

Electronic Supplementary Information

Practice of Function-Oriented Synthesis: High-Efficiency CO₂ Conversion and Knoevenagel Condensation by Two Novel In₃-based MOFs with High-Density Active Sites under Mild Conditions

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S1. Synthesis of H₃BTCTBA ligand.

Synthesis of 4,4',4''-[1,3,5-benzenetriyltris(carbonylimino)]trisbenzoic acid (H₃BTCTBA)

H₃BTCTBA ligand was synthesized according to a literature with slight modifications.¹ 4-aminobenzoic acid (6.0 g, 23.4 mmol) and K₂CO₃ (13.0 g, 92 mmol) were dissolved in 120 mL dry acetone and stirred under N₂ atmosphere at room temperature for 15 min. A solution of 1,3,5-benzenetricarbonylchloride (12.6 g, 92 mmol) in 60 mL acetone was added slowly in 10 min. The mixture was stirred at 65 °C overnight. After cooling, the resultant precipitate was separated by filtration, washed with water (200 mL) and acetone (30 mL), and then dried at 60 °C under vacuum to obtain H₃BTCTBA as white solid.

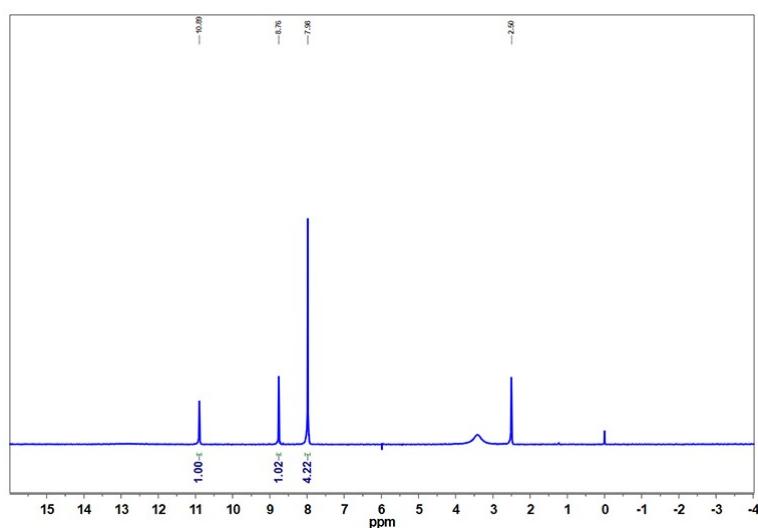


Fig. S1. ¹H NMR spectrum of H₃BTCTBA.

S2. Crystallographic data and structures of JLU-MOF116 and 117.

Table S1. Crystal data and structure refinement of **JLU-MOF116**.

Compound	JLU-MOF116
Formula	C ₄₆ H ₂₆ In ₃ N ₄ O ₂₀
Formula Weight	1299.17
Temperature (K)	293(2)
Crystal System	Tetragonal
Space Group	P4 ₂ /mnm
a (Å)	30.241(4)
b (Å)	30.241(4)
c (Å)	18.297(4)
α (°)	90
β (°)	90
γ (°)	90
V (Å ³)	16734(6)
Z, ρ _{calc} (g/cm ³)	4, 0.516
μ (mm ⁻¹)	0.434
F(000)	2548.0
2θ Range for Data Collection (°)	5.984 to 54.962
Reflections Collected	153985
Independent Reflections	10112 [R _{int} = 0.1321, R _{sigma} = 0.0526]
Data/Restraints/Parameters	10112/275/254
Goodness-of-fit on F ²	1.016
Final R Indexes [I ≥ 2σ (I)]	R ₁ ^a = 0.0542, wR ₂ ^b = 0.1543
Final R Indexes [all data]	R ₁ ^a = 0.0779, wR ₂ ^b = 0.1648

^a R₁ = Σ ||F_o|| - |F_c|| / Σ |F_o|, ^b wR₂ = [Σ w(|F_o|² - |F_c|²) / Σ |w(F_o)²|] ^{1/2}

Table S2. Crystal data and structure refinement of **JLU-MOF117**.

Compound	JLU-MOF117
Formula	C ₆₀ H ₃₀ In ₃ N ₆ O ₂₂
Formula Weight	1531.36
Temperature (K)	302.16
Crystal System	Orthorhombic
Space Group	<i>Imma</i>
a (Å)	20.1206(4)
b (Å)	28.9071(7)
c (Å)	29.2586(7)
α (°)	90
β (°)	90
γ (°)	90
V (Å ³)	17017.6(7)
Z, ρ _{calc} (g/cm ³)	4, 0.598
μ (mm ⁻¹)	0.433
F(000)	3020.0
2θ Range for Data Collection (°)	4.508 to 50.700
Reflections Collected	33311
Independent Reflections	8133 [R _{int} = 0.0851, R _{sigma} = 0.0989]
Data/Restraints/Parameters	8133/20/263
Goodness-of-fit on F ²	0.947
Final R Indexes [I ≥ 2σ (I)]	R ₁ ^a = 0.0411, wR ₂ ^b = 0.1065
Final R Indexes [all data]	R ₁ ^a = 0.0602, wR ₂ ^b = 0.1132

^a R₁ = Σ | |F_o| - |F_c| | / Σ |F_o|, ^b wR₂ = [Σw(|F_o|² - |F_c|²) / Σ |w(F_o)²|] ^{1/2}

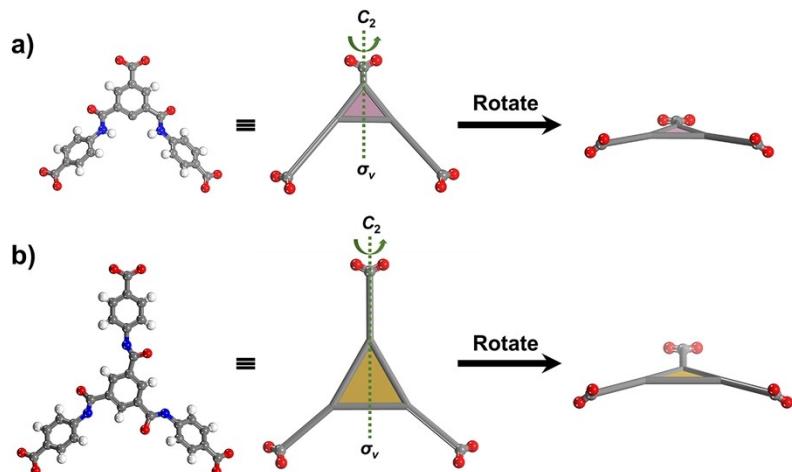


Fig. S2. Molecular symmetries of BCPACBA and BTCTBA.

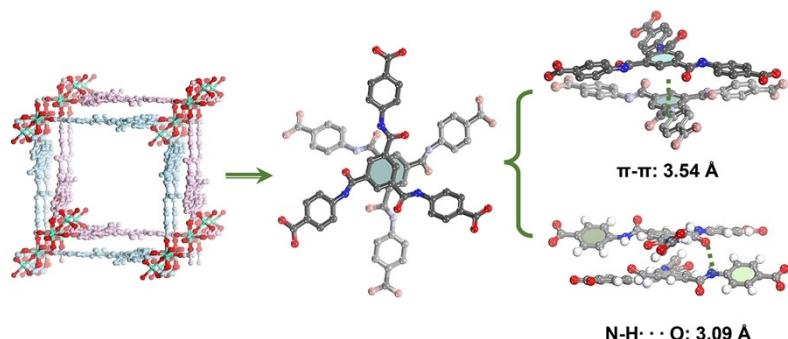


Fig. S3. The 2-fold interpenetration framework of **JLU-MOF117** showing N-H···O hydrogen-bond interactions and π - π interactions between the two adjacent BTCTBA ligands.

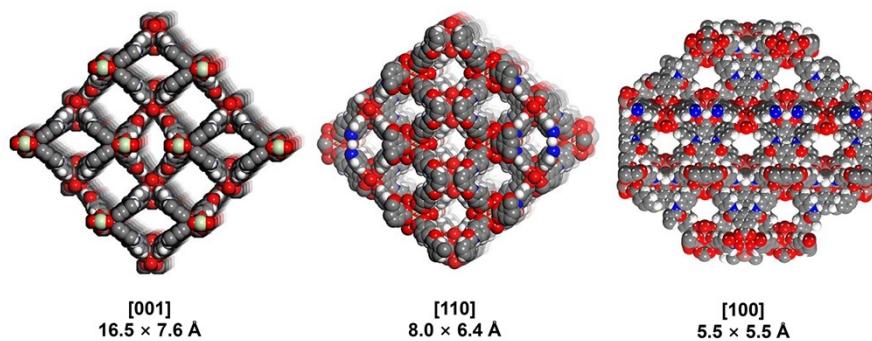


Fig. S4. CPK models of **JLU-MOF116** framework along the [001], [110], and [100] directions.

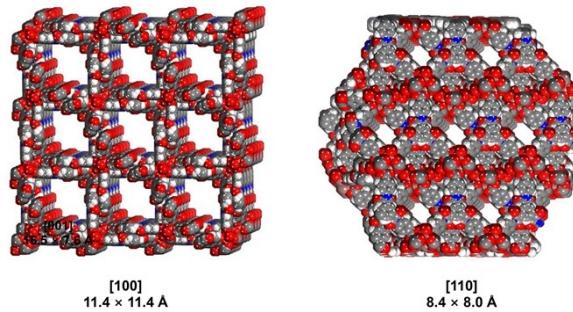


Fig. S5. CPK models of **JLU-MOF117** framework along the [100] and [110] directions.

S3. PXRD patterns, TGA analyses and N₂ sorption isotherms of JLU-MOF116 and 117.

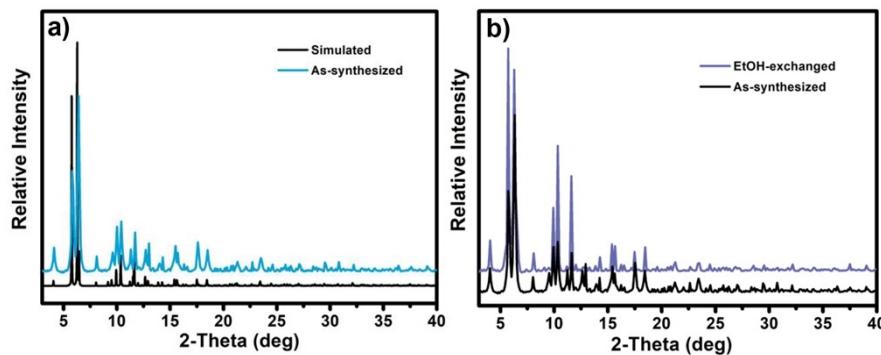


Fig. S6. PXRD patterns of a) simulated, as-synthesized, and b) EtOH-exchanged **JLU-MOF116**.

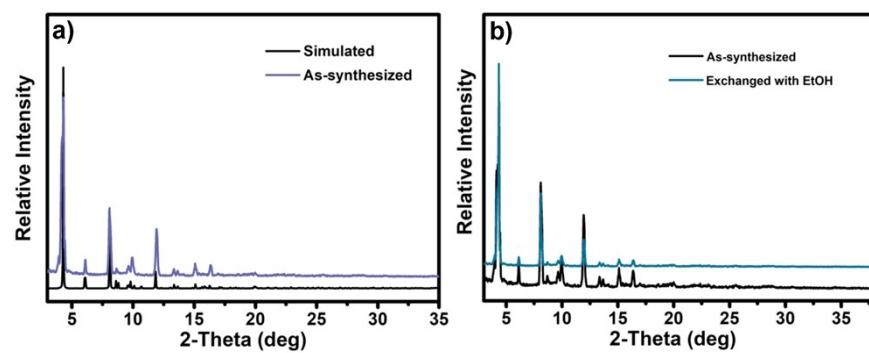


Fig. S7. PXRD patterns of a) simulated, as-synthesized, and b) EtOH-exchanged **JLU-MOF117**.

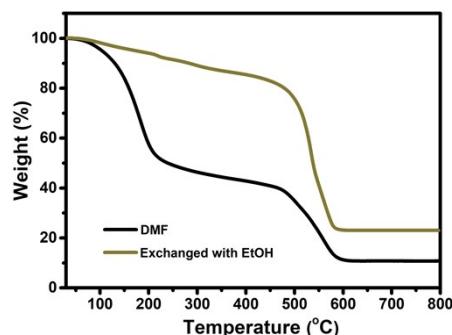


Fig. S8. TGA curves of the as-synthesized and ethanol-exchanged **JLU-MOF116**.

