

Recent progress in the direct synthesis of γ -valerolactone from biomass-derived sugars catalyzed by RANEY® Ni–Sn alloy supported on aluminium hydroxide

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Electronic Supplementary Information

Recent progress in the direct synthesis of γ -valerolactone from biomass-derived sugars catalyzed by Raney Ni–Sn alloy supported on aluminium hydroxide

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1. Experimental sections

1.1. Materials

Raney Ni–Al was purchased from Kanto Chemical Co. Inc. (50%wt Ni+ 50%wt Al = 50:50). Tin dihydrate ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$, 99%), SnO (99%), cellobiose (98%), sucrose (98%), glucose (98%), maltol (99%), and sorbitol (98%) were purchased from Wako Pure Chemical and used as received. fructose (97%) was purchased from Kanto Chemical Co. Inc. and used as received. Levulinic acid (96%) and γ -valerolactone (98%) were purchased from Tokyo Chemical Industry (TCI) and used as received.

1.2. Catalyst preparation

Raney®Ni Typical procedure for the synthesis of the conventional Raney®Ni is described as follows:¹ Raney Ni–Al alloy powder (1.0 g) was slowly added to an aqueous solution of NaOH (6 M, 10 mL) at 363 K under gentle stirring and was kept on stirring for 30 min for further alkali leaching. The precipitated was washed by distilled water until the supernatant became neutral and then stored under water for activity test.

Synthesis of RNi/AlOH. Typical procedure of the synthesis of Raney nickel supported on aluminium hydroxide catalyst (denoted as Raney Ni/AlOH or RNi/AlOH) is described as follows:² Raney Ni–Al alloy powder (1.0 g) was slowly added to a dilute aqueous solution of NaOH (0.31 M, 8 mL) at room temperature. The temperature was raised to 363 K and 1 mL of 3.1 M NaOH solution was subsequently added and stirred for 30 min. The mixture was placed into a sealed-Teflon autoclave reactor for hydrothermal treatment at 423 K for 2 h. The resulting precipitate was filtered, washed with distilled water until filtrate was neutralized, and then stored in water. The catalyst was dried under vacuum before the catalytic reaction.

Synthesis of Raney Ni-Sn(x)/AlOH. Typical procedure of the synthesis of Raney nickel-tin alloy supported on aluminium hydroxide (denoted as RNi–Sn(0.45)/AlOH; 0.45 is Sn loading amount in mmol) is described as the follows:^{3,4} RNi/AlOH that was obtained from the above procedure was mixed with an ethanol solution that contained 0.45 mmol $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ at room temperature and stirred for 2 h. The mixture was then placed into a sealed-Teflon autoclave reactor for the hydrothermal treatment at 423 K for 2 h. The resulting precipitate was filtered, washed with distilled water until the filtrate was neutralized, and then stored in water. The RNi–Sn(x)/AlOH was reduced with H_2 at 673–873 K for 1.5 h in order to investigate the effect of temperature reduction on the formation of the Ni–Sn alloy, which produce RNi–Sn(x)/AA, where AA is amorphous alumina. The catalyst was dried under vacuum before the catalytic reaction.

1.3. Catalyst characterization

The prepared catalysts were characterized by powder X-ray diffraction on a Mac Science M18XHF instrument using monochromatic $\text{CuK}\alpha$ radiation ($\lambda = 0.15418$ nm). It was operated at 40 kV and 200 mA with a step width of 0.02° and a scan speed of 2°min^{-1} . The mean crystallite size of Ni was calculated from the full width at half maximum (FWHM) of the Ni(111) diffraction peak according to the Scherrer's equation and confirmed by H_2 chemisorption. The bulk compositions of the catalysts were determined by inductively coupled plasma-atomic emission spectroscopy (ICP-AES), using a SPS1700 HVR of SII instrument.

