

Supporting Information

Enhancing Continuous-Flow Reaction via Compression-Molding of Solid Catalysts and Dilutants in Packed-Bed Systems

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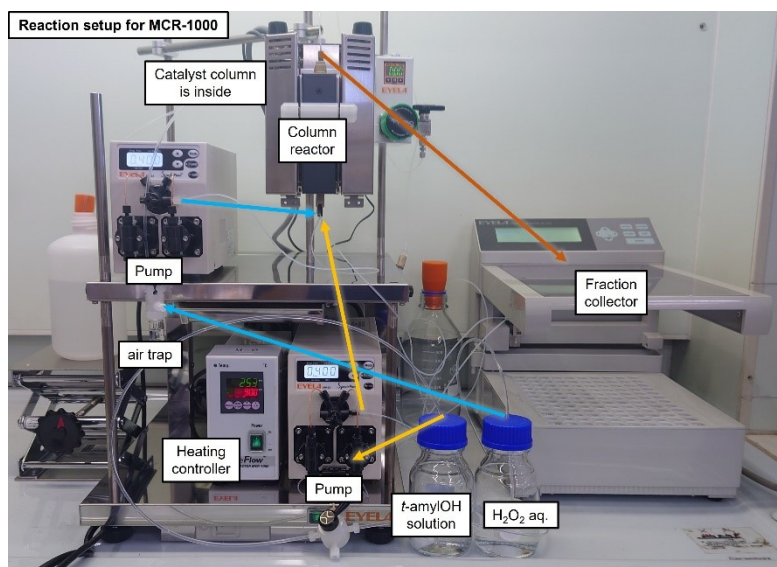
10. Procedure for the scale-up experiment for the flow reaction

1. General

Gas chromatography was measured on Shimadzu GC-2014 spectrometer with N₂ gas as a carrier, using Shimadzu SH-Stabilwax-DA (Length: 30 m, I.D.: 0.250 mm, Film: 0.25 μm). A dual plunger pump (UI-22 series) was purchased from EYELA, and flow reactor (MCR-1000, HCR-1000), Teflon filter, SUS laminated metal filter, and fraction collector (DC-1000) were purchased from EYELA. ICP-AES was measured by HORIBA ULTIMA2. Particle size distribution was measured by MICROTRACBELL MT3000II. SEM was measured by JEOL JSM-IT800. XPS was measured by Shimadzu KRATOS ULTRA2. Press machine (CDM-5PA) was purchased from RIKEN KIKI CO., Ltd. Molds (mold for heat press machine φ14 and LD-3025) were purchased from AS ONE CORPORATION and Labonect CO., Ltd. H₂O₂ and Wakogel C-200 were purchased from FUJIFILM Wako pure chemicals. *t*-AmylOH and benzyl alcohol were purchased from Tokyo Chemical Industry Co., Ltd. SiO₂-60-S was purchased from KANTO CHEMICAL CO., INC. Pt black powder was purchased from N.E. Chemcat.

2. Experimental setup for the flow reaction

- Reaction setup for MCR-1000 (column size ID: 10 mm, L: 100 mm)

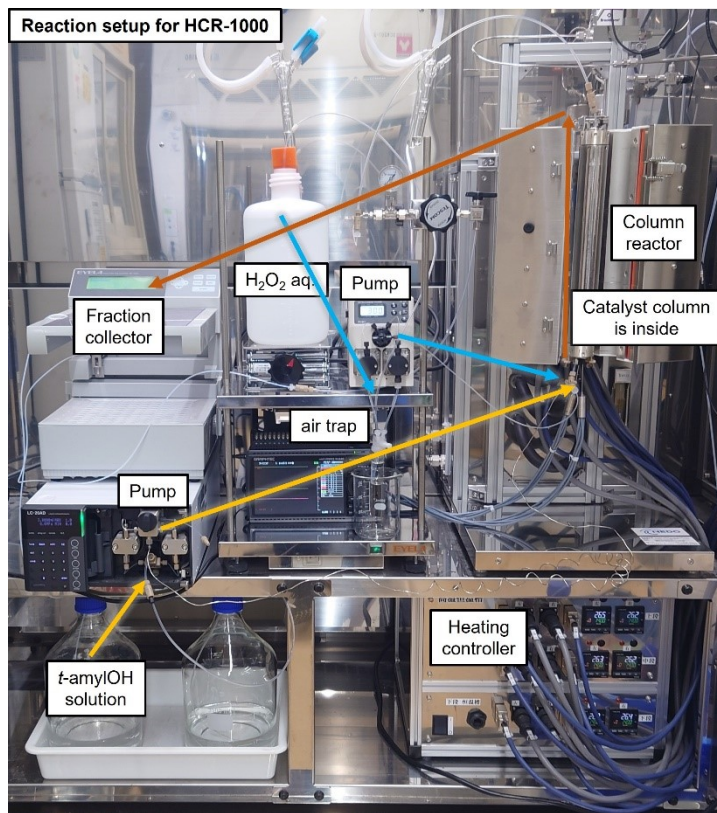


Pump: UI-22 110P, EYELA

Flow reactor (heating controller and column reactor): MCR-1000, EYELA

Fraction collector: DC-1500, EYELA

- Reaction setup for HCR-1000 (column size ID: 37 mm, L: 300 mm)



