), 3.98 (t, *J* = 9.7 Hz, 1H), 3.86 (s, 1H), 3.83 (s, 3H).



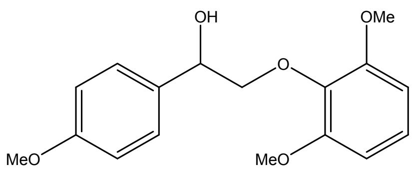
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55 (d, *J* = 6.8 Hz, 2H), 7.37– 7.29 (m, 3H), 6.97 (t, *J* = 7.5 Hz, 1H), 6.73 (d, *J* = 7.5 Hz, 2H), 5.16 – 5.12 (m, 1H), 4.58 (dd, *J* = 12.5, 7.0 Hz, 1H), 4.22 (dd, *J* = 12.5, 7.0 Hz, 1H), 3.80 (s, 6H), 3.23 (d, *J* = 4.9 Hz, 1H).



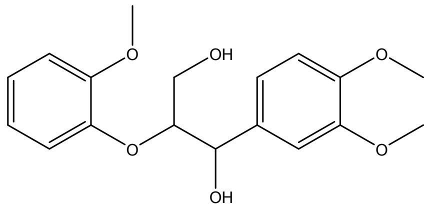
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (d, *J* = 8.4 Hz, 2H), 7.25 (dd, *J* = 8.7, 7.2 Hz, 2H), 6.94 (t, *J* = 7.2 Hz, 1H), 6.90 – 6.88 (m, 4H), 5.02 (dd, *J* = 8.3, 3.6 Hz, 1H), 4.00 (qd, *J* = 9.6, 6.1 Hz, 2H), 3.77 (s, 3H), 3.27 (s, 1H).



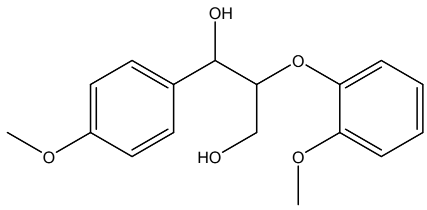
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.5 Hz, 2H), 7.00 – 6.96 (m, 1H), 6.93 – 6.88 (m, 5H), 5.07 (d, *J* = 9.2 Hz, 1H), 4.13 (dd, *J* = 10.0, 2.9 Hz, 1H), 3.98 (t, *J* = 9.7 Hz, 1H), 3.86 (s, 3H), 3.81 (s, 3H), 2.24 (s, 1H).



^1H NMR (400 MHz, $CDCl_3$) δ 7.34 (d, *J* = 6.7 Hz, 2H), 6.97 (t, *J* = 7.4 Hz, 1H), 6.88 (d, *J* = 7.5 Hz, 2H), 6.76 (d, *J* = 7.5 Hz, 2H), 5.12 (dt, *J* = 6.9, 6.0 Hz, 1H), 4.59 (dd, *J* = 12.4, 7.1 Hz, 1H), 4.22 (dd, *J* = 12.4, 7.1 Hz, 1H), 3.80 (s, 9H), 3.23 (d, *J* = 4.9 Hz, 1H).



^1H NMR (400 MHz, $CDCl_3$) δ 7.04 (td, *J* = 8.1, 1.9 Hz, 1H), 6.98 – 6.87 (m, 5H), 6.82 (d, *J* = 8.3 Hz, 1H), 4.97 (t, *J* = 4.1 Hz, 1H), 4.15 (dd, *J* = 8.8, 5.3 Hz, 1H), 3.94 – 3.88 (m, 1H), 3.86 (s, 9H), 3.69 – 3.65 (m, 2H), 2.90 (dd, *J* = 7.2, 5.7 Hz, 1H).



^1H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.7 Hz, 1H), 7.31 (d, *J* = 8.7 Hz, 1H), 7.14 – 7.03 (m, 2H), 6.97 – 6.87 (m, 4H), 4.99 (d, *J* = 7.7 Hz, 1H), 4.15 (dt, *J* = 7.8, 3.4 Hz, 0.5H), 4.05 – 4.01 (m, 0.5H), 3.89 (d, *J* = 12.6 Hz, 3H), 3.80 (s, 3H), 3.67 – 3.54 (m, 2H), 3.45 (ddd, *J* = 12.3, 8.0, 3.9 Hz, 1H), 2.87 – 2.76 (m, 1H).