Nickel surface area was determined by H_2 chemisorption. After the catalyst was heated at 393 K under vacuum for 30 min, it was then heated at 673 K under H_2 for 30 min and under vacuo for 30 min, followed by evacuation to room temperature for 30 min. The adsorption of H_2 was conducted at 273 K. The active surface area was calculated from the volume of H_2 desorbed by assuming an H/Ni stoichiometry of 1 and a surface area of $6.77 \times 10^{-20} \text{ m}^2$ per atom Ni based on an equal distribution of the three lowest index planes of nickel (fcc) according to a method described in the literature.^{5,6}

Nitrogen adsorption isotherms at 77 K were measured using a Belsep Max (BEL Japan). The samples were degassed at 473 K for 2 h to remove physisorbed gases prior to the measurement. The amount of nitrogen adsorbed onto the samples was used to calculate the specific surface area by means of the BET equation. The total pore volume was estimated to be the liquid volume of nitrogen at a relative pressure of about 0.995. The Barrett–Joyner–Halenda (BJH) approach was used to calculate total pore volume and pore size distribution from desorption data.⁷

Temperature-programmed reduction (TPR) was carried out on Micrometrics AutoChem II Chemisorption Analyzer Instrument according to procedure previously reported.⁸ The catalyst samples were outgassed at 423 K for 1 h and then cooled to 313 K under Ar flow of 70 ml/min. TPR profiles were registered while heating the samples from 313 K to 1073 K by 283 K/min heating rate under 50 ml/min flow of 10% H_2 -Ar mixture.

SEM images of the synthesized catalysts were recorded on a JEOL JSM-610SEM after the samples were coated using JEOL JTC-1600autofine coater. TEM image analyses were recorded on a Hitachi High-Tech H-7650 with an emissive gun, operated at 150 kV. Raman spectra were collected on a JASCO NRS-2100 laser-Raman Spectrophotometer with an Ar beam lamp at excitations of 488 nm and 514.5 nm. Thermal gravimetric analyses (TGA) were performed on a Rigaku

Thermal Analysis system (Thermo plus Evo TG 8120 apparatus) under N₂ gas flow (250 mL min⁻¹) using Pt pans in the range room temperature to 1073 K (5 K min⁻¹).

The ammonia-temperature programmed-desorption (NH₃-TPD) was carried out on a Belsorp Max (BEL Japan). The samples were degassed at elevated temperature of 373-473 K for 2 h to remove physisorbed gases prior to the measurement. The temperature was then kept at 473 K for 2 h while flushed with He gas. NH₃ gas (balanced NH₃, 80% and He, 20%) was introduced at 373 K for 30 min, then evacuated by helium gas to remove the physisorbed also for 30 min. Finally, temperature programmed desorption was carried out at temperature of 273-1073 K and the desorbed NH₃ was monitored by TCD.

1.4. Catalytic reactions

1.4.1 Typical reaction procedure for direct conversion of sucrose.

A typical reaction of sucrose was carried out in the following manner. The catalyst (0.06 g), sucrose solution (mmol sucrose/Ni metal = 85; ~0.35 mmol of sucrose in 5 ml H₂O) were placed in an autoclave reactor system of Taiatsu Techno Japan (a Pyrex tube was fitted inside of a sus316 jacket to protect the vessel from corrosion in acidic media). The reactor was flushed by pressurizing with H₂ 0.25 MPa for 10 times. After H₂ was introduced into the reactor at an initial pressure of H₂ was 3.0 MPa at room temperature, then the temperature of the reactor was raised to 443 K for a reaction time of 3 h. A xylitol (0.02 mmol) as an internal standard was added after the reaction. The used RNi-Sn(1.04)/AlOH catalyst was easily separated using either simple centrifugation or filtration, then finally dried overnight under vacuum at room temperature prior to reusability testing.

1.4.2. Hydrogenation of LA or EL

Catalyst (0.05 g), LA (2.0 mmol), H₂O (3 ml) as a solvent, and 1,8-octandiol (0.2 mmol) as an internal standard were placed into a glass reaction tube, fitted inside a stainless steel reactor. The reactor was flushed by pressurizing with H₂ 0.25 MPa for 10 times. After H₂ was introduced into the reactor with an initial H₂ pressure of 4.0 MPa at room temperature, the temperature of the reactor was raised to 443 K. After 120 min, the conversion of LA and the yield of GVL were determined via GC analysis. For reusability test, the used RNi-Sn(1.04)/AlOH catalyst was easily separated using either simple centrifugation or filtration in air, and then it was utilized repeatedly without any additional treatments.