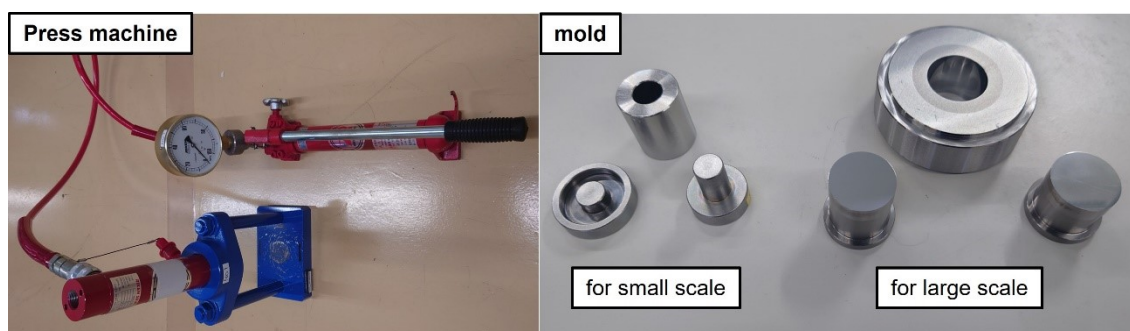
Pump for H_2O_2 aq.: UI-22 110P, EYELA

Pump for *t*-amylOH solution: LC-20AD, Shimadzu

Flow reactor (heating controller and column reactor): HCR-1000, EYELA

Fraction collector: DC-1500, EYELA

- Press machine and molds



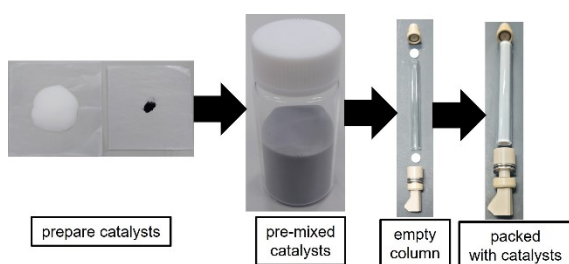
Press machine: RIKEN KIKI CO., Ltd, CDM-5PA

Mold for small scale: AS ONE CORPORATION, mold for heat press machine $\phi 14$

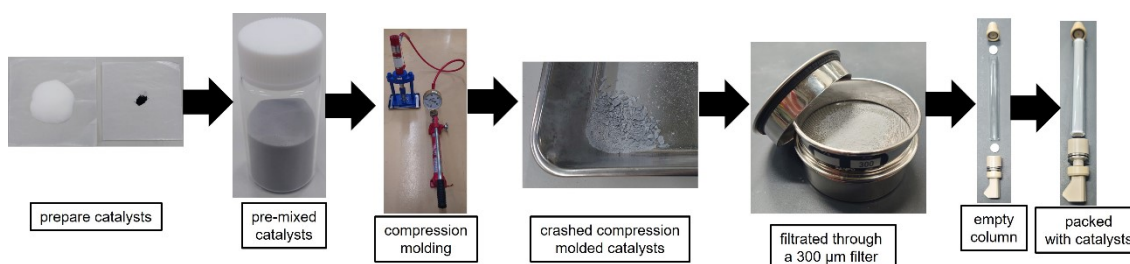
Mold for large scale: Labonect, LD-3025, $\phi 30 \times 25$ mm

3. Preparation of the reaction column for MCR-1000 and HCR-1000

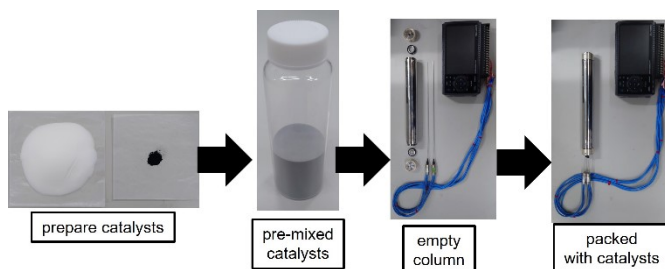
- Preparation of the reaction column for MCR-1000 without the compression molding