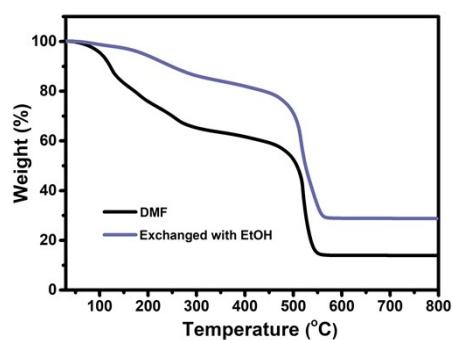


Fig. S9. TGA curves of the as-synthesized and ethanol-exchanged **JLU-MOF117**.

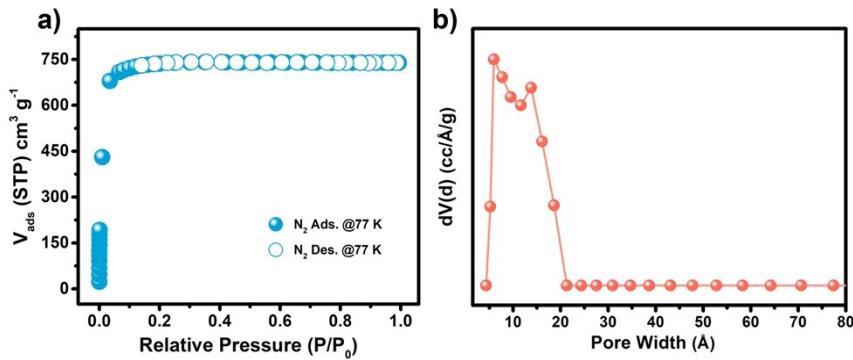


Fig. S10. a) N_2 adsorption-desorption isotherm and b) pore size distribution of **JLU-MOF116**.

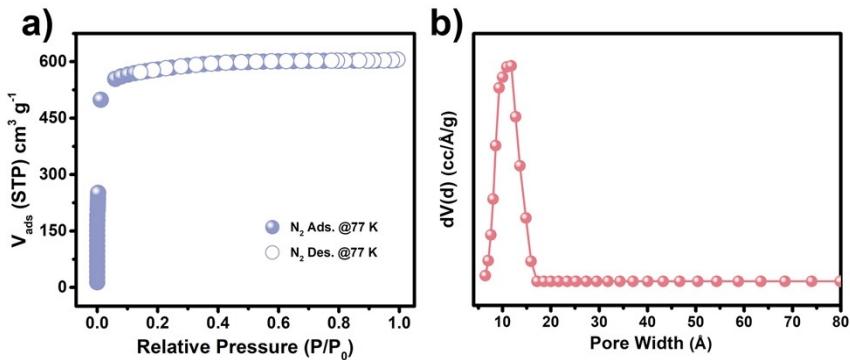


Fig. S11. a) N_2 adsorption-desorption isotherm and b) pore size distribution of **JLU-MOF117**.

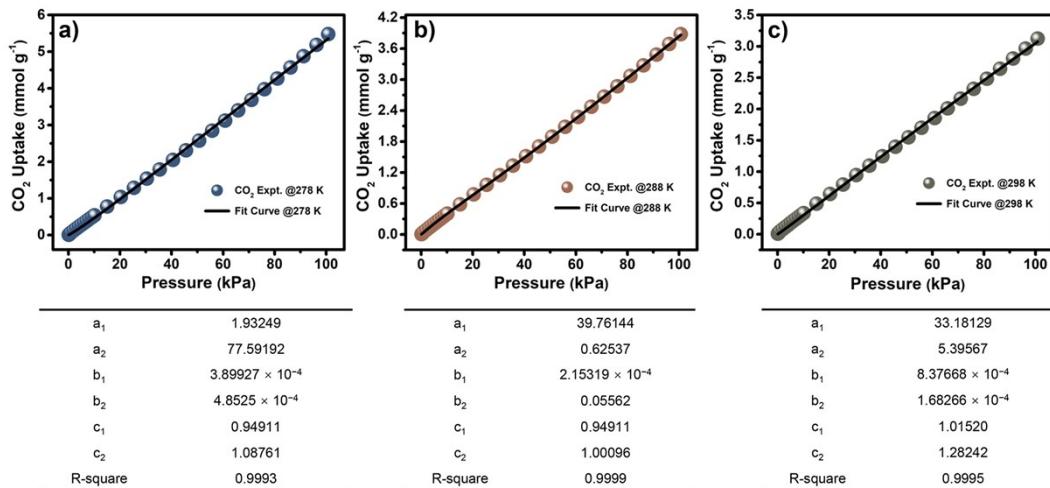


Fig. S12. Fitting curves and parameters of CO_2 adsorption isotherms of **JLU-MOF116** at a) 278, b) 288, and c) 298 K.

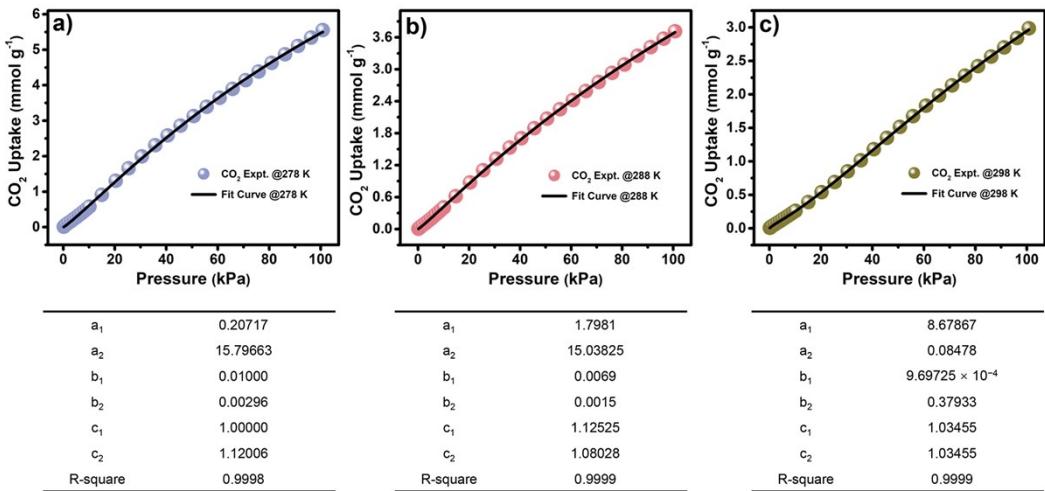


Fig. S13. Fitting curves and parameters of CO₂ adsorption isotherms of **JLU-MOF117** at a) 278, b) 288, and c) 298 K.

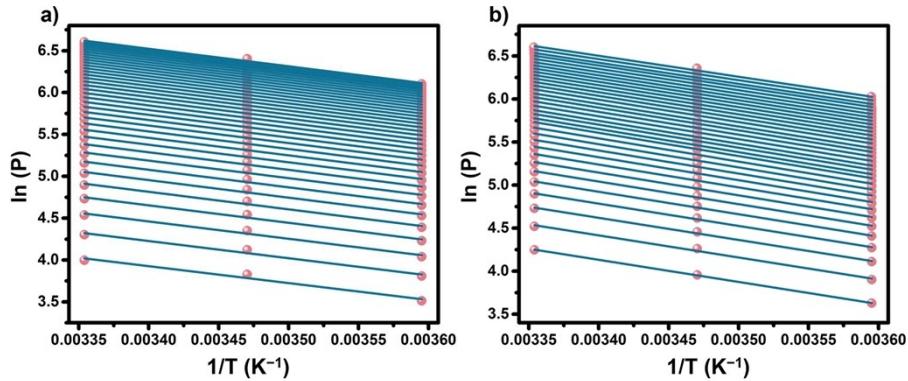


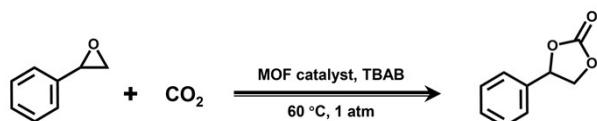
Fig. S14. The isosters of Q_{st} values for a) **JLU-MOF116** and b) **JLU-MOF117**.

S4. CO₂ conversions with epoxides catalyzed by JLU-MOF116 and JLU-MOF117.

Table S3. Comparisons of volumetric densities of amide groups in JLU-MOF116, 117, and other amide-based MOF materials.

Compound	Volumetric density of amide group (mol L ⁻¹)	Ref.
3W-ROD-1	2.5	2
sph-MOF-1	2.4	3
JLU-MOF117	2.4	This work
M ₂ (carboxylate) ₄	2.1	4
JLU-MOF116	1.6	This Work
DUT-32	0.9	5

Table S4. The investigation of optimal conditions for CO₂ conversion by **JLU-MOF116** and **JLU-MOF117**.



Entry ^a	JLU-MOF116				Entry ^a	JLU-MOF117			
	Cat. (mol%)	TBAB (mol%)	Time (h)	Yield (%)		Cat. (mol%)	TBAB (mol%)	Time (h)	Yield (%)
1	0.075	5	8	59	14	0.05	5	6	38
2	0.15	5	8	> 99	15	0.1	5	6	94
3	0.3	5	8	> 99	16	0.2	5	6	97
4	0.15	2	8	95	17	0.1	2	6	86
5	0.15	8	8	> 99	18	0.1	8	6	> 99
6	0.15	5	1	51	19	0.1	5	1	47
7	0.15	5	2	69	20	0.1	5	2	59
8	0.15	5	3	91	21	0.1	5	3	72
9	0.15	5	4	96	22	0.1	5	4	86
10	0.15	5	5	97	23	0.1	5	5	92
11	0.15	5	6	99	24	0.1	5	8	99
12	None	5	6	32	25	None	5	6	32
13 ^b	0.15	5	6	41	26 ^b	0.15	5	6	39

Reaction conditions: ^a Activated MOF catalysts, styrene oxide (10 mmol) and 1 atm CO₂ pressure.

^b As-synthesized MOF catalysts.

In order to determine the optimal reaction condition, a sequence of **JLU-MOF116**-catalyzed CO₂ conversion reactions with styrene oxide was performed at 60 °C and 1 atm CO₂ pressure. The amount of MOF catalyst was first explored within a prolonged reaction time and an appropriate TBAB amount of 5 mol%. CO₂ conversion with styrene oxide catalyzed by 0.075 mol% **JLU-MOF116** resulted in only 59% yield while gained nearly 100% yields by 0.15 and 0.3 mol% catalysts (Table S4, Entry 1-3). Accordingly, the appropriate catalyst amount was determined as 0.15 mol%. As an efficient nucleophile, the amount of TBAB co-catalyst also has an impact on CO₂ conversion. The yield was 95% and 99%, when 2 mol% and 5 mol% TBAB engaged in the reaction, respectively (Table S4, Entry 4 and 11). Thereby, the amount of TBAB was determined as 5 mol%. The optimal catalyst amounts were investigated in the same manner to be 0.1 mol% **JLU-MOF117** and 5 mol% TBAB (Table S4, Entry 15-19), which is also easy for comparison with **JLU-MOF116**. The kinetic curves of the CO₂ conversion reactions by **JLU-MOF116** and **117** were further investigated. The CO₂ conversions have already gained 51% and 47% yields for **JLU-MOF116** and **117** within the initial 1 h, respectively, reached up to 97% and 94% yields within the next 4 hours for **JLU-MOF116** and 5 hours for **JLU-MOF117**, respectively, and achieved equilibriums thereafter (Table S4, Entry 6-11, 20-25).

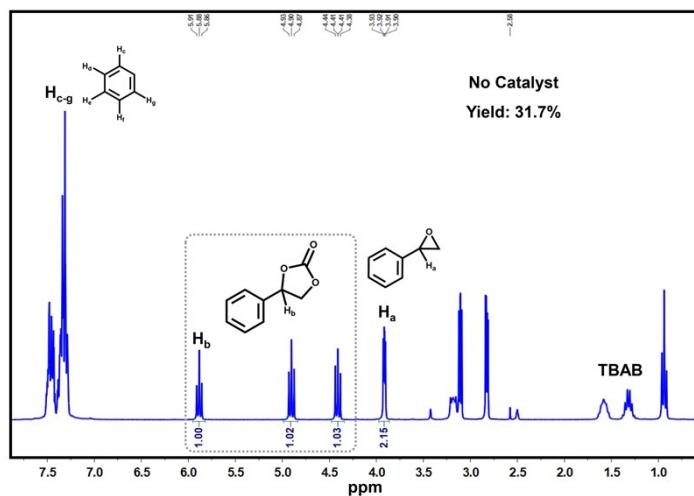


Fig. S15. ¹H NMR spectrum of CO₂ conversion with styrene oxide without MOF catalyst.