1.5. Analytical methods and sample preparation

1.5.1. One-pot conversion of sugars.

The reactant and products in the reactor were analyzed by using a JASCO RI-930 HPLC using an internal standard method. Xylitol was used as an internal standard to calibrate the liquid product concentrations and carbon balances. The calibration factors are given in the Table S3. All solution samples were filtered through a 0.22 mm PVDF syringe filter prior to injection in the HPLC. Analyses of glucose, fructose, mannitol, gamma-valerolactone, levulinic acid were performed by using a Shodex SH1821 column (H₂O eluent), refractive index (RI) intelligent detector, pressure of 17 kg/m², column temperature of 343 K and a flow rate of 0.6 ml/min. Analyses of sorbitol, sucrose and cellobiose were performed by using an NH₂ column-Inertsil (Acetonitril-H₂O eluent) using an internal standard method, refractive index (RI) intelligent detector, pressure of 45-50 kg/cm², column temperature of 313 K and a flow rate of 1.0 mL/min. The concentrations of each compound in the product mixture were determined using the calibration curves obtained from the injection of solutions of authentic samples with known concentrations. The conversions and yields of products were expressed as mole percent, on the basis of the total sugars amount.

1.5.2. Hydrogenation of LA or EL

Analysis of GVL for the hydrogenation of LA and EL in H₂O was performed on a Shimadzu GC-8A equipped with a flame ionisation detector and with Silicone OV-101 packing. 1,8-octandiol was used as an internal standard to calibrate the liquid product concentrations and carbon balances. The calibration factors are given in the Table S3. The assignments of products were determined by GC-MS. Gas chromatography-mass spectrometry (GC-MS) was performed on a Shimadzu GC-17B equipped with a thermal conductivity detector and with an RT-βDEXsm capillary column. ¹H and ¹³C NMR spectra were obtained on a JNM-AL400 spectrometer at 400 MHz; samples were dissolved in chloroform-*d*₁ or D₂O with TMS as an internal standard. Products were identified by GC-MS, ¹H, and ¹³C NMR spectra. The conversions and yields of products were expressed as mole percent, on the basis of the total LA or EL amount.

The sugar conversion and the product yield were evaluated on a carbon basis,⁹ as shown below:
Sugars conversion (%):

$$X = \left(1 - \frac{\text{sucrose concentration in product}}{\text{sucrose concentration in the loaded sample}}\right) \times 100\%$$

Product yield (%):

$$Y = \frac{\text{moles of carbon product}}{\text{moles of carbon in sucrose introduced}} \times 100\%$$

2. Results of catalyst characterisation

Table S1. The bulk composition of the catalysts, H₂ uptake, average Ni(111) crystallites sizes, specific surface area (S_{BET}), and the amount of acid sites.⁴

Entry	Catalyst ^a	Composition ^a (mmol. g ⁻¹)			H ₂ uptake ^b (μmol. g ⁻¹)	D_{Ni} ^c (nm)	S_{BET} ^d (m ² . g ⁻¹)	Amount of acid site ^e (mmol. g ⁻¹)
		Ni	Al	Sn				
1	Raney®Ni	3.98	0.63	-	121	8.6	66	195 ¹⁰
2	Raney Ni/AlOH	3.46	3.80	-	104	8.8	151	474
3	RNi-Sn(0.26)/AlOH	3.82	3.66	0.26	103	7.0	90	477
4	RNi-Sn(0.45)/AlOH	3.58	3.44	0.45	101	6.2	77	487
5	RNi-Sn(1.04)/AlOH	3.13	4.91	1.04	110	5.3	145	491
6	RNi-Sn(2.14)/AlOH	3.01	3.48	2.14	103	4.8	76	489
7	RNi-Sn(3.96)/AlOH	3.54	3.71	3.96	98	4.6	72	490
8	γ-Al ₂ O ₃	-	-	-	-	-	100	180 ¹¹

^aValue in the parenthesis are Sn loading amount, the bulk compositions are determined by ICP-AES. ^bBased upon total the H₂ uptake at 273 K (after corrections for physical and chemical adsorption). ^cAverage Ni(111) or Ni-Sn crystallite sizes were calculated from the full width at the half maximum (FWHM) of the Ni(111) diffraction peak according to the Scherrer's equation. ^dDetermined by N₂ adsorption at 77 K. ^eAmount of acidic sites (μmol. g⁻¹) was derived from the NH₃-TPD spectra.