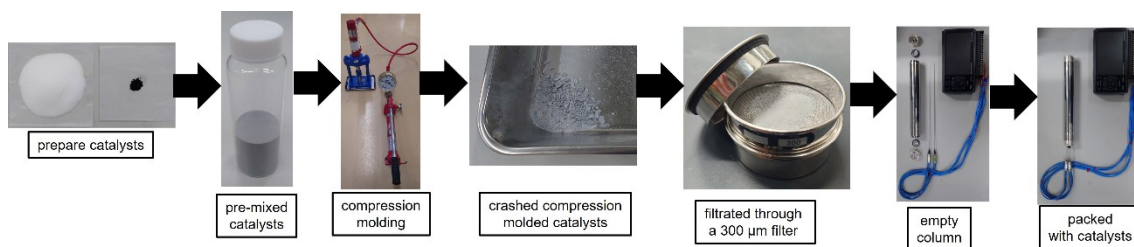
- Preparation of the reaction column for MCR-1000 with the compression molding



- Preparation of the reaction column for HCR-1000 without the compression molding

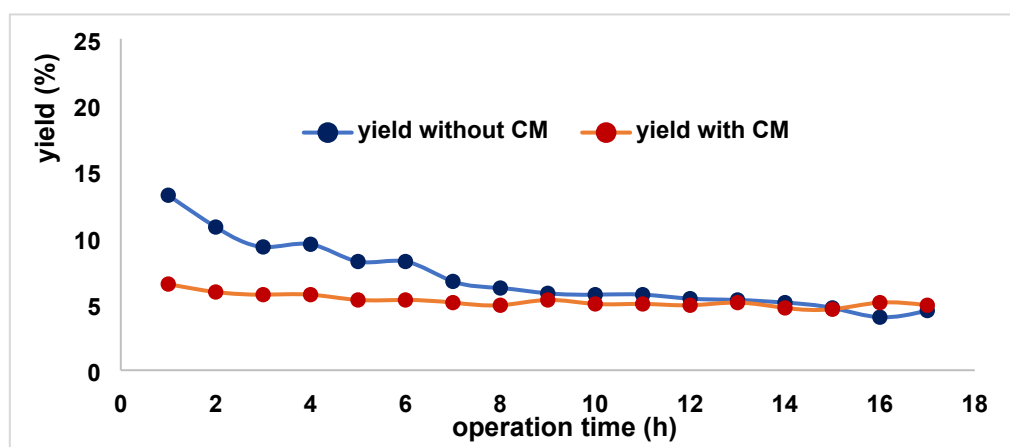


- Preparation of the reaction column for HCR-1000 with the compression molding



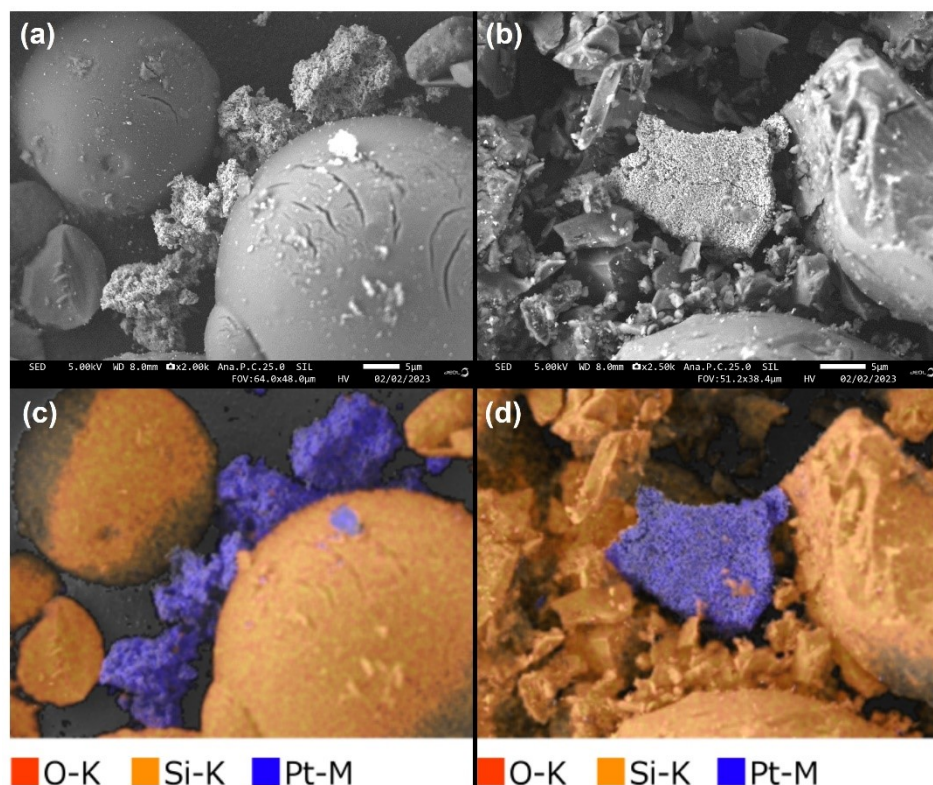
4. Procedure for EDX analysis of Pt in the column before/after the flow reaction (Table 1)