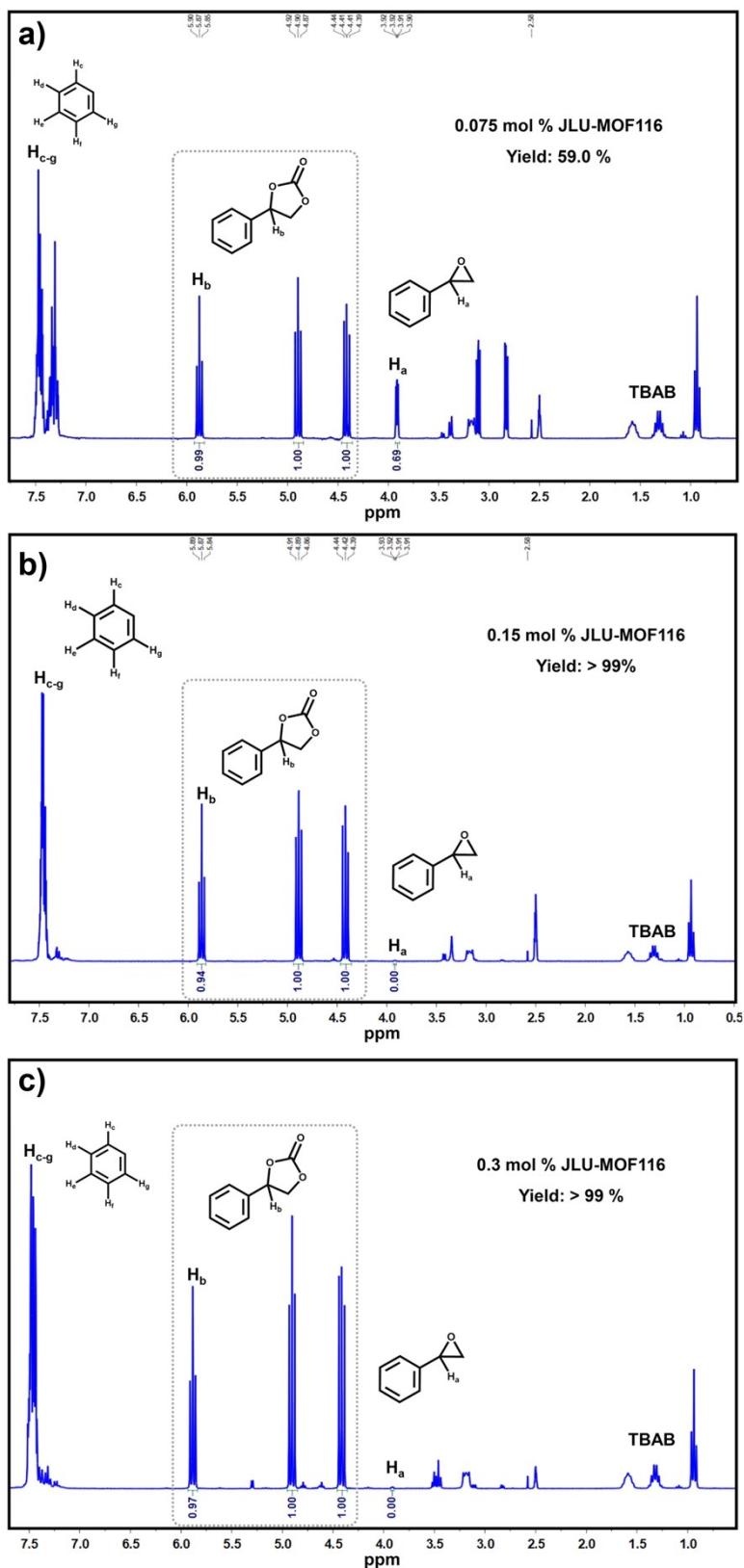


Fig. S16. ^1H NMR spectra of CO_2 conversion catalyzed by a) 0.075 mol%, b) 0.15 mol%, and c) 0.3 mol% **JLU-MOF116**.

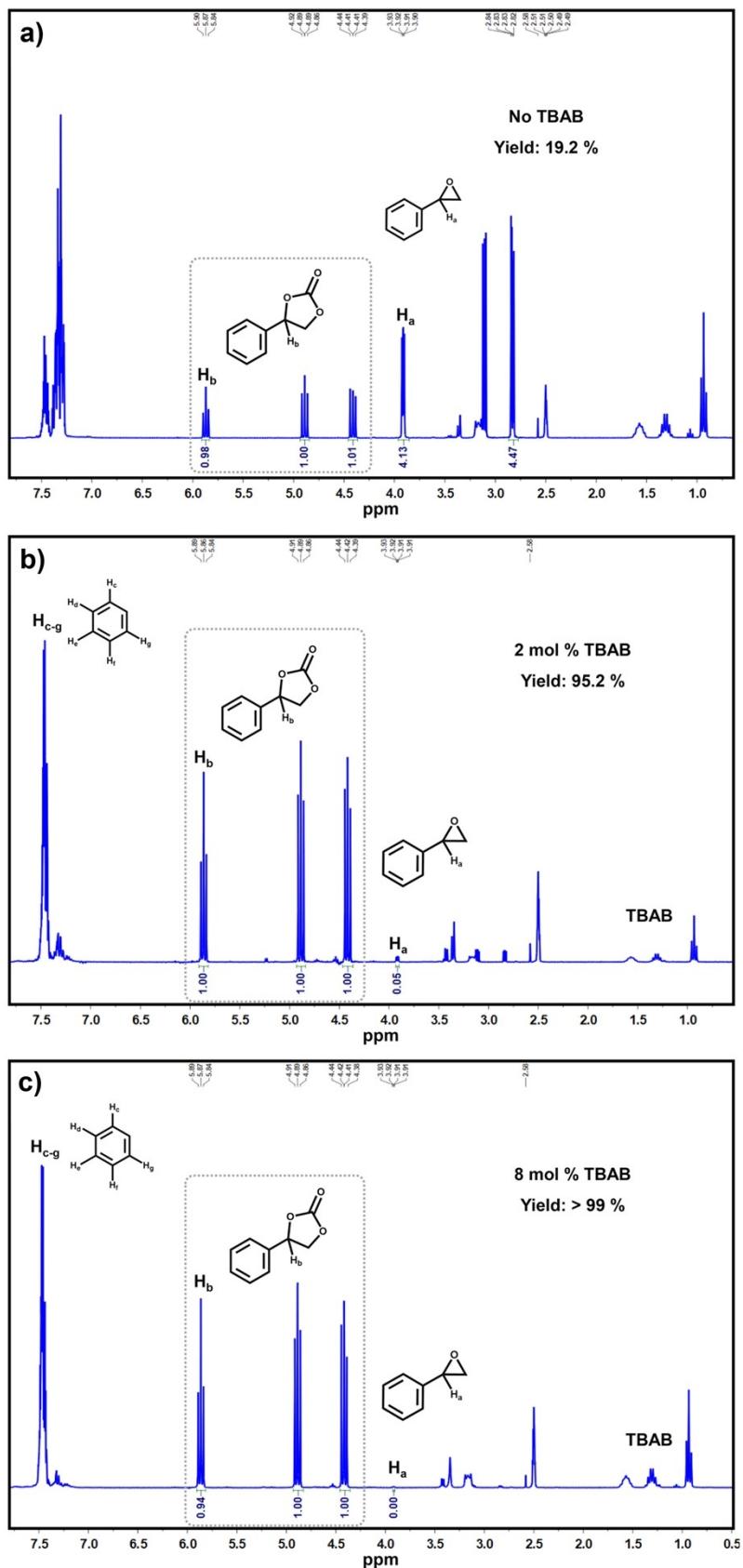


Fig. S17. ^1H NMR spectra of CO_2 conversion catalyzed by a) 0, b) 2 mol%, and c) 8 mol% TBAB and **JLU-MOF116**.

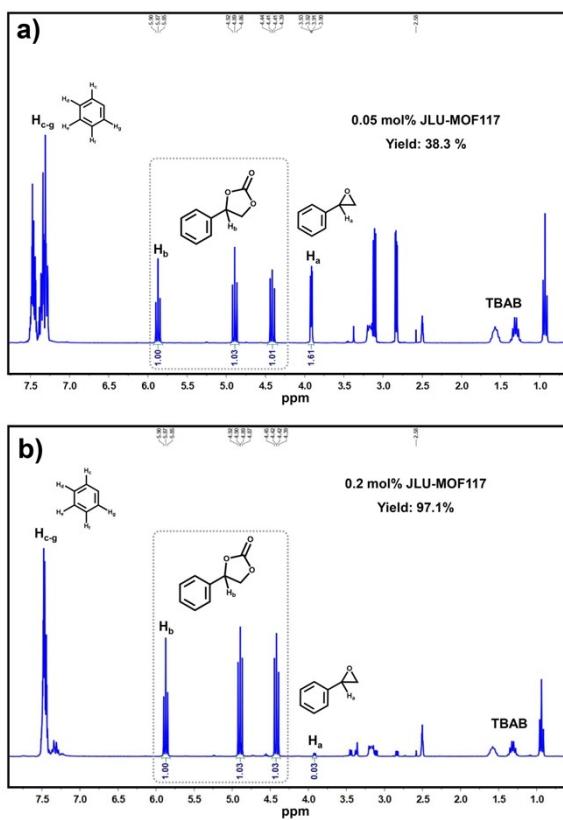


Fig. S18. ^1H NMR spectra of CO_2 conversion catalyzed by a) 0.05 mol% and b) 0.2 mol% **JLU-MOF117**.

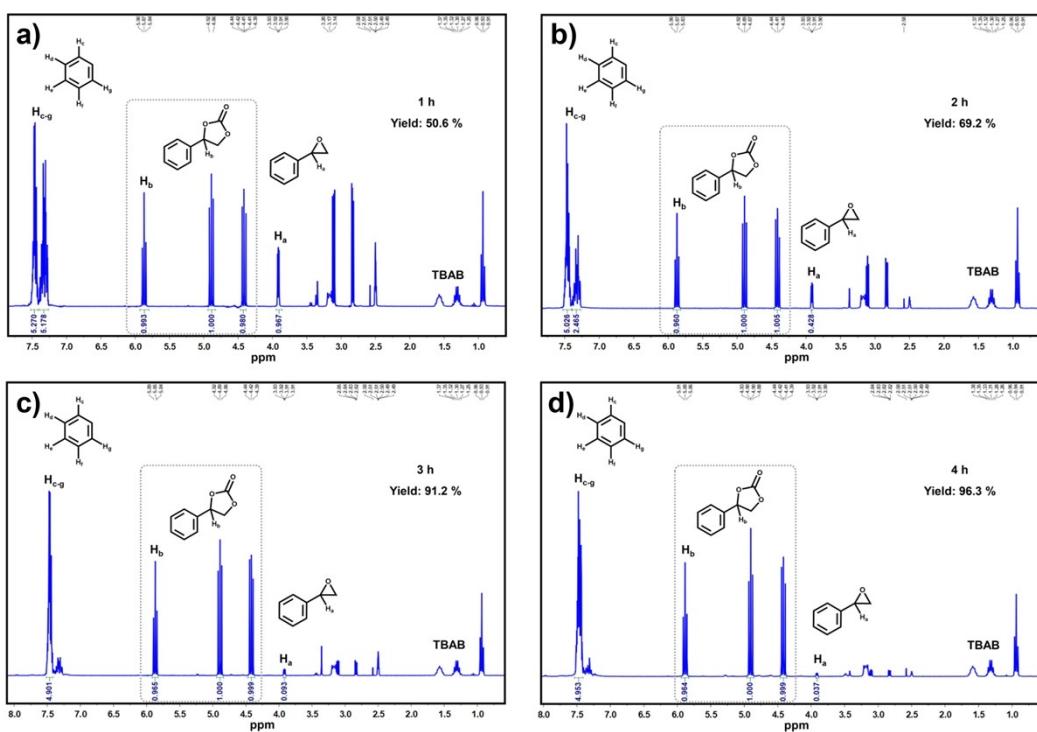


Fig. S19. ^1H NMR spectra of CO_2 conversion catalyzed by **JLU-MOF116** within a) 1 h, b) 2 h, c) 3 h, and d) 4 h.