Table S2. Physico-chemical properties of synthesized RNi-Sn(x)/AA catalysts (x = 3.0, 1.4, and 1.0) after reduction with H₂ at different temperature of 673–873 K for 1.5 h.

Entry	Catalysts ^a	S_{BET} ^b (m ² .g ⁻¹)	V_p ^b (cm ³ .g ⁻¹)	d_{pore} ^b (nm)	H ₂ uptake ^c (μmol.g ⁻¹)	S_{Ni} ^c (m ² .g ⁻¹ .cat)	D^d/nm
1	RNi-Sn(1.04)/AA 673 K	42	0.079	2.8	81	35.2	nd
2	RNi-Sn(1.04)/AA 773 K	72	0.065	3.6	67	31.8	nd
3	RNi-Sn(1.04)/AA 873 K	122	0.098	3.2	53	22.7	nd
4	RNi-Sn(2.14)/AA 673 K	50	0.065	3.2	45	13.6	14.7
5	RNi-Sn(2.14)/AA 773 K	27	0.063	9.3	32	9.4	16.6
6	RNi-Sn(2.14)/AA 873 K	26	0.073	11.2	17	5.1	28.4
7	RNi-Sn(3.96)/AA 673 K	26	0.040	4.9	66	32.1	nd
8	RNi-Sn(3.96)/AA 773 K	61	0.086	5.7	59	24.4	nd
9	RNi-Sn(3.96)/AA 873 K	126	0.079	5.6	56	21.3	nd
10 ^e	Ni ₃ Sn/γ-Al ₂ O ₃ 673 K	120	0.469	15.6	9.0	nd	11.4
11 ^e	Ni ₃ Sn ₂ /γ-Al ₂ O ₃ 673 K	79	0.503	25.5	8.2	nd	15.0

^aThe value in the parenthesis is Ni/Sn molar ratio. ^bComposition was determined by using ICP-AES. ^cDetermined by N₂ adsorption at 77 K. ^dBased on total H₂ uptake at 273 K (noted after corrected for physical and chemical adsorption). ^eAverage crystallite sizes of Ni₃Sn₂(101) alloy phases at 2θ = 30.37°, calculated by using Scherer equation. ^fThe catalysts were synthesised from nickel(II)chloride hexahydrate and tin (II) chloride dihydrate supported on commercial γ-Al₂O₃ with similar procedure to the bulk Ni-Sn(1.5)¹² then treated by H₂ at 673 K and the XRD patterns of the catalysts are shown in Fig. 9. nd = not determined.

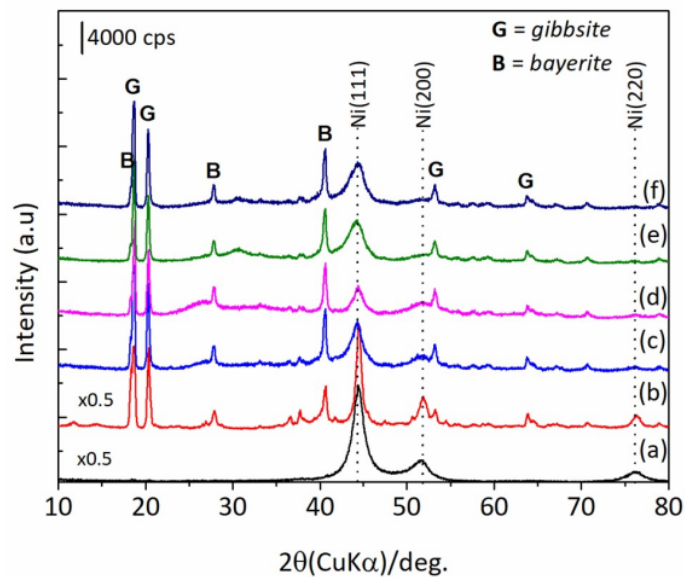


Fig. S1 XRD patterns of synthesized (a) Raney®Ni, (b) RNi/AlOH, and RNi-Sn(x)/AlOH with different Sn loading amount of (c) 0.45 mmol g⁻¹, (d) 1.04 mmol g⁻¹, (e) 2.14 mmol g⁻¹, and (f) 3.96 mmol g⁻¹.

