Electronic Supplementary Information (ESI)

Continuous-flow liquid-phase dehydrogenation of 1,4-cyclohexanedione in a structured multichannel reactor

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S.1. Supplementary Information

The experiments for liquid phase dehydrogenation of 1,4-cyclohexanedione were performed in a mm-scale structured multichannel reactor which was developed at University of Bath[1] as shown in Fig. S1. The reactor consists of five catalytic channels of square shape with dimensions of 2 mm × 2 mm (one channel), 3 mm × 3 mm (three channels), and 5 mm × 5 mm (one channel). Each channel is equipped with upfront 6 cm long static mixer and inlet ports for liquid feed and gas streams. The static mixer served as feed preheater for endothermic dehydrogenation reaction. The total length of catalytic channel is 10 cm and the channel outlet is designed in a way the catalyst particles can be packed and removed easily from the reactor.

The scheme of experimental setup used in this study is shown in Fig. 1. The liquid feed was stored in the feed vessel and pumped at flow rates (F_L) of 0.10 – 0.50 mL/min (0.7 – 3.3 $m^3\ m^{-2}\ h^{-1}$ at STP) using a HPLC isocratic pump (Series I, 10 ml/min). The N_2 gas flow (F_G) was controlled at 5 – 85 mL/min (33.3 – 602.1 $m^3\ m^{-2}\ h^{-1}$ at STP) with a mass flow controller (Bronkhorst, 100 ml/min). The reactor temperature was controlled by using integrated heat exchange system which circulated silicon oil (Sil 260) as heat transfer fluid (HTF) via a Phoenix-II (P1-B7) heating re-circulator. A thermocouple was inserted at the HTF inlet to monitor the reactor temperature (T). The reactor outlet pressure was maintained at 1.5 bar using the pressure control valve (PCV) and the back pressure regulator (BPR). The pressure drop across the reactor was monitored using a Bronkhorst differential pressure transducer. The reactor effluent was passed through a low dead volume six-way valve (Valco) to collect samples for analysis. The liquid was stored in a stainless-steel gas-liquid separator from where the

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gaseous effluent was cooled at 20 ℃ in a condenser and condensable was separated in liquid trap for the purpose to reduce load on the hydrocarbon trap. The temperature of reactor outlet tubing and of gas-liquid separator was maintained at 30 ℃ by using electric trace heating. The LabVIEW software was utilized for monitoring, controlling, and data acquisition of temperature and pressure drop during experimental evaluation.

The overall liquid-solid mass transfer co-efficient (k_{LS}) was calculated from Dwidevi and Upadhyay correlation[2] as given by Eq. (S1) which is valid for liquids with Reynold number values > 0.01 in fixed bed reactors.

$$k_{LS} = \frac{Sh\, D_{AB}}{d_p} \tag{S1}$$

where ε_b is the catalyst bed voidage (0.40), *Sh* is the Sherwood number, D_{AB} is the diffusion coefficient, and d_p is the average diameter of catalyst particles (64.4 µm).

Sherwood number (*Sh*)[3] was calculated as

$$Sh = J_D\, Sc^{1/3}\, Re \tag{S2}$$

$$\varepsilon_b J_D = \frac{0.765}{Re^{0.82}} + \frac{0.365}{Re^{0.386}} \tag{S3}$$

Schmidt number (*Sc*)[3] was calculated as

$$Sc = \frac{\mu_L}{\rho_L\, D_{AB}} \tag{S4}$$

Reynold number (*Re*)[3] was calculated as

$$Re = \frac{d_p\, u_L\, \rho_L}{\mu_L} \tag{S5}$$

where μ_L is the feed mixture viscosity (Pa.s), ρ_L is the feed mixture density (kg/m^3), and u_L is the superficial liquid feed velocity (m/s).

Diffusion coefficient (D_{AB}) of 1,4 cyclohexanedione in solvent liquid was calculated by using Wilke-Change equation[4] described by Eq. (S6).

$$D_{AB} = \frac{117.3 \times 10^{-18} (x\, M_B)^{1/2}\, T}{\mu_L\, v_A^{0.6}} \tag{S6}$$

where M_B is the molecular weight of tetraethylene glycol dimethyl ether (kg/kmol), x is the interaction coefficient (1.0 for ether), T is the temperature (K), and v_A is the molar volume of 1,4 cyclohexanedione at normal boiling point (m^3/kmol).

The effective diffusivity (D_{eff})[5-7] was estimated by Eq. (S7) with one empirical constant for a given material.

$$D_{eff} = D_{AB}\frac{(1-\lambda)^2}{1+P\lambda} \quad \text{(S7)}$$

where λ is the ratio of the radius of diffusing molecule (0.51 nm) to the pore radius (2.05 nm) and P is the fitting parameter (16.3).

The tortuosity (τ_b=1.58) of catalyst packed bed was calculated using Eq. (S8) as given in the study by Puncochar and Drahos.[8]

$$\tau_b = \frac{1}{\sqrt{\varepsilon_b}} \quad \text{(S8)}$$

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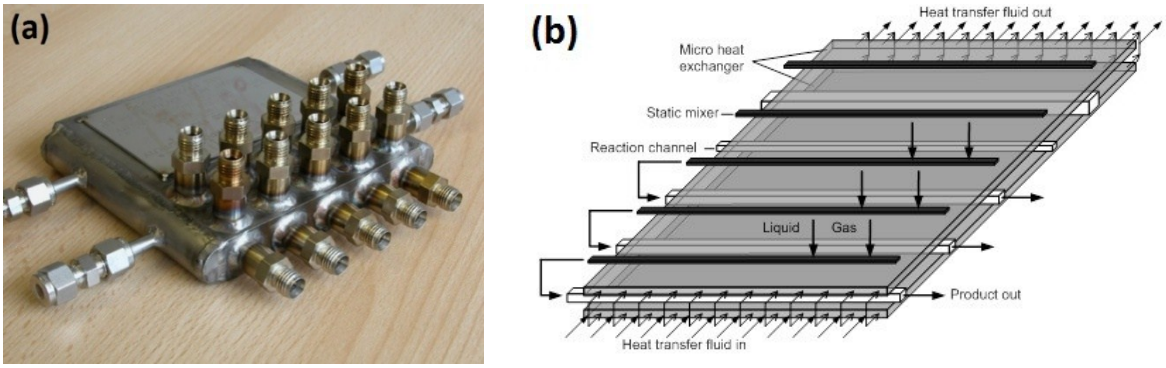

Fig. S1. The mm-scale structured multichannel catalytic reactor: (a) reactor assembly, (b) schematic diagram of reactor internals.