Pt black powder was pre-mixed with silica-gel and then packed into a column (ID: 10 mm, L: 100 mm). In the cases of entries 5 and 6, the compression molding was conducted for the pre-mixed catalysts using a press machine (RIKEN KIKI CO., Ltd, CDM-5PA) and mold (AS ONE CORPORATION, mold for heat press machine $\phi 14$) before packing to the reaction column. The catalyst column was set onto the reactor (EYELA, MCR-1000), and H₂O was flowed at each 0.4 mL/min (pump: EYELA, UI-22 110P) for 1 h. The catalyst column was heated at 90 °C (heating controller and column reactor: MCR-1000). After stabilization the reaction was started by changing into feed solution of 2wt% H₂O₂ aq. and benzyl alcohol (**1**, 0.05 M) solution in *t*-amylOH. After 24 hours, feed solutions were changed into blank solvent, and reactor was cooled to room temperature. The catalysts were taken out from the reaction column and dried up in vacuo. The EDX of a part of dried catalysts was measured, and the amount of Pt in the reaction column was calculated.



Yields of benzaldehyde using Pt catalyst with and without CM (Table 1 entry 2 vs. entry 3)

5. Images of SEM and EDX mapping (Figure 1)



(a-1): SEM image of pre-mixed Pt and SiO₂-60-S. (b-1): SEM image of compression molded Pt and SiO₂-60-S. (c): EDX mapping of pre-mixed Pt and SiO₂-60-S. (d): EDX mapping of compression molded Pt and SiO₂-60-S.

6. Procedure and calculation for the void volume of the catalyst column

0.4wt% Pt with SiO₂-60-S was packed into a column (I.D. $\phi 10 \times 100$ mm). The catalyst column was sealed with plugs and weighted. Sealed plugs were removed, and the catalyst column was set onto the flow reactor. The catalyst column was filled with *t*-amy1OH by the pump at 0.5 mL/min for 30 min. After the solvent filling, the catalyst column was sealed with plugs again and weighted. Therefore, the void volume of the catalyst column was calculated as follows with the density of *t*-amy1OH at 20 °C (0.81 g/mL).

entry	CM	Pt and SiO ₂ -60-S	amount of <i>t</i> -amy1OH filled in column	void volume
1	without	3.25 g	5.15 g	6.36 mL
2	with	5.03 g	3.82 g	4.72 mL

7. Results of particle size distribution (Figure S1, 2, 3)

Figure S1

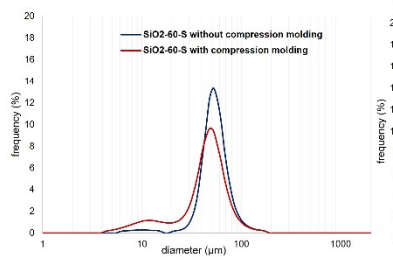


Figure 2

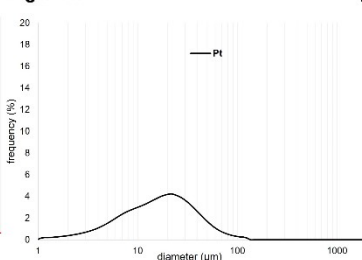
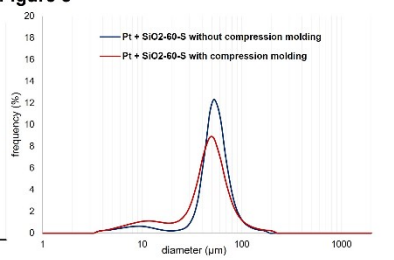


Figure 3



compression molding	MV (μm)	MN (μm)	MA (μm)	CS (m ² /mL)
without	53.59	14.15	44.76	0.13
with	45.11	8.52	29.47	0.20

MV (μm)	MN (μm)	MA (μm)	CS (m ² /mL)
8.37	3.84	6.42	0.93

compression molding	MV (μm)	MN (μm)	MA (μm)	CS (m ² /mL)
without	51.80	6.515	34.46	0.17
with	46.40	6.945	27.53	0.22