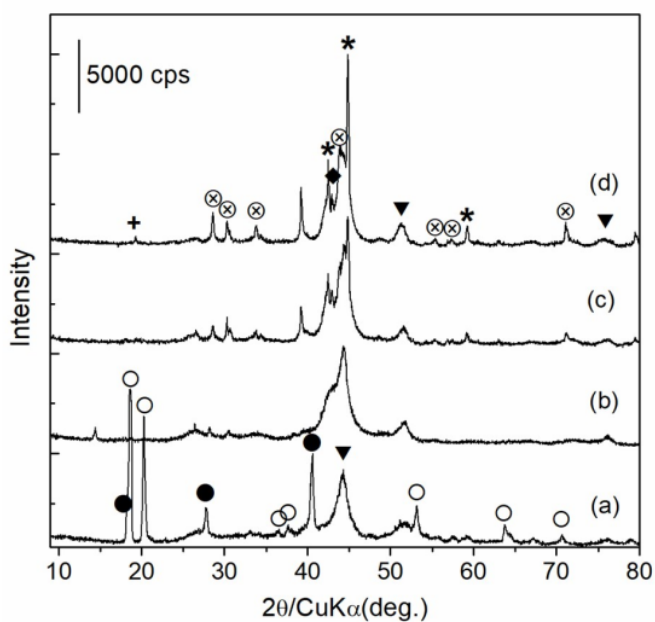


Fig. S2 XRD patterns of (a) as-prepared Ni-Sn(0.45)/AlOH catalysts and after reduction with H₂ at (b) 673 K, (c) 773 K, and

(d) 873 K for 1.5 h. (\triangleright) Ni_3Sn . (\oplus) Ni_3Sn_2 . (\odot) $\text{Ni}(0)$. (+) $\beta\text{-Sn}$.^{13,14}

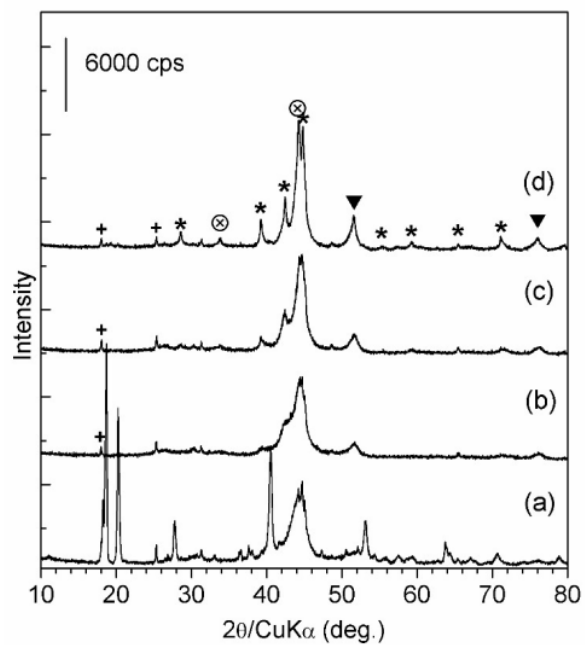


Fig. S3 XRD patterns of (a) *as-prepared* Ni-Sn(3.96)/AlOH catalysts and after reduction with H_2 at (b) 673 K, (c) 773 K, and (d) 873 K for 1.5 h.^{13,14}