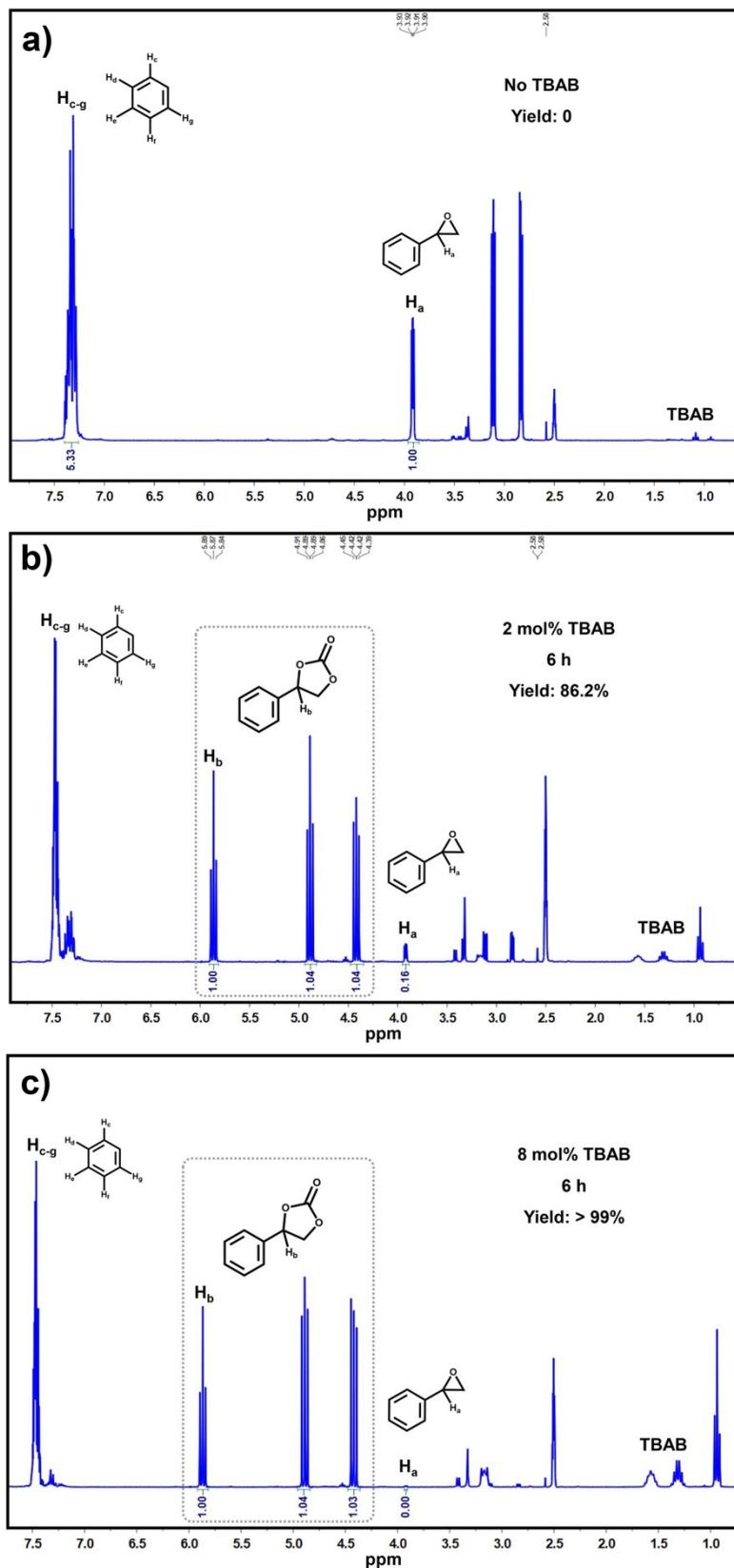


Fig. S20. ^1H NMR spectra of CO_2 conversion catalyzed by a) 0, b) 2 mol%, and c) 8 mol% TBAB and **JLU-MOF117**.

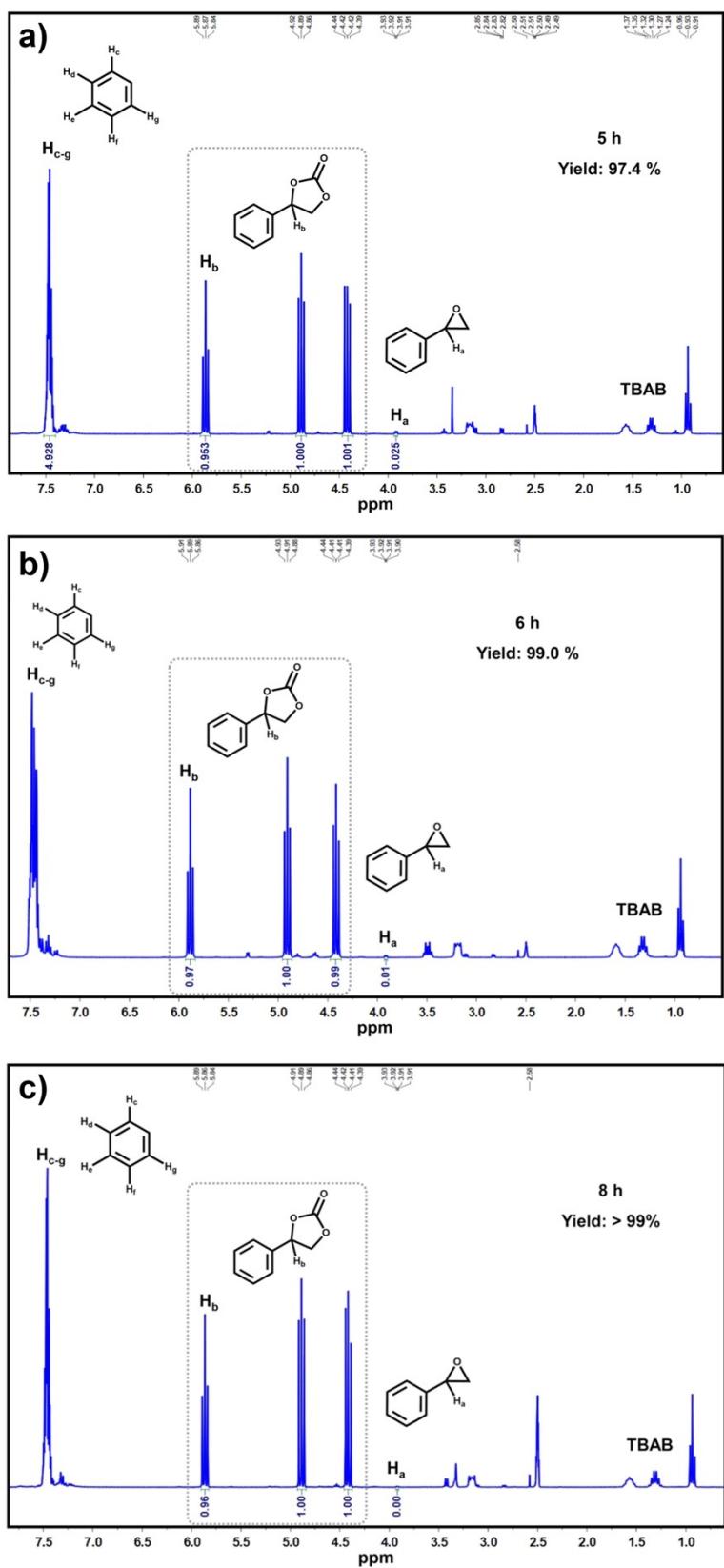


Fig. S21. ^1H NMR spectra of CO_2 conversion catalyzed by JLU-MOF116 within a) 5 h, b) 6 h, and c) 8 h.

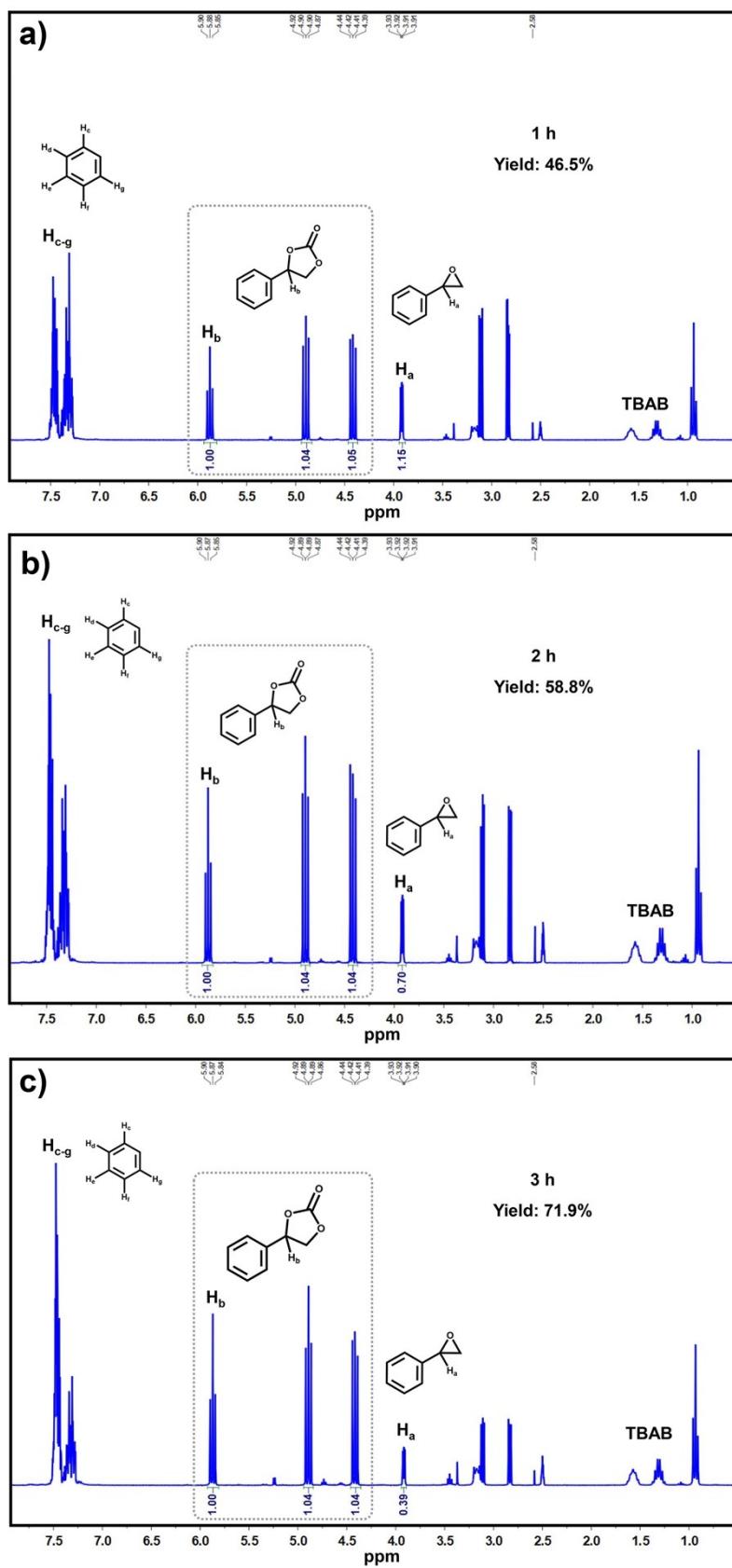


Fig. S22. ¹H NMR spectra of CO₂ conversion catalyzed by **JLU-MOF117** within a) 1 h, b) 2 h, and c) 3 h.