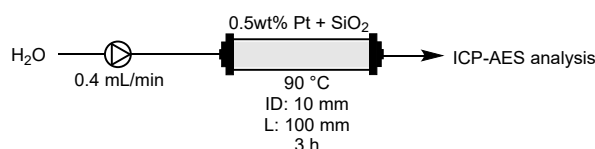
Figure S1 SiO ₂ -60-S		Figure S1 SiO ₂ -60-S		Figure 2 only Pt		Figure 3 Pt+SiO ₂ -60-S		Figure 3 Pt+SiO ₂ -60-S	
without CM	with CM	without CM	with CM	without CM	with CM	without CM	with CM	without CM	with CM
μm	frequency (%)	μm	frequency (%)	μm	frequency (%)	μm	frequency (%)	μm	frequency (%)
296.0	0.0	296.0	0.0	296.0	0.0	296.0	0.0	296.0	0.0
271.4	0.0	271.4	0.0	271.4	0.0	271.4	0.0	271.4	0.0
248.9	0.0	248.9	0.0	248.9	0.0	248.9	0.0	248.9	0.0
228.2	0.0	228.2	0.0	228.2	0.0	228.2	0.0	228.2	0.0
209.3	0.0	209.3	0.0	209.3	0.0	209.3	0.0	209.3	0.2
191.9	0.0	191.9	0.0	191.9	0.0	191.9	0.0	191.9	0.3
176.0	0.2	176.0	0.2	176.0	0.0	176.0	0.2	176.0	0.3
161.4	0.2	161.4	0.2	161.4	0.0	161.4	0.3	161.4	0.3
148.0	0.3	148.0	0.3	148.0	0.0	148.0	0.3	148.0	0.4
135.7	0.4	135.7	0.3	135.7	0.0	135.7	0.4	135.7	0.5
124.5	0.5	124.5	0.4	124.5	0.2	124.5	0.5	124.5	0.6
114.1	0.7	114.1	0.6	114.1	0.3	114.1	0.7	114.1	0.8
104.7	1.0	104.7	0.8	104.7	0.3	104.7	1.0	104.7	1.0
96.0	1.5	96.0	1.2	96.0	0.3	96.0	1.5	96.0	1.4
88.0	2.2	88.0	1.7	88.0	0.4	88.0	2.3	88.0	2.0
80.7	3.5	80.7	2.5	80.7	0.5	80.7	3.6	80.7	2.7
74.0	5.3	74.0	3.6	74.0	0.6	74.0	5.4	74.0	3.8
67.9	7.6	67.9	5.0	67.9	0.8	67.9	7.5	67.9	5.1
62.2	10.5	62.2	6.7	62.2	1.0	62.2	10.2	62.2	6.6
57.1	12.4	57.1	8.2	57.1	1.2	57.1	11.8	57.1	7.9
52.3	13.4	52.3	9.4	52.3	1.5	52.3	12.3	52.3	8.8
48.0	12.4	48.0	9.6	48.0	1.8	48.0	11.2	48.0	8.9
44.0	9.6	44.0	8.8	44.0	2.2	44.0	8.4	44.0	8.1
40.4	6.8	40.4	7.5	40.4	2.5	40.4	5.9	40.4	6.8
37.0	4.0	37.0	5.6	37.0	2.9	37.0	3.5	37.0	5.2
33.9	2.2	33.9	4.1	33.9	3.2	33.9	2.0	33.9	3.9
31.1	1.2	31.1	2.9	31.1	3.5	31.1	1.1	31.1	2.8