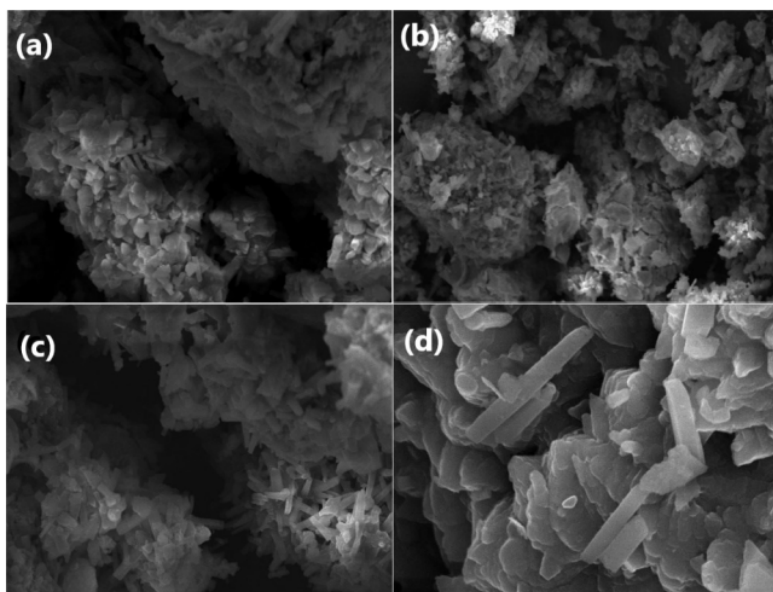


Fig. S4 Representative SEM images of the as-prepared (a) RNi-Sn(0.45)/AlOH, (b) RNi-Sn(1.04)/AlOH, (c) RNi-Sn(2.14)/AlOH, and (d) RNi-Sn(3.96)/AlOH catalysts.

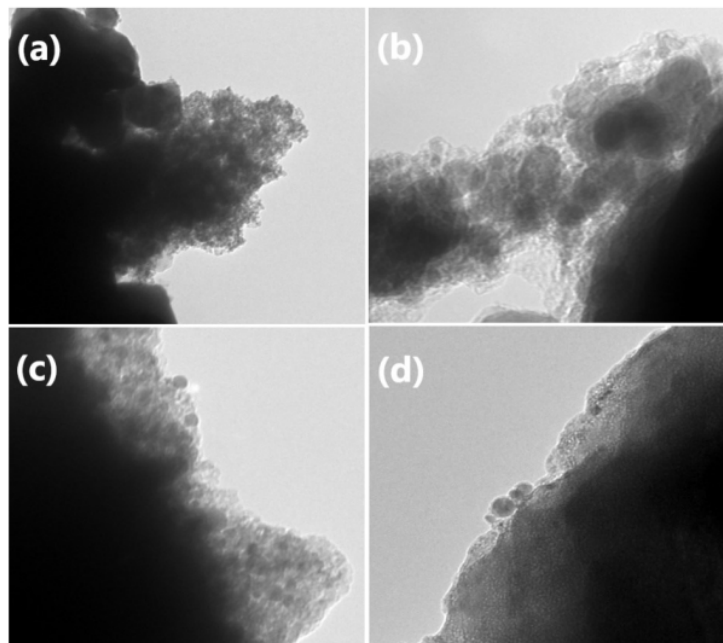


Fig. S5 Representative TEM images of the as-prepared (a) RNi-Sn(0.45)/AlOH, (b) RNi-Sn(1.04)/AlOH, (c) RNi-Sn(2.14)/AlOH, and (d) RNi-Sn(3.96)/AlOH catalysts