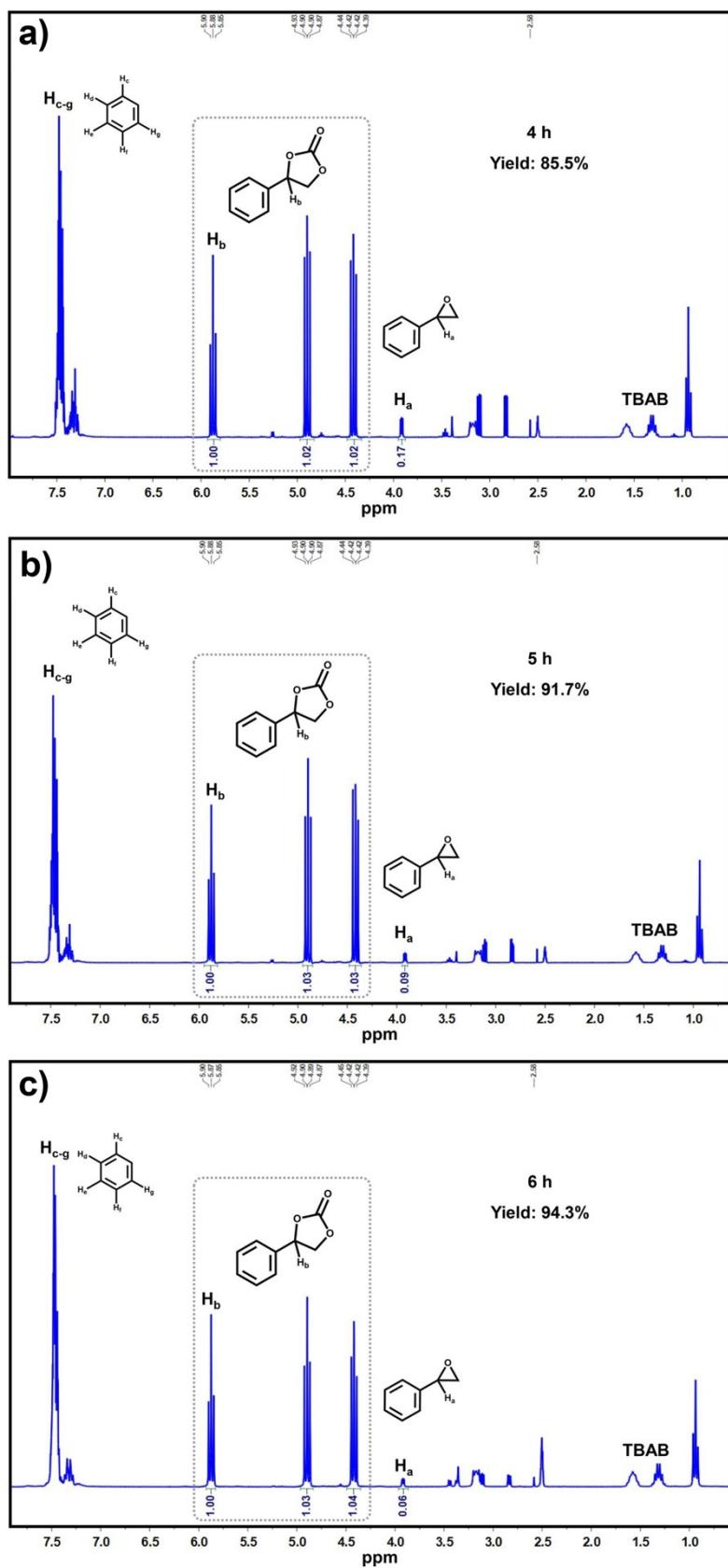


Fig. S23. ¹H NMR spectra of CO₂ conversion catalyzed by **JLU-MOF116** within a) 4 h, b) 5 h, and c) 6 h.

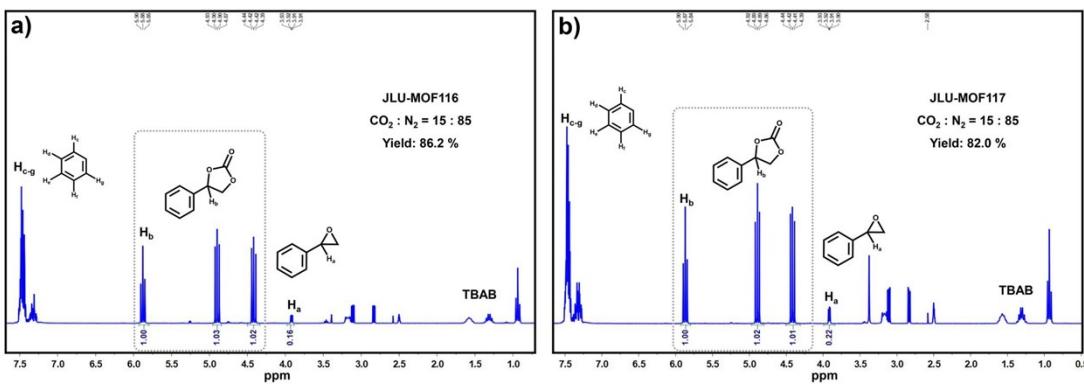


Fig. S24. CO_2 conversions by **JLU-MOF16** and **117** under 1 atm of CO_2 and N_2 mixed gases (CO_2/N_2 , 15/85, v/v).

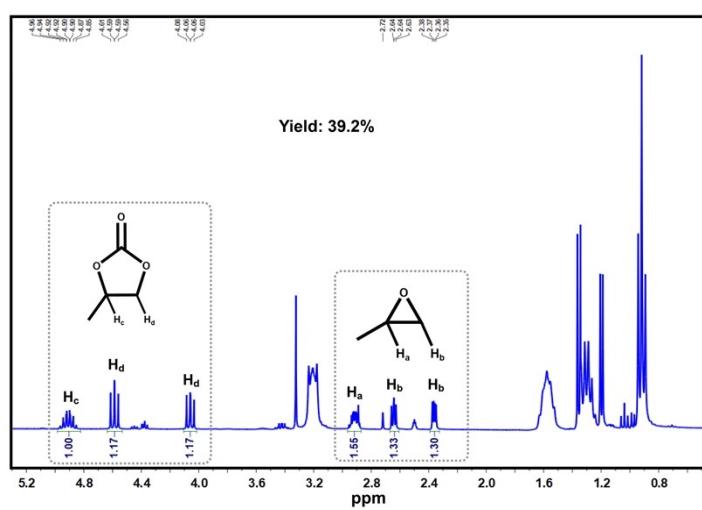


Fig. S25. ¹H NMR spectrum of CO_2 conversion with propylene oxide catalyzed by **JLU-MOF16**.

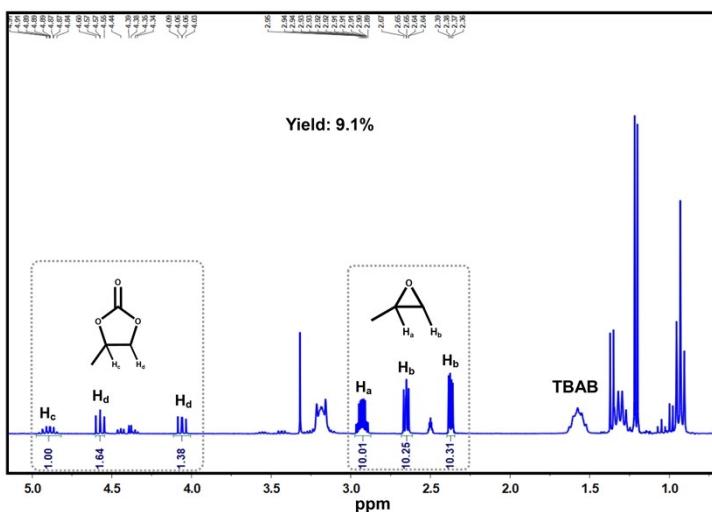


Fig. S26. ¹H NMR spectrum of CO_2 conversion with propylene oxide catalyzed by **JLU-MOF117**.

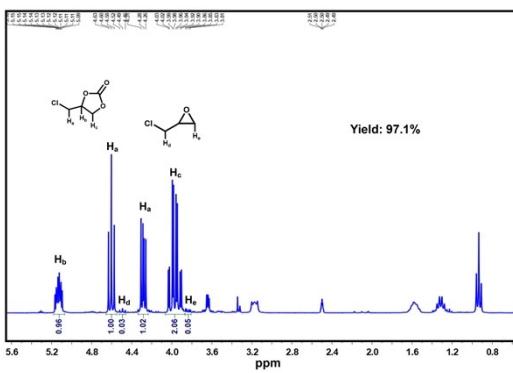


Fig. S27. ¹H NMR spectrum of CO₂ conversion with epoxy chloropropane catalyzed by **JLU-MOF16**.

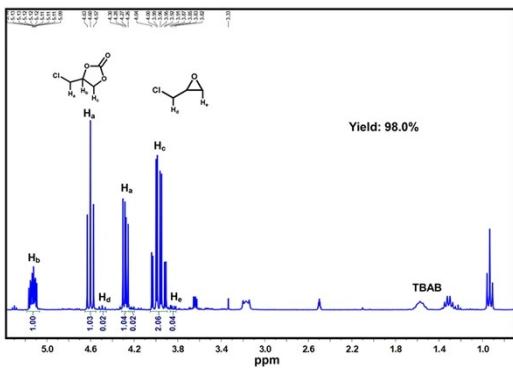


Fig. S28. ¹H NMR spectrum of CO₂ conversion with epoxy chloropropane catalyzed by **JLU-MOF17**.

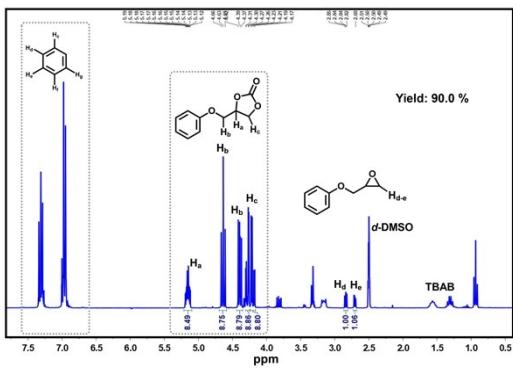


Fig. S29. ¹H NMR spectrum of CO₂ conversion with epoxypropyl phenyl ether catalyzed by **JLU-MOF16**.

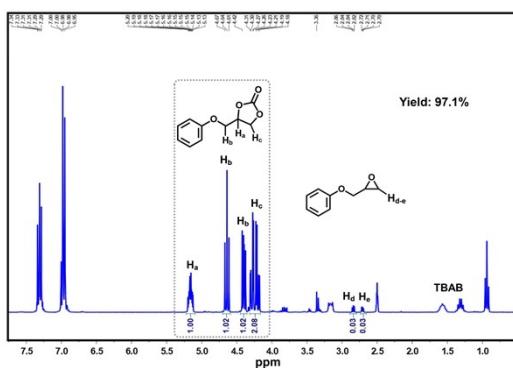


Fig. S30. ¹H NMR spectrum of CO₂ conversion with epoxypropyl phenyl ether catalyzed by **JLU-MOF17**.

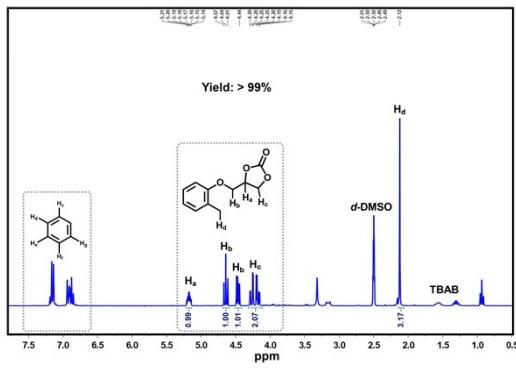


Fig. S31. ¹H NMR spectrum of CO₂ conversion with o-tolyl glycidyl ether catalyzed by **JLU-MOF116**.

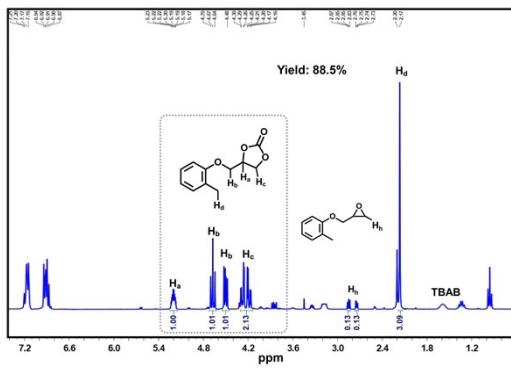


Fig. S32. ¹H NMR spectrum of CO₂ conversion with o-tolyl glycidyl ether catalyzed by **JLU-MOF117**.