28.5	0.7	28.5	2.0	28.5	3.8	28.5	0.6	28.5	2.0
26.2	0.4	26.2	1.5	26.2	4.0	26.2	0.4	26.2	1.5
24.0	0.3	24.0	1.2	24.0	4.1	24.0	0.3	24.0	1.2
22.0	0.2	22.0	1.0	22.0	4.2	22.0	0.2	22.0	1.0
20.2	0.1	20.2	0.9	20.2	4.2	20.2	0.2	20.2	0.9
18.5	0.0	18.5	0.9	18.5	4.1	18.5	0.2	18.5	0.9
17.0	0.0	17.0	1.0	17.0	4.0	17.0	0.3	17.0	0.9
15.6	0.1	15.6	1.0	15.6	3.8	15.6	0.3	15.6	1.0
14.3	0.2	14.3	1.1	14.3	3.6	14.3	0.4	14.3	1.1
13.1	0.2	13.1	1.1	13.1	3.5	13.1	0.5	13.1	1.1
12.0	0.3	12.0	1.2	12.0	3.3	12.0	0.5	12.0	1.1
11.0	0.3	11.0	1.1	11.0	3.1	11.0	0.6	11.0	1.1
10.1	0.3	10.1	1.1	10.1	3.0	10.1	0.6	10.1	1.1
9.3	0.3	9.3	1.0	9.3	2.9	9.3	0.6	9.3	1.0
8.5	0.3	8.5	0.9	8.5	2.7	8.5	0.6	8.5	1.0
7.8	0.2	7.8	0.8	7.8	2.6	7.8	0.6	7.8	0.9
7.1	0.2	7.1	0.6	7.1	2.4	7.1	0.6	7.1	0.8
6.5	0.2	6.5	0.5	6.5	2.2	6.5	0.5	6.5	0.7
6.0	0.1	6.0	0.4	6.0	2.0	6.0	0.4	6.0	0.6
5.5	0.0	5.5	0.3	5.5	1.8	5.5	0.4	5.5	0.5
5.0	0.0	5.0	0.3	5.0	1.6	5.0	0.3	5.0	0.4
4.6	0.0	4.6	0.2	4.6	1.4	4.6	0.3	4.6	0.3
4.2	0.0	4.2	0.1	4.2	1.2	4.2	0.3	4.2	0.3
3.9	0.0	3.9	0.0	3.9	1.0	3.9	0.2	3.9	0.2
3.6	0.0	3.6	0.0	3.6	0.9	3.6	0.2	3.6	0.1
3.3	0.0	3.3	0.0	3.3	0.8	3.3	0.0	3.3	0.0
3.0	0.0	3.0	0.0	3.0	0.7	3.0	0.0	3.0	0.0
2.8	0.0	2.8	0.0	2.8	0.6	2.8	0.0	2.8	0.0
2.5	0.0	2.5	0.0	2.5	0.5	2.5	0.0	2.5	0.0
2.3	0.0	2.3	0.0	2.3	0.5	2.3	0.0	2.3	0.0
2.1	0.0	2.1	0.0	2.1	0.4	2.1	0.0	2.1	0.0
1.9	0.0	1.9	0.0	1.9	0.4	1.9	0.0	1.9	0.0
1.8	0.0	1.8	0.0	1.8	0.3	1.8	0.0	1.8	0.0
1.6	0.0	1.6	0.0	1.6	0.3	1.6	0.0	1.6	0.0
1.5	0.0	1.5	0.0	1.5	0.3	1.5	0.0	1.5	0.0
1.4	0.0	1.4	0.0	1.4	0.2	1.4	0.0	1.4	0.0
1.3	0.0	1.3	0.0	1.3	0.2	1.3	0.0	1.3	0.0
1.2	0.0	1.2	0.0	1.2	0.2	1.2	0.0	1.2	0.0

1.1	0.0	1.1	0.0	1.1	0.1	1.1	0.0	1.1	0.0
1.0	0.0	1.0	0.0	1.0	0.0	1.0	0.0	1.0	0.0
0.9	0.0	0.9	0.0	0.9	0.0	0.9	0.0	0.9	0.0
0.8	0.0	0.8	0.0	0.8	0.0	0.8	0.0	0.8	0.0
0.8	0.0	0.8	0.0	0.8	0.0	0.8	0.0	0.8	0.0

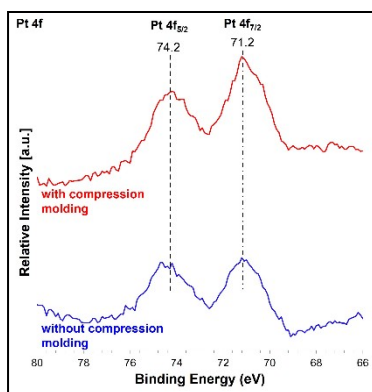
8. Procedure for ICP-AES analysis of Pt with/without compression molding (Table 2)

Pt black powder was pre-mixed with silica-gel and then packed into a column (ID: 10 mm, L: 100 mm). In the case of entry 2, the compression molding was conducted for the pre-mixed catalysts using a press machine (RIKEN KIKI CO., Ltd, CDM-5PA) and mold (AS ONE CORPORATION, mold for heat press machine ϕ 14) before packing to the reaction column. The catalyst column was set onto the reactor (EYELA, MCR-1000), and the catalyst column was heated at 90 °C (heating controller and column reactor: MCR-1000). Water was flowed at 0.4 mL/min (Pump: UI-22 110P, EYELA). After 3 hours, pumps were stopped. The whole liquid which passed through the reaction column was evaporated. Obtained powder was dissolved by aqua regia, and the solution was analyzed the amount of Pt by the inductivity coupled plasma atomic emission spectroscopy (ICP-AES: HORIBA ULTIMA2).



entry	CM	try	Amount of Pt packed in the column	ICP-AES analysis of Pt	
				amount of Pt flowing out	rate of Pt flowing out from column
1	without	1	17.95 mg	0.48 mg	2.66%
2	without	2	16.40 mg	0.68 mg	4.12%
3	with	1	21.20 mg	0.03 mg	0.13%
4	with	2	19.15 mg	0.24 mg	1.25%