Table S1. Experimental design for continuous dehydrogenation of 1,4-cyclohexanedione

Experiment Name	Run Order	Process parameters				Response		
		T (X_1)	F_G (X_2)	F_L (X_3)	C_o (X_4)	Conversion (Y_1)	Selectivity (Y_2)	Pressure Drop (Y_3)
N1	14	199	5	0.1	1	14.1	99.2	1.3
N2	12	220	40	0.1	1	60.5	99.3	3.1
N3	6	180	80	0.1	1	19.9	99.4	4.2
N4	20	235	80	0.1	1	67.7	97.4	3.5
N5	17	235	5	0.2	1	61.2	99.4	1.7
N6	28	181	5	0.3	1	1.5	99.8	3.0
N7	23	236	5	0.5	1	15.3	99.4	3.0
N8	22	199	20	0.5	1	3.6	99.2	5.9
N9	21	181	80	0.5	1	2.6	99.4	8.8
N10	18	235	80	0.5	1	15.1	99.5	7.2
N11	19	179	5	0.1	5	8.0	99.5	1.4
N12	16	199	80	0.2	5	40.0	99.8	4.6
N13	13	239	20	0.3	5	57.7	99.3	3.7
N14	11	219	5	0.5	5	9.9	99.4	2.2
N15	15	179	40	0.5	5	0.9	99.3	2.7
N16	24	199	5	0.1	10	17.8	99.6	1.6
N17	2	240	5	0.1	10	79.1	91.9	0.5
N18	27	180	80	0.1	10	23.1	99.6	4.1
N19	30	239	80	0.1	10	60.7	95.0	2.0
N20	3	180	20	0.2	10	4.5	99.5	3.0
N21	26	220	5	0.32	10	13.4	99.6	2.2
N22	4	199	40	0.3	10	8.9	99.8	5.5
N23	1	180	5	0.5	10	0.6	99.6	3.3
N24	25	239	5	0.5	10	16.9	97.7	3.6
N25	5	238	40	0.5	10	25.9	99.5	5.9
N26	10	180	80	0.5	10	1.5	99.2	6.6
N27	29	220	80	0.5	10	18.1	99.6	7.0
N28	8	220	40	0.3	5	28.9	99.2	5.0
N29	9	221	40	0.3	5	27.1	99.7	5.3
N30	7	220	40	0.3	5	29.8	99.7	4.6
N31	34	233	30	0.1	10	79.8	93.3	2.7
N32	33	233	60	0.1	10	74.0	97.4	4.1
N33	32	233	60	0.5	10	18.4	99.1	8.3
N34	31	233	80	0.5	10	18.0	99.0	11.7

Y_1 = conversion (X, %) of 1,4-cyclohexanedione, Y_2 = selectivity (S, %) to hydroquinone, Y_3 = pressure drop (ΔP, bar), X_1 = temperature (T, °C), X_2 = nitrogen flow (F_G, mL/min), X_3 = liquid feed flow (F_L, mL/min), X_4 = 1,4-cyclohexanedione concentration (C_o, wt %) in tetraethylene glycol dimethyl ether.

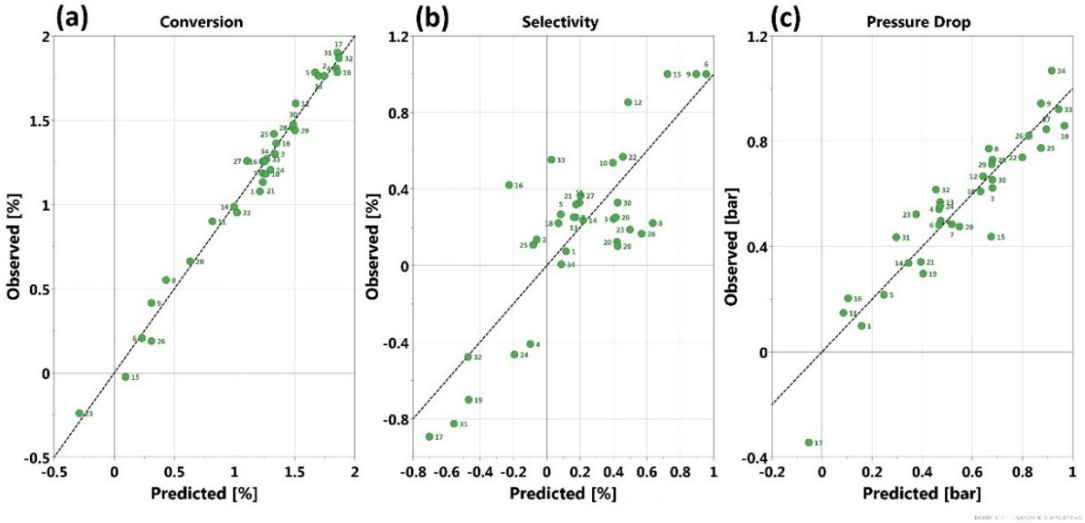

Fig. S2. The predicted versus observed values of (a) conversion of 1,4-cyclohexanedione (%), (b) selectivity to hydroquinone (%), (c) pressure drop (bar) for continuous multiphase dehydrogenation of 1,4-cyclohexanedione.