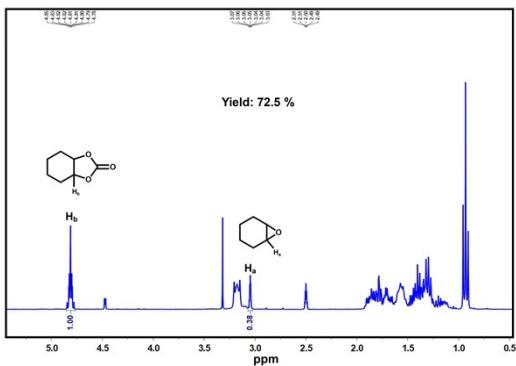


Fig. S33. ¹H NMR spectrum of CO₂ conversion with cyclohexene oxide catalyzed by **JLU-MOF116**.

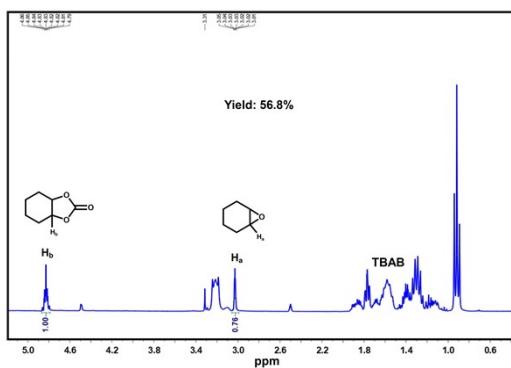


Fig. S34. ¹H NMR spectrum of CO₂ conversion with cyclohexene oxide catalyzed by **JLU-MOF117**.

Table S5. CO₂ cycloaddition catalytic efficiencies of **JLU-MOF116** and **117** in comparison with other MOF materials.

Substrate	Compound ^a	Time (h)	Yield (%)	TON	TOF (h ⁻¹)	Ref.
SO	Ce ₂ NDC ₃	8	89	360	45.0	6
	MOF1	24	11	65	15.9	7
	Hf-NU-1000	56	100	400	7.1	8
	In ₂ (OH)(btc)(Hbtc) _{0.4} (L) _{0.6} ·3H ₂ O	48	32	139	2.9	9
	{[Ba ₂ (BDPO)(H ₂ O)]·DMA} _n	48	19.8	105	2.2	10
	{[Sr(BDPO) _{0.5} (H ₂ O)]·2H ₂ O} _n	48	18	21	0.4	11
	JLU-MOF117^b	6	94	940	156.7	This Work
ECH	JLU-MOF116^b	5	97	647	129.4	This Work
	Ce ₂ NDC ₃	8	92	372	46.5	6
	MOF1	24	99	582	24.3	7
	FJI-H7(Cu)	60	67	333	5.5	12
	{[Ba ₂ (BDPO)(H ₂ O)]·DMA} _n	48	90	180	3.8	10
	[Cu(bpy) ₂ (EDS)] _n	Not Mentioned	92	92		13
	JLU-MOF117^b	6	98	980	163.3	This Work
	JLU-MOF116^b	5	97	647	129.4	This Work

^a Room temperature, 1 bar CO₂ pressure; ^b 60 °C, 1 bar CO₂ pressure.

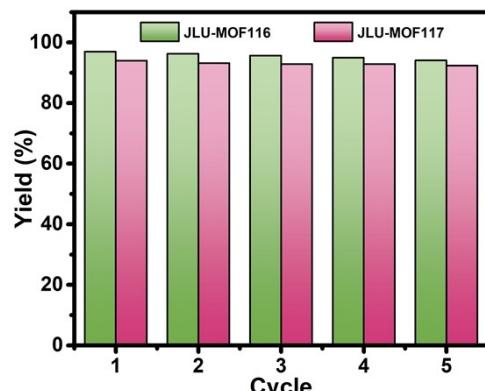


Fig. S35. The catalytic efficiencies of **JLU-MOF116** and **117** for CO₂ conversion within five cycles.

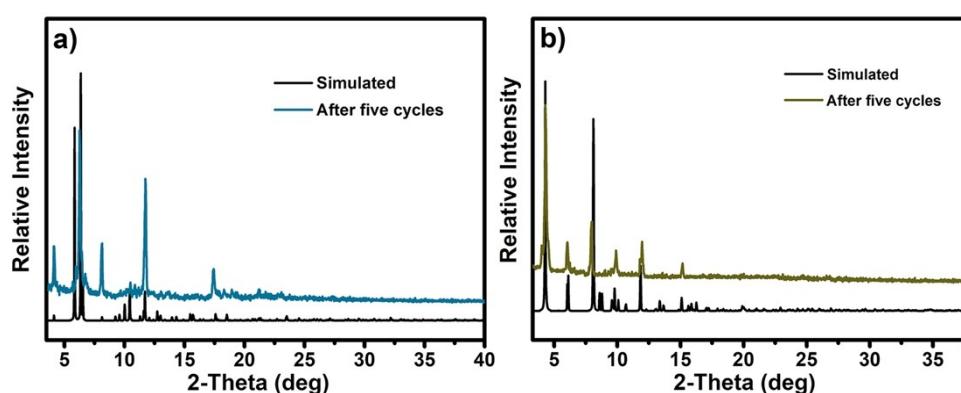
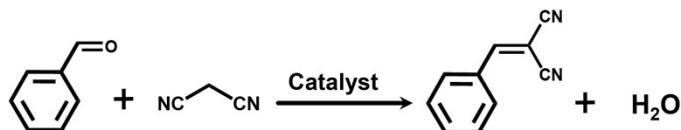


Fig. S36. PXRD patterns of a) **JLU-MOF116** and b) **JLU-MOF117** after five cycles of cycloadditions of styrene oxide with CO₂.

S5. Knoevenagel condensation reactions catalyzed by **JLU-MOF116 and **JLU-MOF117**.**

Table S6. The investigation of optimal reaction conditions of Knoevenagel condensation for **JLU-MOF116** and **JLU-MOF117**.



Entry	JLU-MOF116				Entry	JLU-MOF117			
	Cat. (mol%)	Temp. (°C)	Time (min)	Yield (%)		Cat. (mol%)	Temp. (°C)	Time (min)	Yield (%)
1	0.125	40	180	89	13	0.25	40	120	99
2	0.250	40	180	98	14	0.50	40	120	> 99
3	0.500	40	180	98	15	0.25	20	120	96
4	0.250	20	180	90	16	0.25	40	30	89
5	0.250	60	180	96	17	0.25	40	60	94
6	0.250	40	30	60	18	0.25	40	90	99
7	0.250	40	60	81	19	L2	40	90	24
8	0.250	40	90	93					
9	0.250	40	120	95					
10	0.250	40	150	97					
11	L1	40	120	3					
12	None	40	120	30	20	None	40	90	21

Reaction conditions: Benzaldehyde (2 mmol), malononitrile (3 mmol), EtOH (4 mL).

Malononitrile (3 mmol) and benzaldehyde (2 mmol) were chosen as substrates to investigate optimal conditions. The amount of MOF catalysts was investigated initially. The reactions catalyzed by 0.25 mol% and 0.5 mol% **JLU-MOF116** resulted in a 98% yield, while the reduction of **JLU-MOF116** to 0.125 mol% led to a negligible decline to an 89% yield (Table S6, Entry 1-3). Thereby, the amount of **JLU-MOF116** was determined as 0.25 mol%. **JLU-MOF117** was also determined as 0.25 mol% with a 99% yield for ease of comparison. The optimal reaction time and temperature were determined by time-varying kinetic curves under different temperatures to be 120 min/40 °C for **JLU-MOF116** and 90 min/40 °C for **JLU-MOF117**, respectively (Table S6, Entry 6-10 and 16-18, and Figure S42).

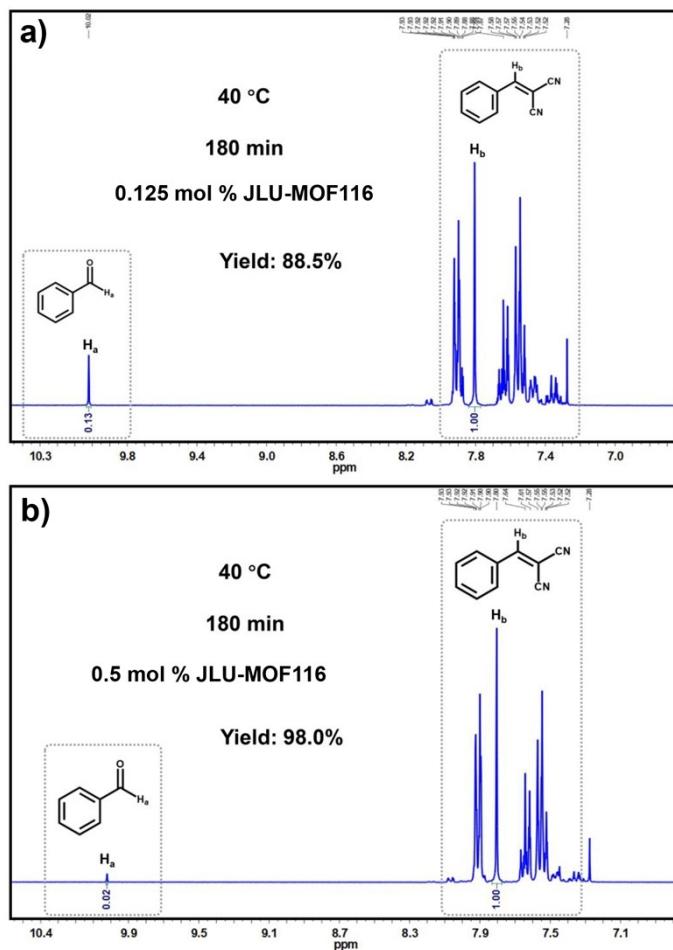


Fig. S37. ^1H NMR spectra of Knoevenagel condensation reactions catalyzed by a) 0.125 mol% and b) 0.5 mol% **JLU-MOF116**.

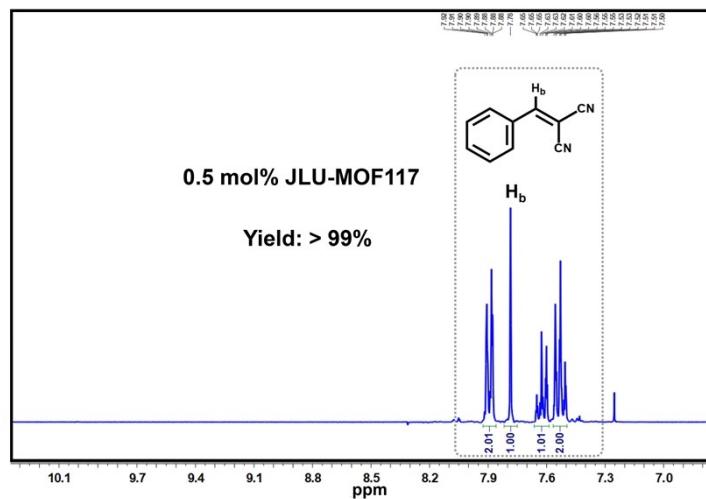


Fig. S38. ^1H NMR spectra of Knoevenagel condensation reactions catalyzed by 0.5 mol% **JLU-MOF117**.

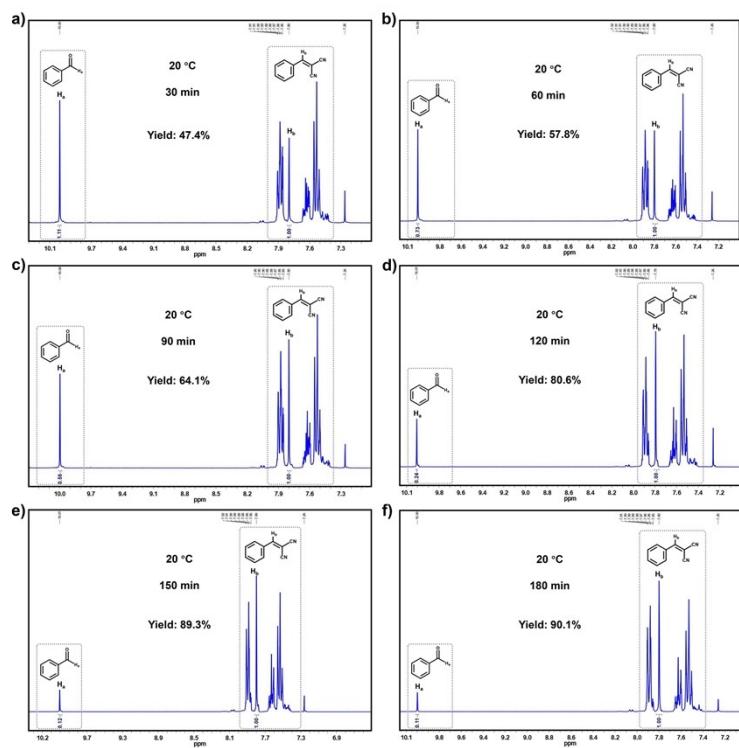


Fig. S39. ^1H NMR of Knoevenagel condensation reactions catalyzed by **JLU-MOF116** at $20\text{ }^\circ\text{C}$ within a) 30, b) 60, c) 90, d) 120, e) 150, and f) 180 min.

