

Supporting Information

Photodimerization of Maleic Anhydride in a Microreactor Without Clogging

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Typical procedure of single flow reaction: Maleic anhydride 10 g was dissolved in ethyl acetate 90 g previously. When a FEP tube of OD = 1.4 mm, ID = 0.8 mm, L = 13.9 m was used as a reactor, the solution was fed by plunger pump at the flow rate of 0.50 ml/min. and mixed with pressure regulated N₂ gas (1.0 MPa) at T connector. N₂ gas flow rate was controlled in exactly by needle valve as the liquid segments length became 2 cm and gas segments length became 0.5 cm. In the condition, the liner velocity of the N₂ segment was 2.13 cm/sec. and the residence time was calculated to 10.9 min. After two hours operation, 55.46 g of the suspension was collected. The suspension was condensed under reducing pressure to remove volatile materials, and then cooled to room temperature. After filtration and drying in a vacuum drying oven (60°C), CBTA 2.15 g was obtained. The conversion was determined by the weight of CBTA to be 39 %. The data of using another size of tubes were given in from Table S-1 to Table S-6.

Table S-1. The flow reaction using a FEP tube size of O.D. 1.0 mm I.D. 0.5 mm Length 19.2 m (2 h.)

flow rate (ml / min)	liner velocity (cm / sec.)	residence time (min.)	solution (g)	CBTA (g)	conversion (%)	efficiency (W / mmol)
0.40	5.89	5.40	43.9	1.42	32	55.24
0.20	2.63	11.8	22.3	1.20	54	65.37
0.10		clogging				

Table S-2. The flow reaction using a FEP tube size of O.D. 1.4 mm I.D. 0.8 mm Length 13.9 m (2 h.)

flow rate (ml / min)	liner velocity (cm / sec.)	residence time (min.)	solution (g)	CBTA (g)	conversion (%)	efficiency (W / mmol)
0.50	2.13	10.9	55.5	2.15	39	36.49
0.40	1.69	13.7	44.5	2.08	47	37.71
0.20	1.06	21.8	22.1	1.54	70	50.94

Table S-3. The flow reaction using a FEP tube size of O.D. 1.4 mm I.D. 1.0 mm Length 13.6 m (2 h.)

flow rate (ml / min)	liner velocity (cm / sec.)	residence time (min.)	solution (g)	CBTA (g)	conversion (%)	efficiency (W / mmol)
0.70	1.69	13.4	78.1	2.58	33	30.41
0.35	0.98	23.1	38.8	2.03	52	38.64

Table S-4. The flow reaction using a FEP tube size of O.D. 1.6 mm I.D. 1.0 mm Length 11.2 m (2 h.)

flow rate (ml / min)	liner velocity (cm / sec.)	residence time (min.)	solution (g)	CBTA (g)	conversion (%)	efficiency (W /mmol)
0.70	1.69	11.0	77.7	2.20	28	35.66
0.50	1.20	15.5	55.7	2.31	42	33.96
0.40	0.99	18.9	43.6	2.25	52	34.86
0.30	0.80	23.1	29.6	1.80	61	43.58

Table S-5. The flow reaction using a FEP tube size of O.D. 1.6 mm I.D. 1.2 mm Length 11.2 m (2 h.)

flow rate (ml / min)	liner velocity (cm / sec.)	residence time (min.)	solution (g)	CBTA (g)	conversion (%)	efficiency (W /mmol)
1.00	1.69	11.0	110.9	2.60	23	30.17
0.70	0.92	20.2	56.1	2.69	48	29.16

Table S-6. The flow reaction using a FEP tube size of O.D. 3.2 mm I.D. 1.6 mm Length 6.4 m (2 h.)

flow rate (ml / min)	liner velocity (cm / sec.)	residence time (min.)	solution (g)	CBTA (g)	conversion (%)	efficiency (W /mmol)
2.00	1.88	5.7	222.9	2.80	13	28.02
1.00	0.96	11.1	110.8	2.70	24	29.05
0.50	0.50	21.3	54.7	2.48	45	31.63

SEM analysis: The particle size of CBTA synthesized with the tube size of tube I.D. = 0.8 mm (residence time: 18.1 min.) was analyzed by SEM (JEOL : JSM-7401F Nisso Chemical Analysis Service Co.,Ltd). Small hexagonal plate particle (14 μ m at long axis) was observed. Particle size distribution has not analyzed, but various particle sizes were observed by SEM.