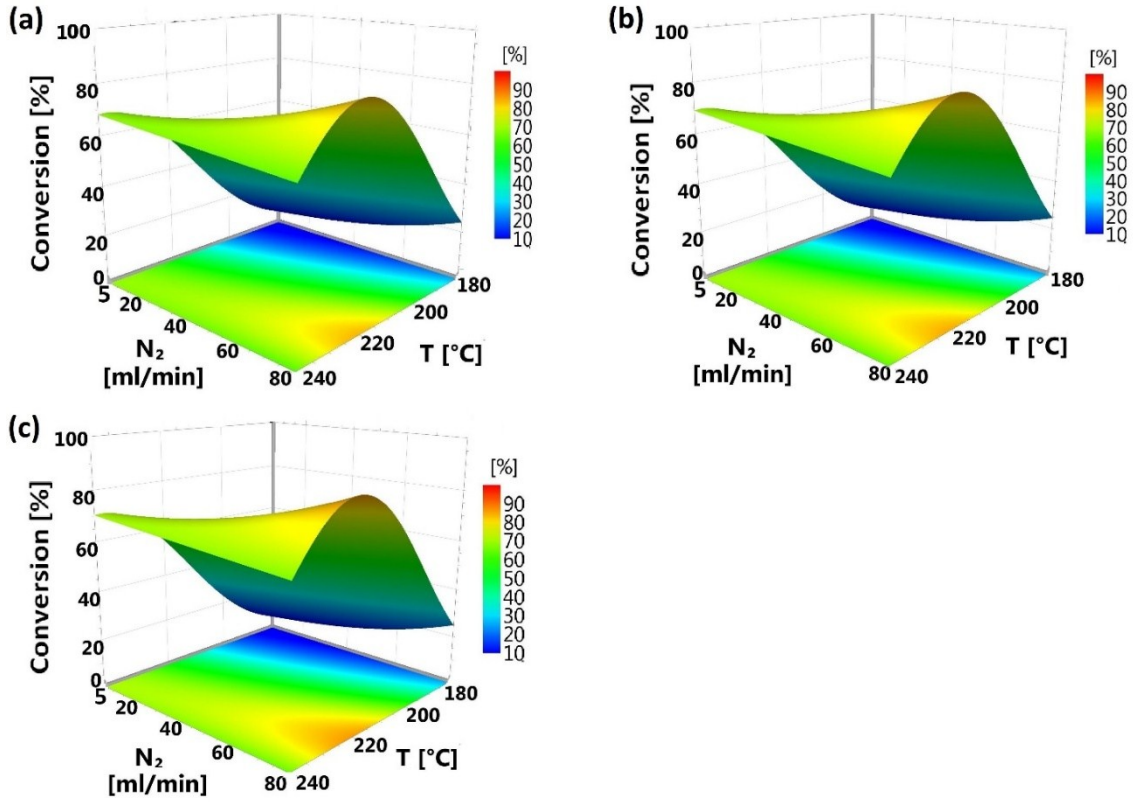

Fig. S3. Influence of temperature (180 – 240°C) and N_2 flow (5 – 80 mL/min, 33.3 – 566.7 $m^3\,m^{-2}\,h^{-1}$ at STP) on the conversion of 1,4-cyclohexanedione for liquid feed flow of 0.1 mL/min (0.7 $m^3\,m^{-2}\,h^{-1}$ at STP) and 1,4-cyclohexanedione concentration of (a) 1 wt%, (b) 5 wt%, (c) 10 wt% in tetraethylene glycol dimethyl ether.

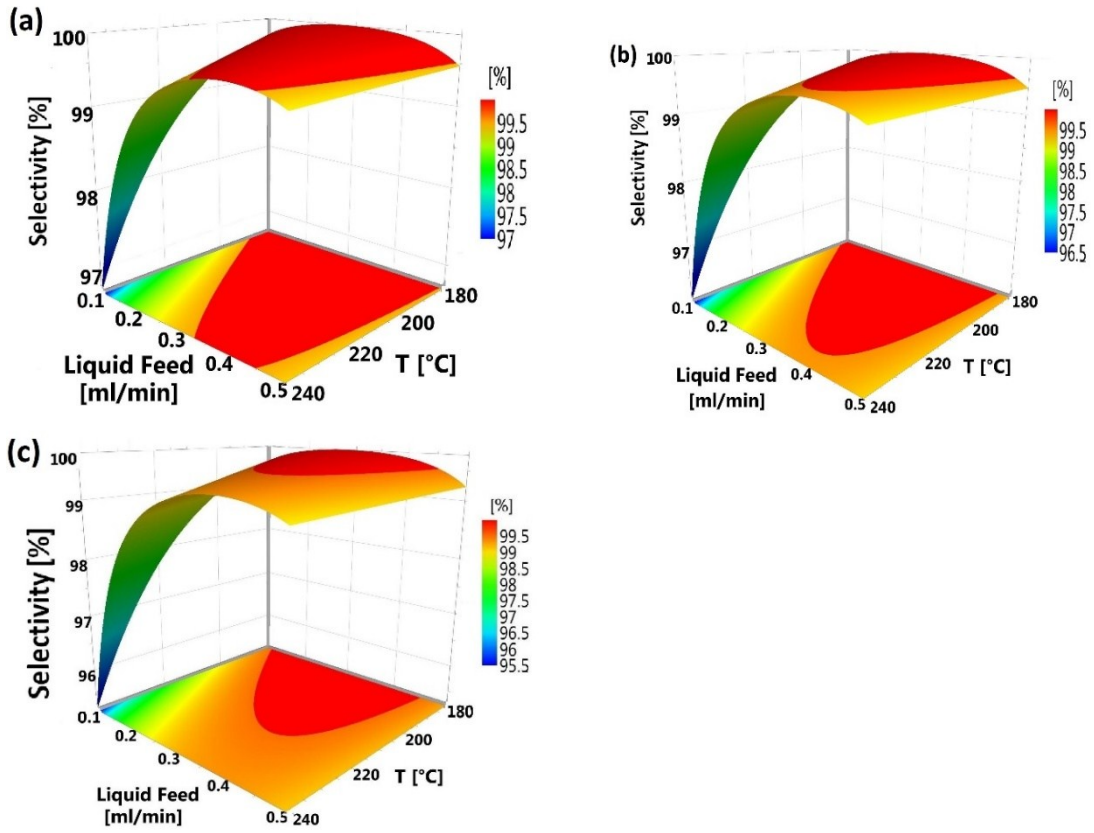

Fig. S4. Influence of temperature (180 – 240°C) and liquid feed flow (0.1 – 0.5 mL/min, 0.7 – 3.3 $m^3\,m^{-2}\,h^{-1}$ at STP) on the selectivity to hydroquinone for N_2 flow of 5 mL/min (33.3 $m^3\,m^{-2}\,h^{-1}$ at STP) and substrate concentration of (a) 1 wt%, (b) 5 wt%, (c) 10 wt% in tetraethylene glycol dimethyl ether.