9. XPS analysis (Figure 4)



without compression molding			with compression molding	
Binding energy (eV)	Counts per second		Binding energy (eV)	Counts per second
84	5164.382562		84	5604.213
83.9	5140.777145		83.9	5592.674
83.8	5092.661311		83.8	5558.812
83.7	5070.639644		83.7	5546.896
83.6	5022.674643		83.6	5518.238
83.5	4977.273809		83.5	5482.264
83.4	4941.149226		83.4	5455.717
83.3	4930.138392		83.3	5463.862
83.2	4890.393808		83.2	5444.48
83.1	4839.864641		83.1	5433.318
83	4786.394224		83	5403.529
82.9	4767.841724		82.9	5395.761
82.8	4781.642974		82.8	5356.393
82.7	4725.759223		82.7	5346.212
82.6	4691.520056		82.6	5358.43
82.5	4663.389639		82.5	5353.226
82.4	4625.455055		82.4	5320.57
82.3	4600.793805		82.3	5277.357
82.2	4606.902555		82.2	5278.111
82.1	4577.414638		82.1	5290.856
82	4548.454638		82	5270.569
81.9	4534.879637		81.9	5255.486
81.8	4499.358387		81.8	5234.218
81.7	4489.931304		81.7	5253.6
81.6	4485.25547		81.6	5201.035
81.5	4501.394637		81.5	5234.143
81.4	4464.968387		81.4	5217.023
81.3	4441.061303		81.3	5206.163
81.2	4425.902553		81.2	5163.93
81.1	4397.395052		81.1	5193.493
81	4394.001302		81	5182.332
80.9	4401.467552		80.9	5154.126
80.8	4364.890469		80.8	5180.069
80.7	4332.235052		80.7	5171.245
80.6	4327.860885		80.6	5135.95

80.5	4322.204635		80.5	5132.858
80.4	4315.190885		80.4	5142.361
80.3	4301.389635		80.3	5120.339
80.2	4282.082968		80.2	5133.99
80.1	4309.006718		80.1	5130.671
80	4303.048801		80	5150.883
79.9	4298.071301		79.9	5100.731
79.8	4300.861718		79.8	5104.125
79.7	4284.345468		79.7	5112.043
79.6	4230.950467		79.6	5091.907
79.5	4272.052551		79.5	5142.135
79.4	4270.921301		79.4	5117.85
79.3	4253.877134		79.3	5100.429
79.2	4223.936717		79.2	5100.58
79.1	4245.430467		79.1	5110.761
79	4209.682967		79	5115.362
78.9	4197.314633		78.9	5120.49
78.8	4226.877967		78.8	5096.357
78.7	4236.154217		78.7	5106.161
78.6	4232.68505		78.6	5128.786
78.5	4216.319634		78.5	5124.035
78.4	4212.020884		78.4	5127.127
78.3	4210.135467		78.3	5128.71
78.2	4205.3088		78.2	5131.501
78.1	4186.228383		78.1	5131.953
78	4184.644633		78	5145.905
77.9	4183.5888		77.9	5164.307
77.8	4181.627967		77.8	5160.008
77.7	4189.094217		77.7	5128.56
77.6	4211.6438		77.6	5174.488
77.5	4212.0963		77.5	5183.689
77.4	4184.644633		77.4	5158.349
77.3	4192.563383		77.3	5166.042
77.2	4224.841717		77.2	5191.08
77.1	4212.699634		77.1	5181.351
77	4217.224634		77	5191.985
76.9	4221.1463		76.9	5173.583

76.8	4234.872134		76.8	5217.551
76.7	4209.909217		76.7	5224.188
76.6	4208.325467		76.6	5199.753
76.5	4225.595884		76.5	5193.87
76.4	4241.961301		76.4	5251.79
76.3	4239.170884		76.3	5257.296
76.2	4297.166301		76.2	5261.821
76.1	4280.046718		76.1	5251.941
76	4273.259218		76	5245.983
75.9	4284.873384		75.9	5324.04
75.8	4263.907551		75.8	5321.551
75.7	4338.720885		75.7	5344.176
75.6	4359.234219		75.6	5372.533
75.5	4371.979635		75.5	5396.817
75.4	4383.518386		75.4	5395.308
75.3	4400.788802		75.3	5439.201
75.2	4428.315886		75.2	5463.108
75.1	4479.37297		75.1	5500.138
75	4501.09297		75	5578.269
74.9	4522.134221		74.9	5610.472
74.8	4546.116721		74.8	5645.164
74.7	4586.238388		74.7	5665.602
74.6	4582.769221		74.6	5664.772
74.5	4551.471304		74.5	5699.841
74.4	4559.390054		74.4	5699.992
74.3	4543.100054		74.3	5714.698
74.2	4579.752555		74.2	5724.2
74.1	4540.611304		74.1	5716.282
74	4493.023387		74	5671.635
73.9	4505.316304		73.9	5666.507
73.8	4482.917553		73.8	5668.995
73.7	4468.135887		73.7	5670.881
73.6	4447.094636		73.6	5611.453
73.5	4434.424636		73.5	5576.459
73.4	4427.410886		73.4	5559.566
73.3	4417.078803		73.3	5557.077
73.2	4361.949219		73.2	5544.558