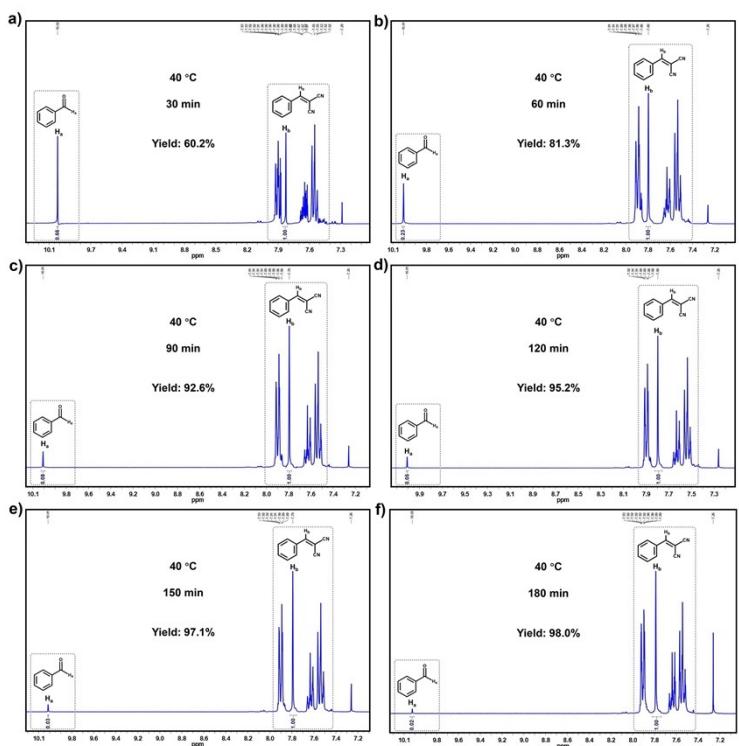


Fig. S40. ^1H NMR of Knoevenagel condensation reactions catalyzed by **JLU-MOF116** at $40\text{ }^\circ\text{C}$ within a) 30, b) 60, c) 90, d) 120, e) 150, and f) 180 min.

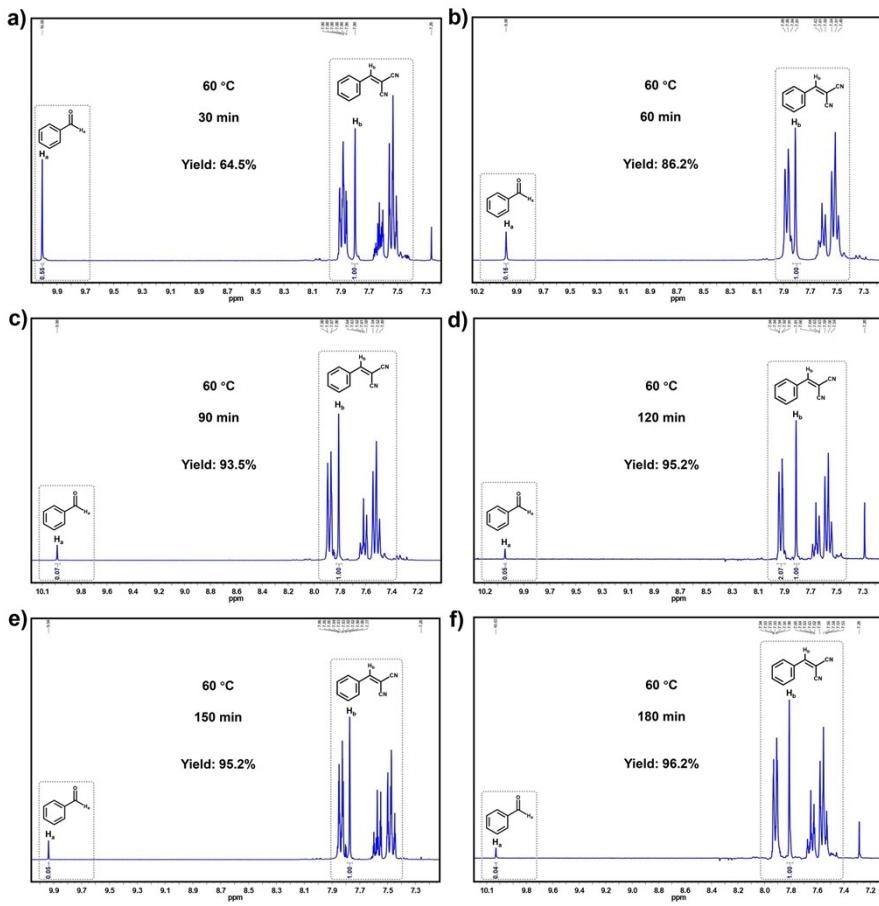


Fig. S41. ^1H NMR of Knoevenagel condensation reactions catalyzed by **JLU-MOF116** at 60 °C within a) 30, b) 60, c) 90, d) 120, e) 150, and f) 180 min.

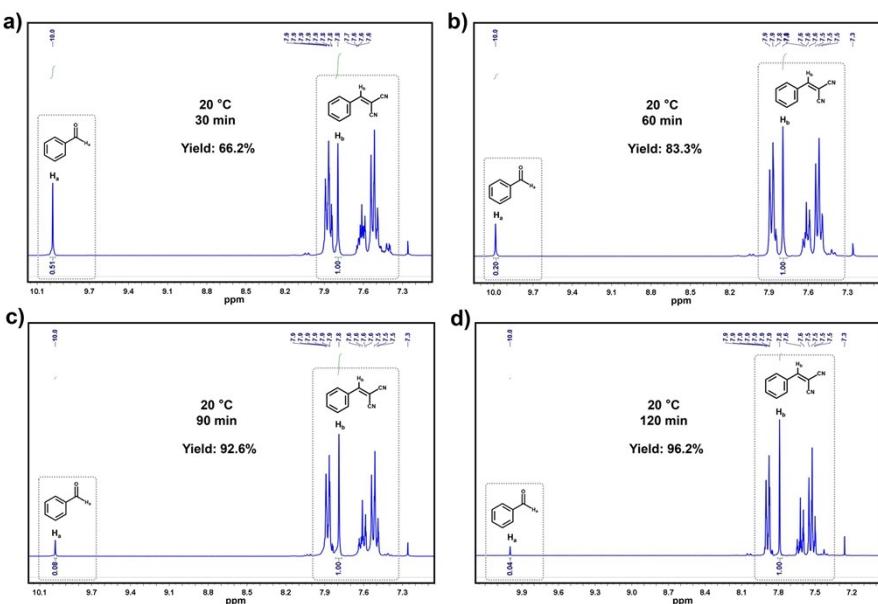


Fig. S42. ^1H NMR of Knoevenagel condensation reactions catalyzed by **JLU-MOF117** at 20 °C within a) 30, b) 60, c) 90, and d) 120 min.

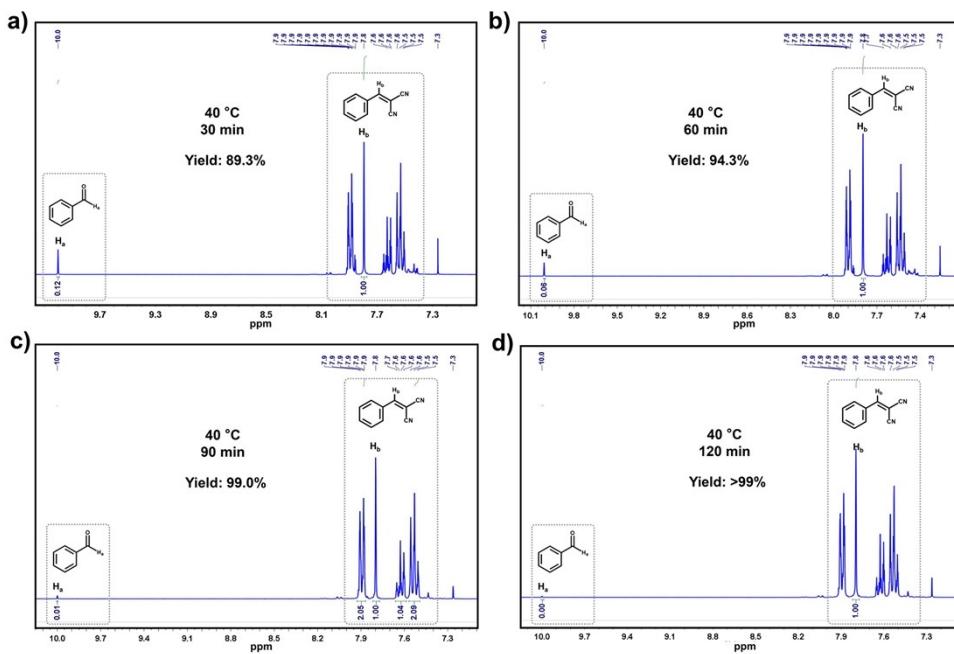


Fig. S43. ^1H NMR of Knoevenagel condensation reactions catalyzed by **JLU-MOF117** at 40 °C within a) 30, b) 60, c) 90, and d) 120 min.

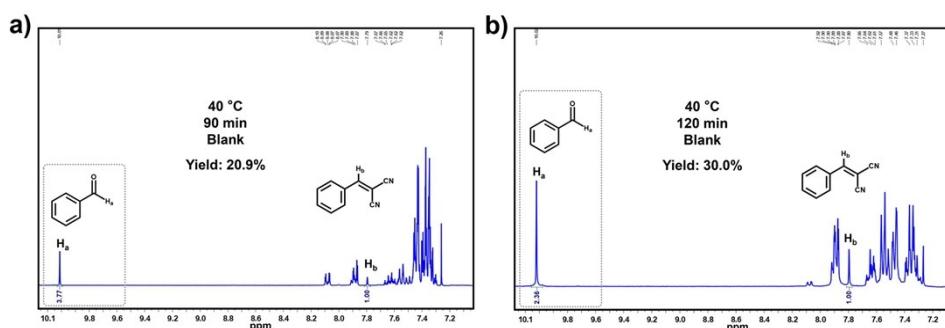


Fig. S44. Knoevenagel condensation reactions without catalyst within a) 90 min and b) 120 min.

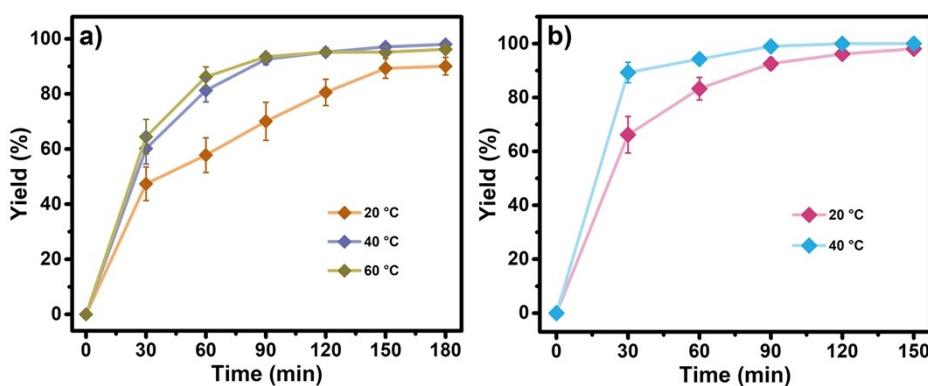


Fig. S45. Time-varying kinetic curves of a) **JLU-MOF116** and b) **JLU-MOF117** for Knoevenagel condensation.