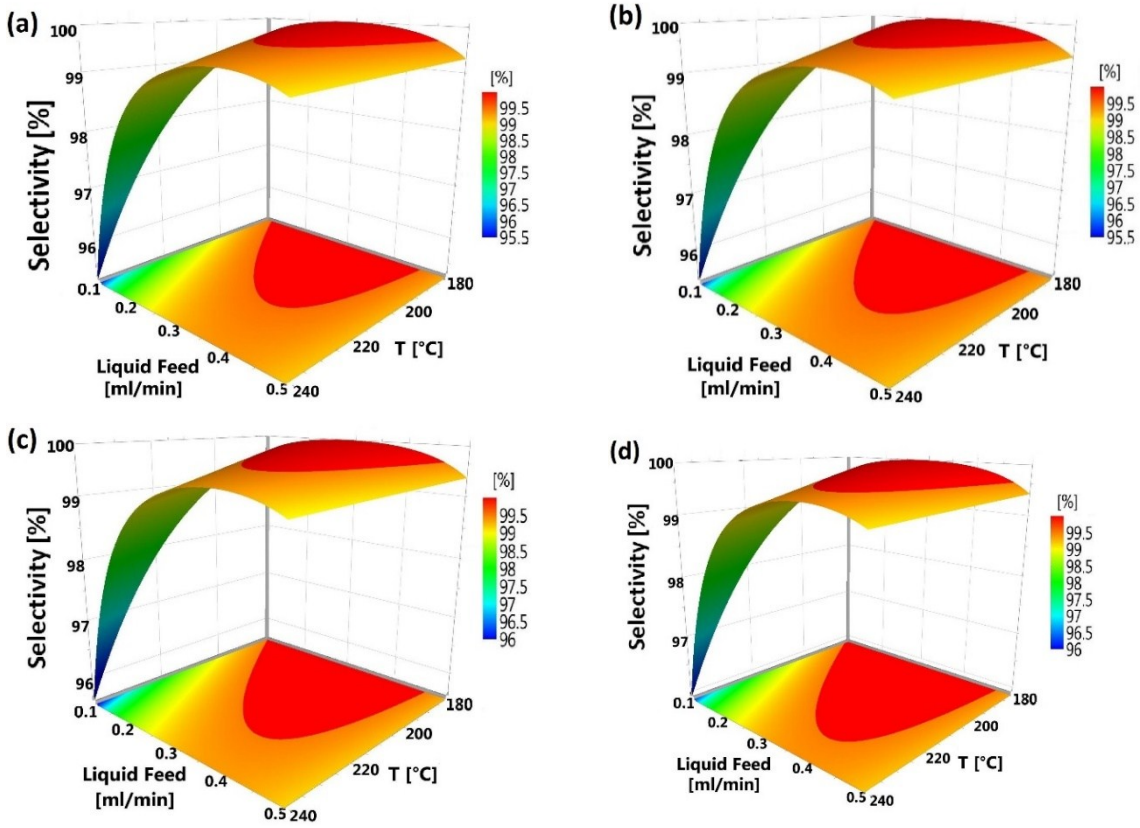

Fig. S5. Influence of temperature (180 – 240°C) and liquid feed flow (0.1 – 0.5 mL/min, 0.7 – 3.3 $m^3\,m^{-2}\,h^{-1}$ at STP) on the selectivity to hydroquinone for 1,4-cyclohexanedione concentration of 10 wt% in tetraethylene glycol dimethyl ether and N_2 flow of (a) 5 mL/min (33.3 $m^3\,m^{-2}\,h^{-1}$ at STP), (b) 20 mL/min (133.2 $m^3\,m^{-2}\,h^{-1}$ at STP), (c) 40 mL/min (266.4 $m^3\,m^{-2}\,h^{-1}$ at STP), (d) 80 mL/min (566.7 $m^3\,m^{-2}\,h^{-1}$ at STP).

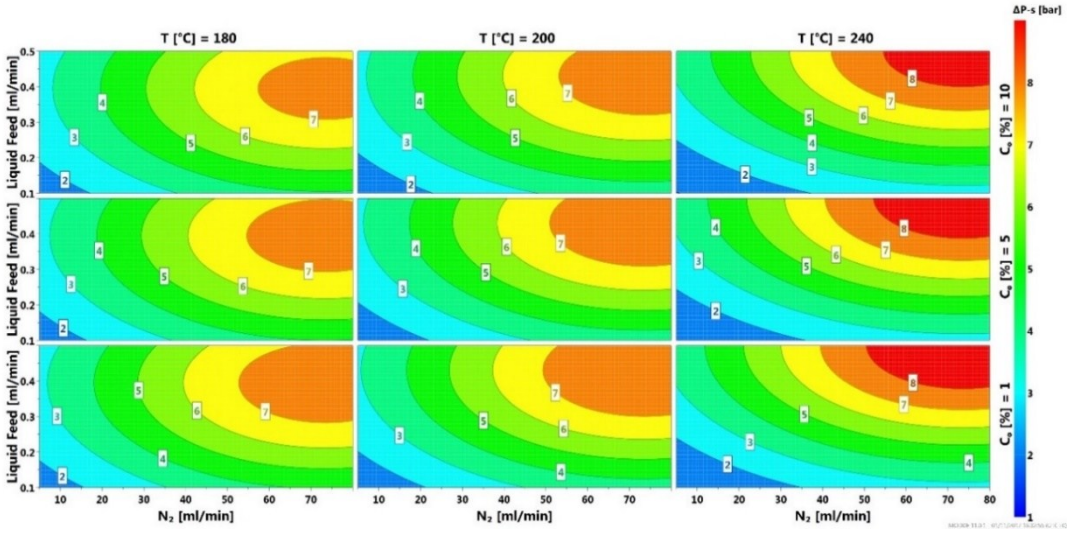

Fig. S6. Influence of operating conditions on the pressure drop for continuous dehydrogenation of 1,4-cyclohexanedione.

Table S2. Experimental design matrix for semi-continuous dehydrogenation of 1,4-cyclohexanedione

Experiment Name	Run Order	Factors				Response		
		T (X_1)	F_G (X_2)	F_L (X_3)	X_{in} (X_5)	Conversion (Y_4)	Selectivity (Y_5)	ΔP (Y_6)
N1	16	200	5	0.1	20	32.3	99.5	2.1
N2	22	242	85	0.5	20	35.7	99.3	9.0
N3	3	200	5	0.1	80	85.2	99.5	1.8
N4	4	239	5	0.1	80	88.2	99.0	1.8
N5	9	200	5	0.5	80	81.7	99.34	5.2
N6	7	200	85	0.1	80	88.7	99.5	6.0
N7	10	201	85	0.5	60	65.6	99.6	9.3
N8	5	200	31.7	0.5	20	21.3	99.7	7.8
N9	14	200	85	0.37	20	24.2	99.6	9.8
N10	18	241	85	0.1	40	93.5	92.9	4.1
N11	11	243	58.3	0.1	20	79.3	89.4	3.4
N12	2	240	5	0.5	60	77.2	99.5	3.6
N13	19	241	31.7	0.5	80	88.1	99.1	10.4
N14	8	243	5	0.23	20	58.6	99.4	2.3
N15	1	240	85	0.23	80	85.6	99.0	7.1
N16	12	213	85	0.1	20	39.4	99.5	5.8
N17	15	213	5	0.5	20	25.5	99.4	4.7
N18	17	227	85	0.5	80	81.8	99.8	11.7
N19	21	222	45	0.3	50	64.3	99.4	6.1
N20	13	220	45	0.3	50	60.4	99.7	6.5
N21	20	221	45	0.3	50	61.6	99.6	6.3
N22	6	220	45	0.3	50	65.6	99.2	5.1
N23	24	200	58.3	0.5	95	95.9	99.5	13.6
N24	26	221	40	0.3	0	58.3	99.6	5.2
N25	25	239	20	0.3	0	59.4	99.6	3.8
N26	23	219	45	0.3	95	96.6	99.2	7.0
N27	28	238	58.3	0.1	100	100.0	90.6	5.2
N28	30	219	45	0.3	100	100.0	99.6	9.1
N29	29	241	85	0.1	95	97.6	96.6	6.0
N30	27	200	5	0.5	100	100.0	99.6	5.3
N31	33	237	85	0.5	100	100.0	99.7	13.6
N32	31	220	5	0.5	0	9.8	99.4	2.2
N33	32	199	80	0.2	0	40.0	99.5	4.6

Y_4 = conversion (X, %), Y_5 = selectivity (S, %), Y_6 = pressure drop (ΔP, bar), X_1 = temperature (T, °C), X_2 = N_2 flow (F_G, mL/min), X_3 = liquid feed flow (F_L, mL/min), X_5 = reactor inlet conversion (X_{in}, %).