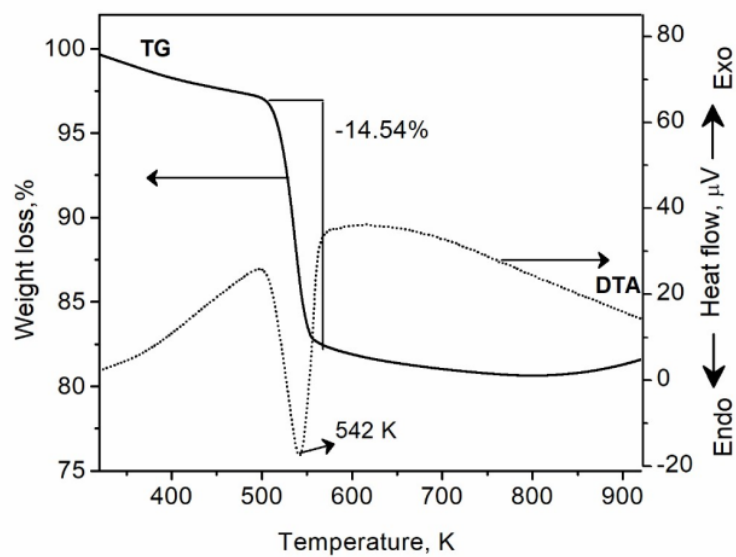


Fig. S6 Representative TG-DTA data the as-prepared RNi-Sn(2.14)/AlOH catalyst.

3. Results of catalytic reaction

Table S3. Calibration factors for HPLC and GC analyses of products

Entry	Name of product	Calibration factor
1	Glucose	0.982 (HPLC)
2	Fructose	0.888 (HPLC)
3	Mannitol	0.871 (HPLC)
4	Sorbitol	0.867 (HPLC)
5	Levulinic acid	2.787 (GC)
6	GVL	1.581 (GC)

Table S4. Results of catalytic one-pot conversion of sucrose over various RNi–Sn(x)/AA catalysts

Entry	Catalyst ^a	Conversion ^b (%)	Yield ^b (%)				
			C6-sugars ^c	C6-sugar alcohols ^d	LA	GVL	Others ^e
1	RNi–Sn(0.45)/AA 673 K	>99	0.0	81.0	0.0	10.1	8.9
2	RNi–Sn(0.45)/AA 773 K	>99	0.0	87.3	0.0	9.2	3.5
3	RNi–Sn(0.45)/AA 873 K	>99	0.0	87.8	0.0	9.0	3.2
4	RNi–Sn(3.96)/AA 673 K	>99	9.2	30.3	0.0	42.7	16.8
5	RNi–Sn(3.96)/AA 773 K	>99	0.0	30.8	0.0	52.3	16.9
6	RNi–Sn(3.96)/AA 873 K	>99	0.0	27.3	0.0	55.5	17.2
7 ^f	Ni ₃ Sn/ γ -Al ₂ O ₃ 673 K	>99	0.0	26.4	0.0	63.9	9.7
8 ^f	Ni ₃ Sn ₂ / γ -Al ₂ O ₃ 673 K	>99	0.0	17.5	0.0	67.1	15.4
9 ^f	Ni ₃ Sn ₂ /AC 673 K	>99	0.0	19.2	0.0	59.8	21.0

Reaction conditions: substrate/Ni=85; sucrose (0.35 mmol); solvent H₂O (5 ml); H₂ (3.0 MPa); temperature (443 K); reaction time (12 h). ^aThe value in the parentheses is Sn loading amount. ^bConversion and yield were determined by HPLC using an internal standard technique. ^cGlucose and fructose are included as C6-sugars. ^dSorbitol and mannitol are included as C6-sugar alcohols. ^eOthers include glucoside and furans (5-hydroxymethyl furfural and furfural). ^fThe Ni₃Sn/ γ -Al₂O₃, Ni₃Sn₂/ γ -Al₂O₃, and Ni₃Sn₂/AC catalysts were synthesized via the hydrothermal treatment of a mixture of NiCl₂·4H₂O and SnCl₂·2H₂O solution at 423 K for 24 h, followed by reduction with H₂ at 673 K for 1.5 h.¹²