73.1	4350.033385		73.1	5510.847
73	4335.628802		73	5462.655
72.9	4347.242968		72.9	5479.85
72.8	4313.757968		72.8	5447.27
72.7	4294.903801		72.7	5439.427
72.6	4278.462968		72.6	5435.732
72.5	4290.755884		72.5	5491.54
72.4	4319.565051		72.4	5502.702
72.3	4330.274218		72.3	5521.179
72.2	4361.949219		72.2	5542.899
72.1	4386.836719		72.1	5566.806
72	4402.674219		72	5623.595
71.9	4427.712553		71.9	5665.526
71.8	4486.38672		71.8	5661.378
71.7	4522.435887		71.7	5714.019
71.6	4537.519221		71.6	5790.793
71.5	4556.675054		71.5	5795.243
71.4	4573.568388		71.4	5846.828
71.3	4580.431305		71.3	5920.51
71.2	4616.706722		71.2	5953.316
71.1	4593.101305		71.1	5940.42
71	4602.302138		71	5917.569
70.9	4575.227555		70.9	5885.818
70.8	4555.694638		70.8	5856.858
70.7	4533.597554		70.7	5838.23
70.6	4515.045054		70.6	5832.348
70.5	4508.55922		70.5	5805.877
70.4	4474.998803		70.4	5770.205
70.3	4420.397136		70.3	5769.375
70.2	4405.841719		70.2	5694.185
70.1	4374.091302		70.1	5642.147
70	4313.003801		70	5613.338
69.9	4276.879218		69.9	5571.18
69.8	4263.455051		69.8	5506.774
69.7	4230.1963		69.7	5469.066
69.6	4206.967967		69.6	5447.874
69.5	4222.352967		69.5	5394.479

69.4	4170.617133		69.4	5382.865
69.3	4134.266299		69.3	5368.158
69.2	4133.361299		69.2	5332.713
69.1	4089.619632		69.1	5349.153
69	4110.660882		69	5360.39
68.9	4101.309216		68.9	5348.55
68.8	4129.665883		68.8	5338.972
68.7	4132.154633		68.7	5339.953
68.6	4129.741299		68.6	5350.285
68.5	4117.448382		68.5	5338.218
68.4	4133.059633		68.4	5352.924
68.3	4137.282966		68.3	5363.407
68.2	4161.340883		68.2	5361.446
68.1	4134.115466		68.1	5340.103
68	4163.603383		68	5362.728
67.9	4170.994216		67.9	5356.243
67.8	4147.388799		67.8	5337.087
67.7	4160.435883		67.7	5319.665
67.6	4162.09505		67.6	5326.528
67.5	4183.664217		67.5	5366.424
67.4	4190.60255		67.4	5380.904
67.3	4178.535883		67.3	5385.429
67.2	4196.862133		67.2	5368.46
67.1	4182.155883		67.1	5370.572
67	4187.43505		67	5344.704
66.9	4180.8738		66.9	5357.525
66.8	4181.401717		66.8	5338.821
66.7	4171.974633		66.7	5338.595
66.6	4206.44005		66.6	5353.603
66.5	4213.4538		66.5	5356.77
66.4	4220.090467		66.4	5354.583
66.3	4225.6713		66.3	5336.333
66.2	4251.312967		66.2	5337.011
66.1	4234.872134		66.1	5323.813
66	4211.040467		66	5325.85

10. Procedure for the scale-up experiment for the flow reaction

Pt black powder (700 mg) was pre-mixed with silica-gel (140 g) and then packed into a column (ID: 37 mm, L: 300 mm). The compression molding was conducted for the pre-mixed catalysts using a press machine (RIKEN KIKI CO., Ltd, CDM-5PA) and mold (Labonect, LD-3025, $\phi 30 \times 25$ mm) under 40 MPa for 2 hours before packing to the reaction column. The catalyst column was set onto the reactor (EYELA, HCR-1000), and H₂O was flowed at each 3.0 mL/min (Pump for H₂O₂ aq.: UI-22 110P, EYELA, Pump for *t*-amy1OH solution: LC-20AD, Shimadzu). The catalyst column was heated at 90 °C (heating controller and column reactor: HCR-1000). After stabilization the reaction was started by changing into feed solution of 10wt% H₂O₂ aq. and benzyl alcohol (**1**, 0.67 M) solution in *t*-amy1OH. The resulted solution was fractionated for every 5 minutes (30 mL) (Fraction collector: DC-1500, EYELA), and selected fractions were analyzed by GC-FID (N₂: 34.0 cm/s, column temperature: 50 °C for 2 min, 10 °C/min heating then 240 °C for 12 min) to determine GC conversion and yield.