Table S3. Analysis of variance (ANOVA) of fitted model for semi-continuous dehydrogenation

Source	DF	SS	MS (variance)	F	p	SD
(a) Conversion, X (Y_4)						
Total	30	154769	5158.98			
Constant	1	133432	133432			
Total corrected	29	21337.30	735.77			27.13
Regression	6	19856.30	2836.62	42.14	0	53.26
Residual	23	1480.96	67.32			8.21
Lack of Fit (LoF)	20	1466.16	73.31	9.91	0.10	8.56
Pure error (pe)	2	14.80	7.40			2.72
RSD × sqrt(F(crit)) = 8.56						
SD-pe × sqrt(F(crit)) = 11.99						
(b) Selectivity, S (Y_5)						
Total	33	6.69	0.20			
Constant	1	0.95	0.95			
Total corrected	32	5.74	0.18			0.42
Regression	6	4.18	0.70	11.62	0	0.83
Residual	26	1.56	0.06			0.24
Lack of Fit (LoF)	22	1.41	0.06	1.74	0.32	0.25
Pure error (pe)	4	0.15	0.04			0.19
RSD × sqrt(F(crit)) = 0.25						
SD-pe × sqrt(F(crit)) = 0.46						
(c) Pressure drop, ΔP (Y_3)						
Total	31	18.05	0.58			
Constant	1	16.27	16.27			
Total corrected	30	1.78	0.06			0.24
Regression	5	1.62	0.32	52.77	0	0.57
Residual	25	0.15	0.01			0.08
Lack of Fit (LoF)	22	0.15	0.01	2.99	0.20	0.08
Pure error (pe)	3	0.01	2.23×10^{-3}			0.05
RSD × sqrt(F(crit)) = 0.08						
SD-pe × sqrt(F(crit)) = 0.14						

DF = degree of freedom, SS = sum of square, MS = mean square, SD = standard deviation, RSD = residual standard deviation, sqrt(F(crit)) = square root of critical F.

Table S4. Regression coefficient of models for semi-continuous dehydrogenation

Model term	Coefficient estimate	Standard error	P
(a) Conversion, X (Y_4)			
Constant	63.01	2.54	1.45×10^{-17}
X_1	8.58	1.77	7.66×10^{-5}
X_2	1.09	1.91	0.58
X_3	-8.74	1.85	1.02×10^{-4}
X_5	32.38	2.22	8.36×10^{-13}
$X_1 \times X_5$	6.36	2.67	7.21×10^{-4}
$X_3 \times X_5$	-9.49	2.65	1.70×10^{-3}
X_5^2	10.49	4.36	0.16
(b) Selectivity, S (Y_5)			
Constant	0.39	0.08	3.23×10^{-5}
X_1	-0.24	0.05	9.36×10^{-5}
X_2	-0.02	0.06	0.76
X_3	0.22	0.05	0.66
X_5	-0.03	0.06	0.09
X_3^2	-0.28	0.10	7.10×10^{-3}
$X_1 \times X_3$	0.23	0.06	6.31×10^{-4}
(c) Pressure drop, ΔP-r (Y_6)			
Constant	0.81	0.03	1.49×10^{-21}
X_1	-0.02	0.01	0.18
X_2	0.21	0.02	5.43×10^{-12}
X_3	0.17	0.02	3.77×10^{-10}
X_5	0.07	0.02	1.85×10^{-4}
X_2^2	-0.15	0.03	6.81×10^{-5}

DF = degree of freedom, SS = sum of square, MS = mean square, SD = standard deviation, RSD = residual standard deviation, and sqrt(F(crit)) = square root of critical F.

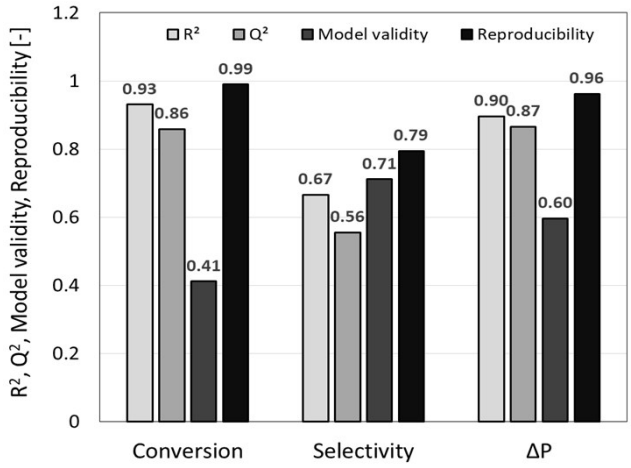

Fig. S7. The summary of fit for semi-continuous dehydrogenation of 1,4-cyclohexanedione