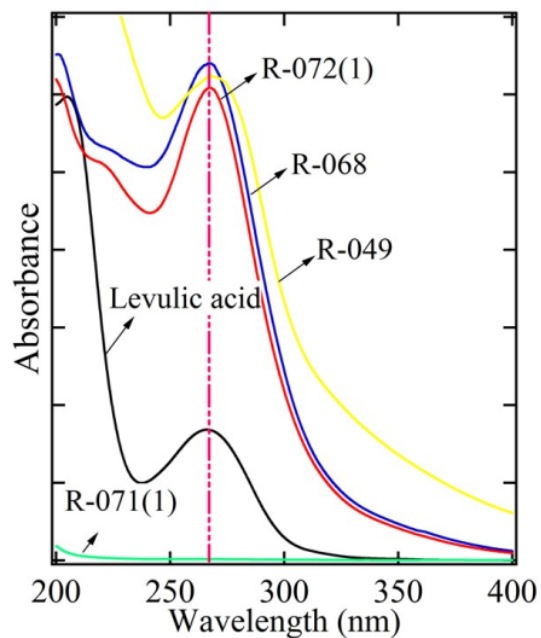


Fig. S7 UV-Vis spectra of reaction mixture from direct conversion of sucrose (R-071(1) over RNi/AlOH catalyst. R-072(1), R-049, and R-068 samples were reaction mixture of direct conversion of sucrose, glucose, and fructose, respectively over RNi-Sn(1.04)/AlOH catalysts.

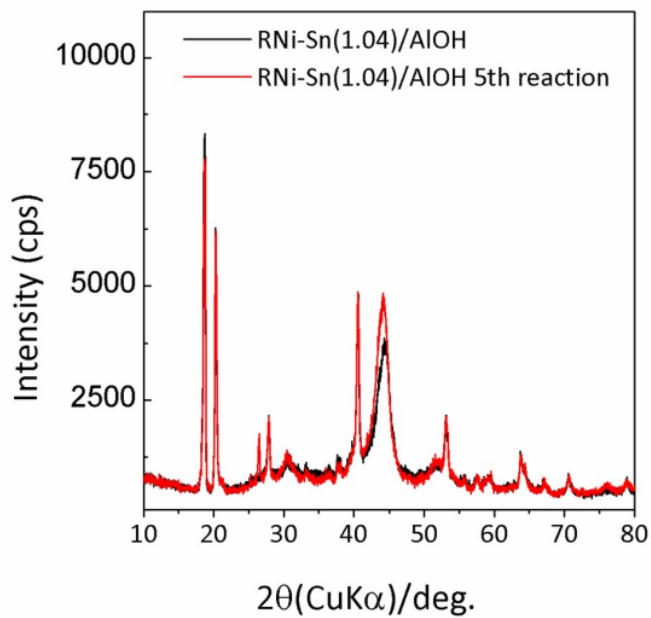


Fig. S8 XRD patterns of fresh and recovered RNi-Sn(1.04)/AlOH catalysts.^{13,14}

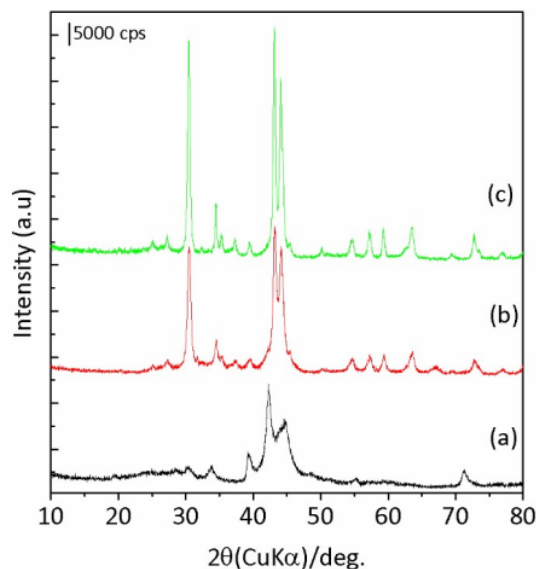


Fig. S9 XRD patterns of (a) $\text{Ni}_3\text{Sn}/\gamma\text{-Al}_2\text{O}_3$, (b) $\text{Ni}_3\text{Sn}_2/\gamma\text{-Al}_2\text{O}_3$, and (c) $\text{Ni}_3\text{Sn}_2/\text{AC}$ after reduction with H_2 at 673 K for 1.5 h.¹³⁻

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Recent progress in the direct synthesis of γ -valerolactone from biomass-derived sugars catalyzed by RANEY® Ni–Sn alloy supported on aluminium hydroxide

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