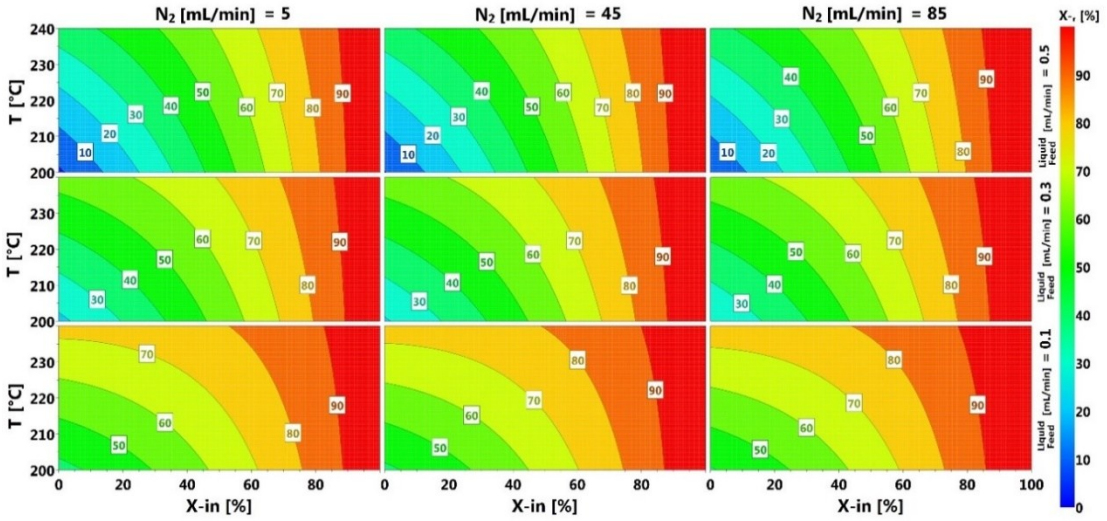

Fig. S8. Influence of operating conditions on recycled reactor conversion

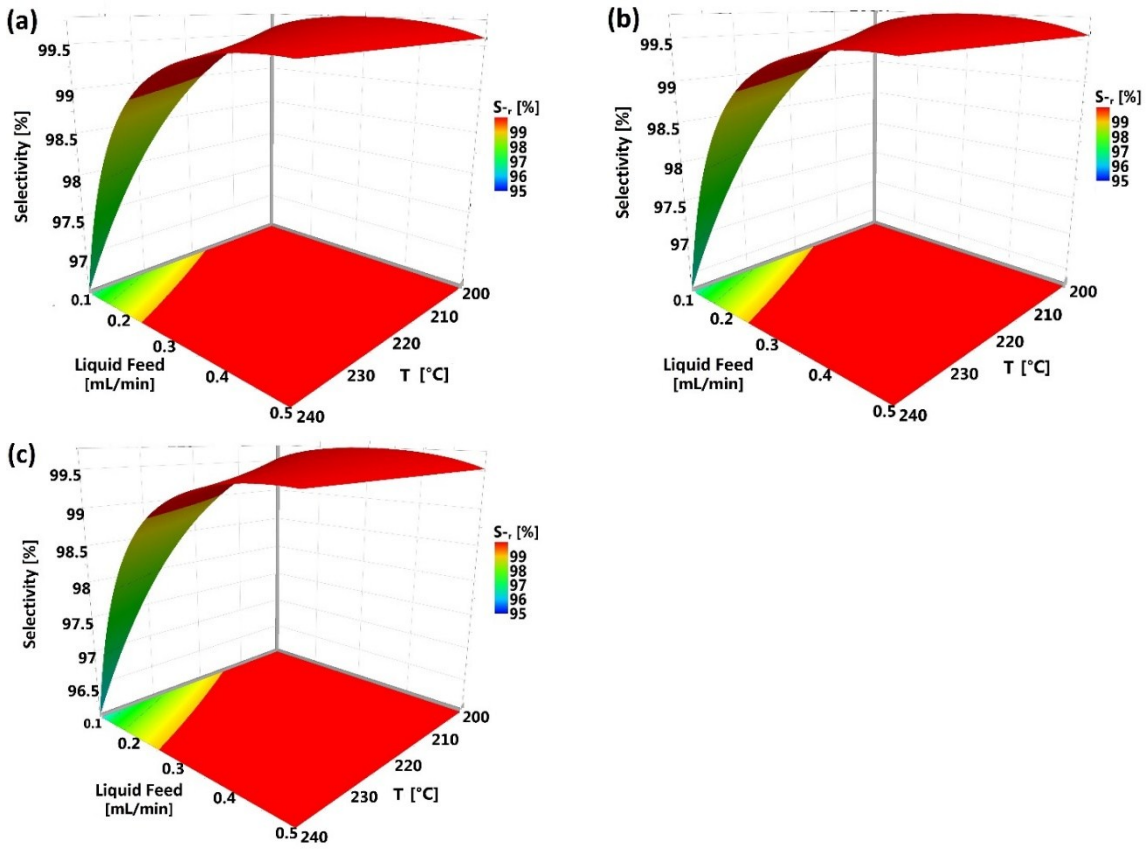

Fig. S9. Influence of temperature (180 – 240°C) and liquid feed flow (0.1 – 0.5 mL/min, 0.7 – 3.3 $m^3\,m^{-2}\,h^{-1}$ at STP) on the selectivity to hydroquinone for N_2 flow of 5 mL/min (33.3 $m^3\,m^{-2}\,h^{-1}$ at STP) and reactor inlet conversion of 1,4-cyclohexanedione of (a) 0 %, (b) 50 %, (c) 100 %.

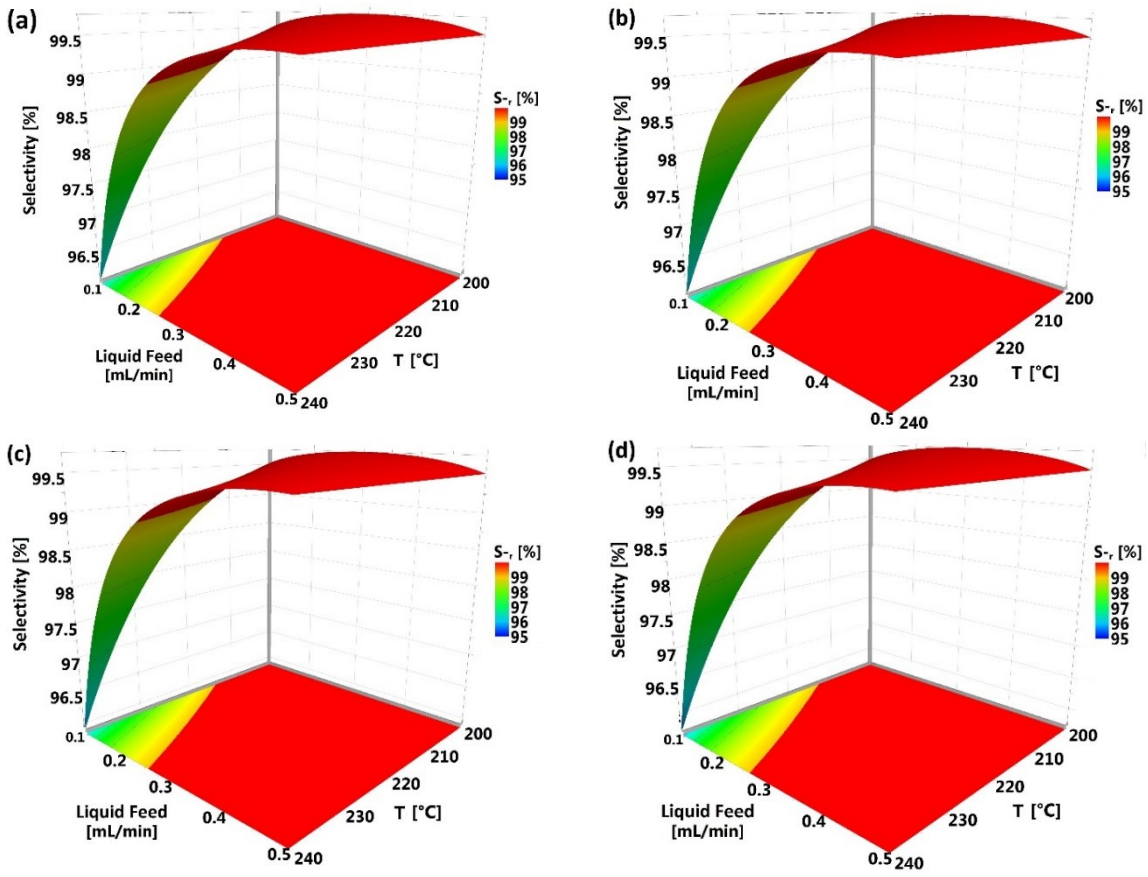

Fig. S10. Influence of temperature (180 – 240°C) and liquid feed flow (0.1 – 0.5 mL/min, 0.7 – 3.3 $m^3\,m^{-2}\,h^{-1}$ at STP) on the selectivity to hydroquinone for reactor inlet conversion of 100% and N_2 flow of (a) 5 mL/min (33.3 $m^3\,m^{-2}\,h^{-1}$ at STP), (b) 20 mL/min (133.2 $m^3\,m^{-2}\,h^{-1}$ at STP), (c) 40 mL/min (266.4 $m^3\,m^{-2}\,h^{-1}$ at STP), (d) 80 mL/min (566.7 $m^3\,m^{-2}\,h^{-1}$ at STP).

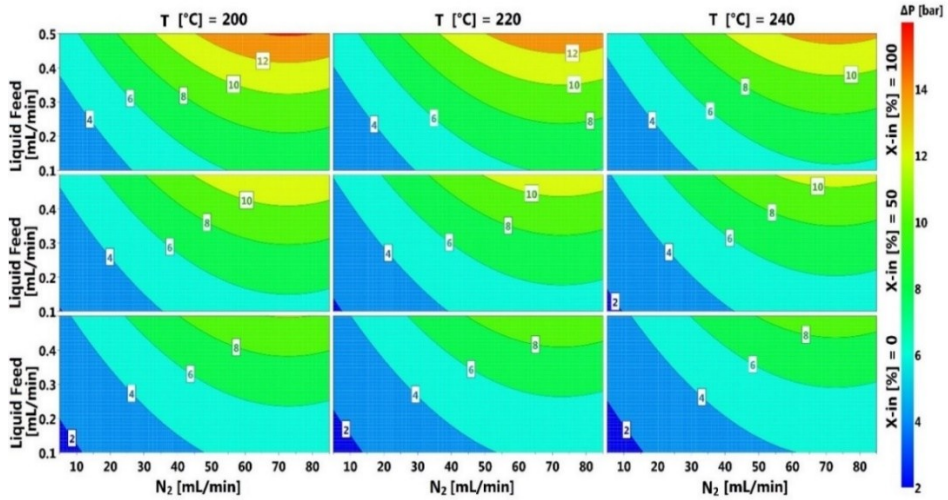

Fig. S11. Influence of operating conditions on the pressure drop for continuous dehydrogenation of 1,4-cyclohexanedione

Table S5. Influence of 1,4-cyclohexanedione conversion on density and viscosity of feed

Conversion	0 %	50 %	100 %
Density kg/m^3	803.3	806.1	808.9
Viscosity g/sec-cm	2.6×10^{-3}	2.7×10^{-3}	2.8×10^{-3}

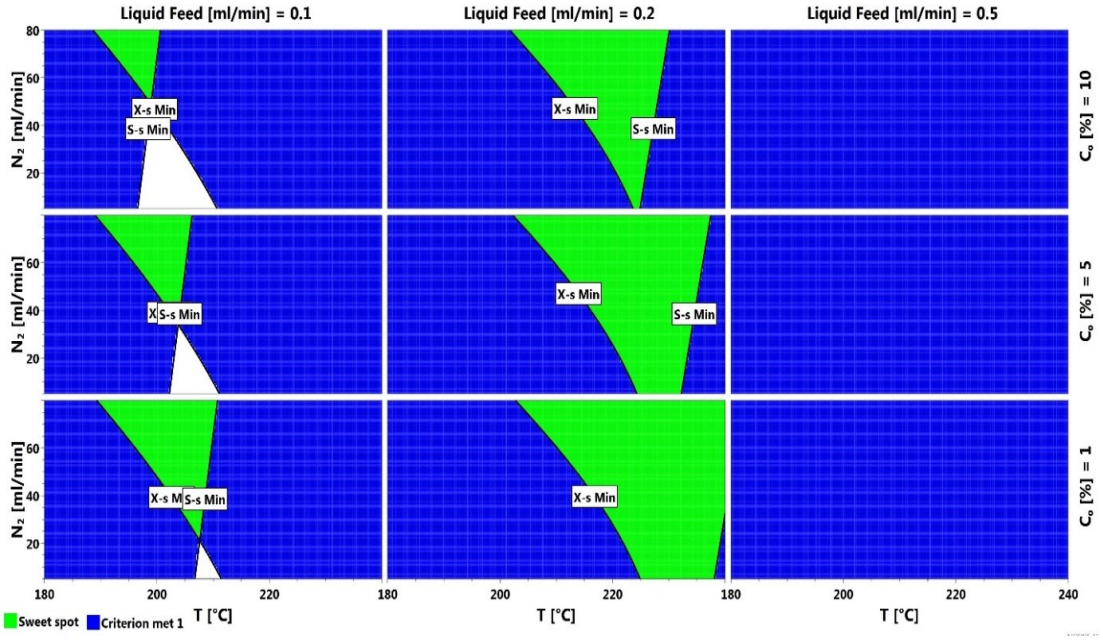

Fig. S12. Sweet spot plot with targets of > 40% conversion and > 99% selectivity with varying operating conditions. T= 180 – 240 °C, Liquid feed flow = 0.1 – 0.5 mL/min (0.7 – 3.3 $m^3\ m^{-2}\ h^{-1}$ at STP), N_2 flow = 5 – 85 mL/min (33.3 – 602.1 $m^3\ m^{-2}\ h^{-1}$ at STP), and C_o=1 wt%, 5 wt%, 10 wt%.

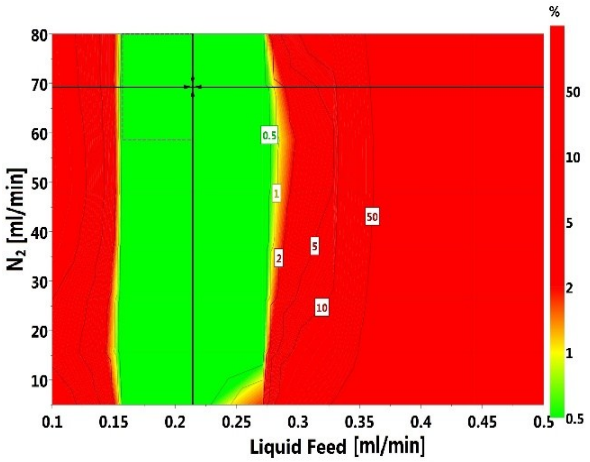

Fig. S13. Design Space for the conversion of 1,4-cyclohexanedione as a function of N_2 flow and liquid feed flow. The green area is the operating window to meet the desired specification and red area is showing risk of failure.

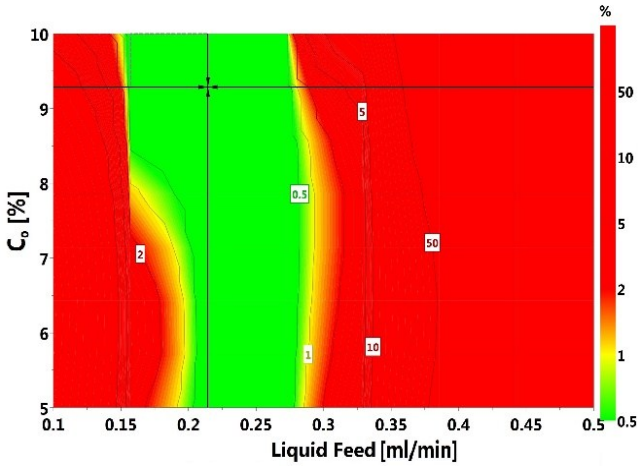

Fig. S14. Design Space for the conversion of 1,4-cyclohexanedione as a function of substrate concentration and liquid feed flow. The green area is the operating window to meet the desired specification and red area is showing risk of failure.

Table S6. Influence of intermediate gas (nitrogen and hydrogen) on activity and selectivity for the dehydrogenation of 1,4-cyclohexanedionein two-channels-in-series in continuous-mode without hydrogen removal (Data of Fig. 6a)

Time on stream	**Conversion**	**Selectivity**	**Conversion**	**Time on stream**	**Conversion**	**Selectivity**	**Conversion**
h	**%**	**%**	**%**	**h**	**%**	**%**	**%**
	Channel 1	**Channel 1**	**Model predicted - Channel 1**		**Channel 2**	**Channel 2**	**Model predicted - Channel 2**
0.5	62.7	99.1	55.4	0.5	75.7	99.0	76.1
1	61.1	99.3	55.4	1	79.2	99.1	76.1
1.5	61.4	99.1	55.4	1.5	79.0	99.3	76.1
2	59.3	99.1	55.4	2	79.4	99.4	76.1
2.5	57.7	99.4	55.4	2.5	78.8	99.1	76.1
3	58.7	99.7	55.4	3.5	78.6	99.2	76.1
3.5	60.2	99.3	55.4	4.5	78.7	99.1	76.1
4	60.2	99.4	55.4	5	78.5	99.7	76.1
4.5	58.6	99.6	55.4	6	76.9	99.5	76.1
5	59.6	99.7	55.4	7	77.9	99.1	76.1
5.5	58.5	99.3	55.4	7.5	78.4	99.7	76.1
6.5	58.1	99.7	55.4	--	--	--	--
7	61.9	99.5	55.4	--	--	--	--
7.5	59.7	99.2	55.4	--	--	--	--

Table S7. Influence of intermediate gas (nitrogen and hydrogen) on activity and selectivity for the dehydrogenation of 1,4-cyclohexanedionein two-channels-in-series in continuous-mode without hydrogen removal (Data of Fig. 6b)

Time on stream	**Conversion**	**Selectivity**	**Conversion**	**Time on stream**	**Conversion**	**Selectivity**	**Conversion**
h	**%**	**%**	**%**	**h**	**%**	**%**	**%**
	Channel 1	**Channel 1**	**Model predicted - Channel 1**		**Channel 2**	**Channel 2**	**Model predicted - Channel 2**
0.53	61.9	99.4	55.4	0.25	68.7	82.8	76.1
1	63.4	99.3	55.4	0.5	82.8	81.5	76.1
1.5	64.7	99.2	55.4	1	87.0	82.4	76.1
2	61.3	99.3	55.4	1.5	85.0	84.4	76.1
2.5	65.5	99.9	55.4	2	86.7	82.7	76.1
3	64.8	99.2	55.4	2.5	85.4	83.7	76.1
3.5	62.1	99.5	55.4	3	86.1	82.6	76.1
4	63.5	99.9	55.4	3.5	86.4	81.5	76.1
4.5	63.7	99.6	55.4	4	86.9	82.9	76.1
5	62.5	99.4	55.4	4.5	86.2	81.2	76.1
5.5	63.0	99.3	55.4	5	86.1	81.8	76.1
6	61.7	99.3	55.4	5.5	85.4	82.7	76.1
6.5	62.1	99.1	55.4	6	85.6	82.3	76.1
7	63.1	99.4	55.4	6.5	84.6	82.9	76.1
7.5	61.2	99.1	55.4	7	85.7	81.6	76.1
8	63.4	99.3	55.4	7.5	85.5	81.7	76.1
8.5	--	--	55.4	8	85.3	80.8	76.1

Table S8. Catalytic activity and selectivity of semi-continuous dehydrogenation (recycle mode) at robust set-point, 231.4 °C, 69.3 mL/min (464.5 $m^3 m^{-2} h^{-1}$ at STP) nitrogen flow, 0.21 mL/min (1.4 $m^3 m^{-2} h^{-1}$ at STP) liquid feed flow and 9.28 wt% substrate concentration as a function of stream time (h). (Data of Fig. 11)

Time on stream	Conversion	Selectivity	Time on stream	Conversion	Selectivity
h	%	%	h	%	%
0.17	1.4	99.3	6	95.9	99.1
0.5	11.1	99.2	6.5	96.7	99.3
1	29.7	99.7	7	97.5	99.5
1.5	48.5	99.2	7.5	98.1	99.2
2	59.3	99.5	8	98.4	99.2
2.5	69.3	99.3	8.5	98.8	99.4
3	76.8	99.2	9	99.0	99.4
3.5	82.3	99.3	9.5	99.2	99.1
4	86.4	99.1	10	99.2	99.1
4.5	89.7	99.5	10.5	99.3	99.5
5	92.4	99.4	11	99.3	99.2
5.5	94.3	99.6	11.5	99.4	99.4