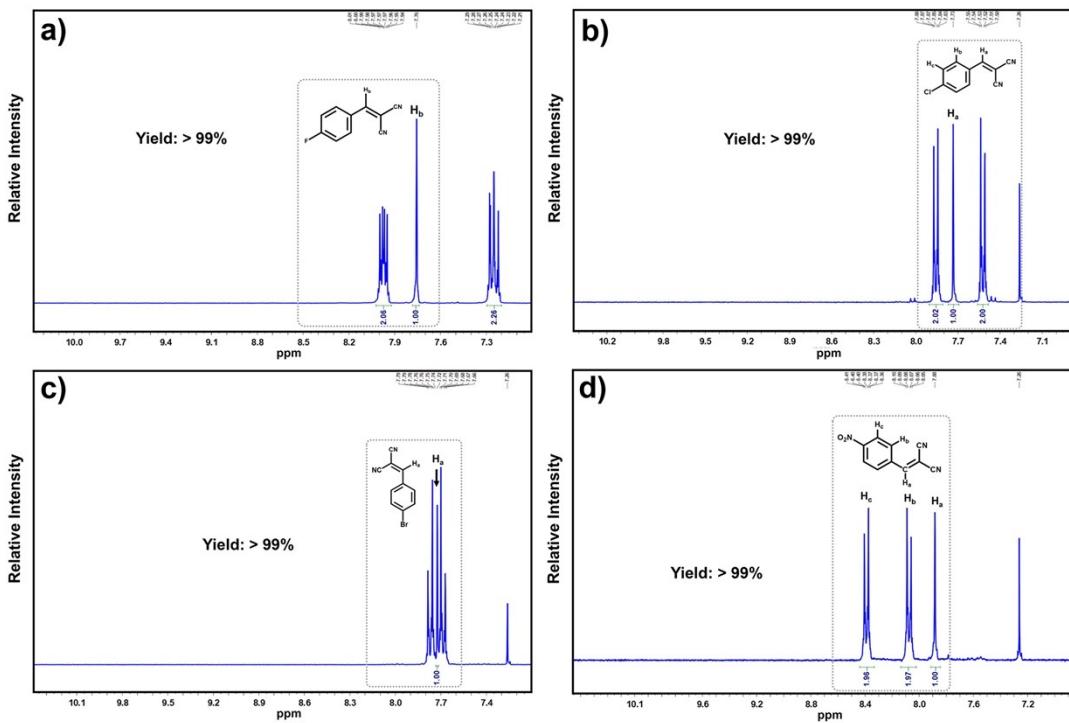


Fig. S46. ^1H NMR spectra of the Knoevenagel condensation by **JLU-MOF116** with a) 4-fluorobenzaldehyde, b) 4-chlorobenzaldehyde, c) 4-bromobenzaldehyde, and d) 4-nitrobenzaldehyde as reactants.

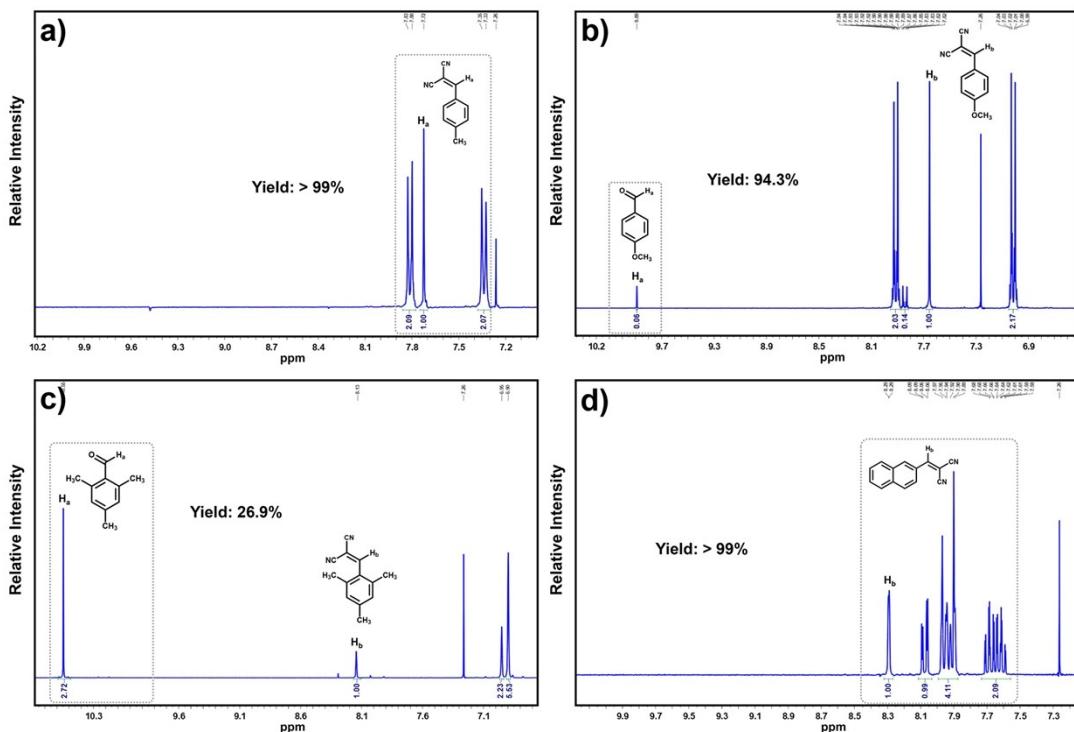


Fig. S47. ^1H NMR spectra of the Knoevenagel condensation by **JLU-MOF116** with a) 4-methylbenzaldehyde, b) 4-anisaldehyde, c) 2,4,6-trimethylbenzaldehyde, and d) 2-naphthaldehyde as reactants.

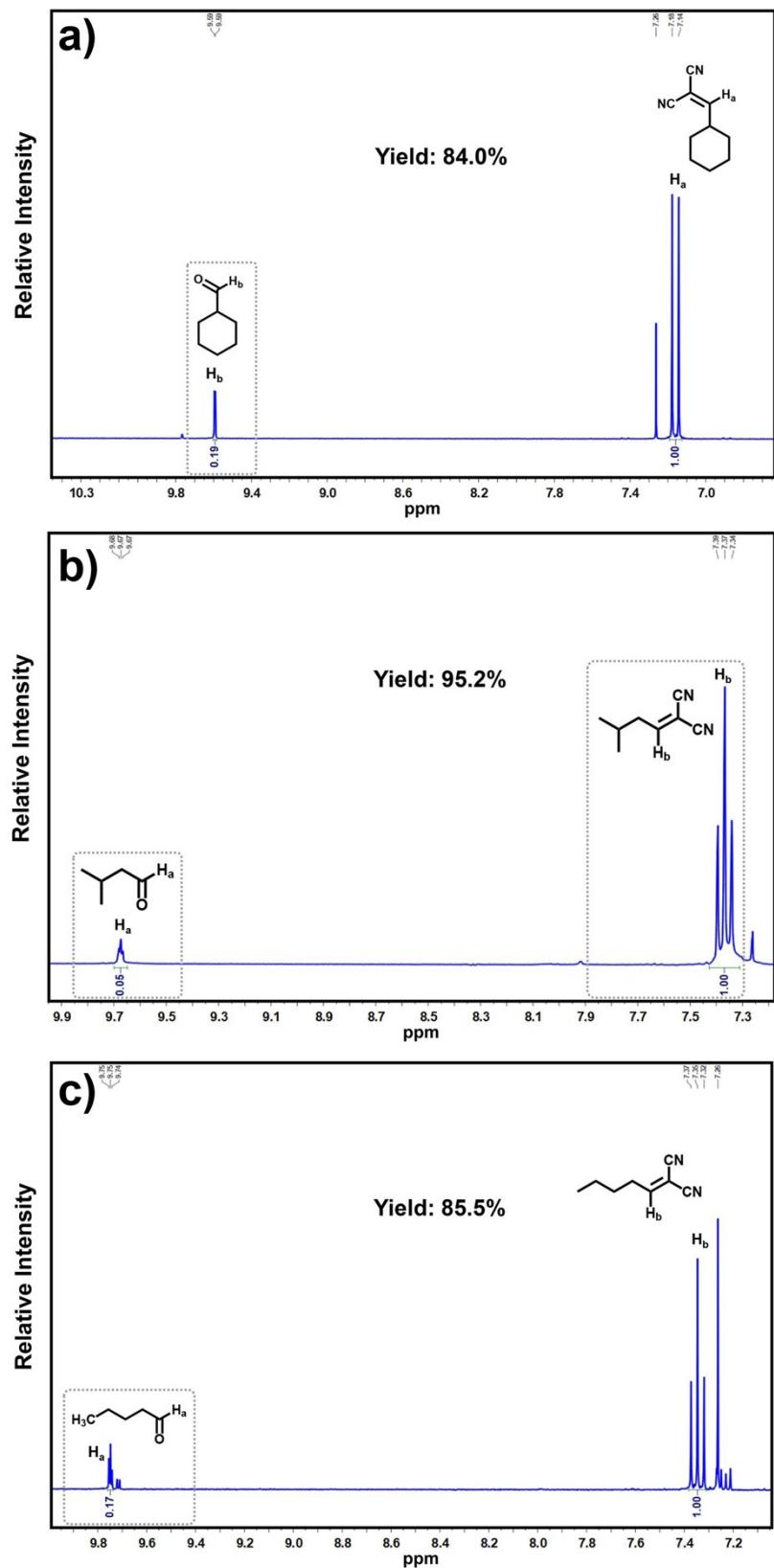


Fig. S48. ¹H NMR spectra of the Knoevenagel condensation by **JLU-MOF16** with a) cyclohexanecarboxaldehyde, b) pentanal, and c) isovaleraldehyde as reactants.

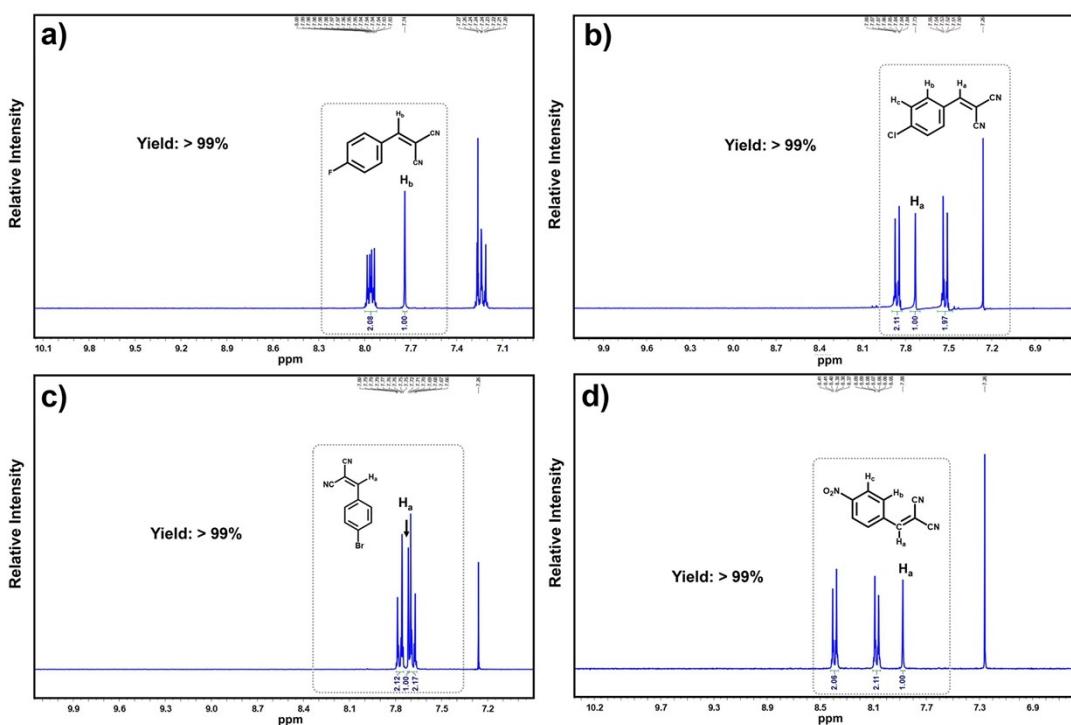


Fig. S49. ^1H NMR spectra of the Knoevenagel condensation by **JLU-MOF117** with a) 4-fluorobenzaldehyde, b) 4-chlorobenzaldehyde, c) 4-bromobenzaldehyde, and d) 4-nitrobenzaldehyde as reactants.

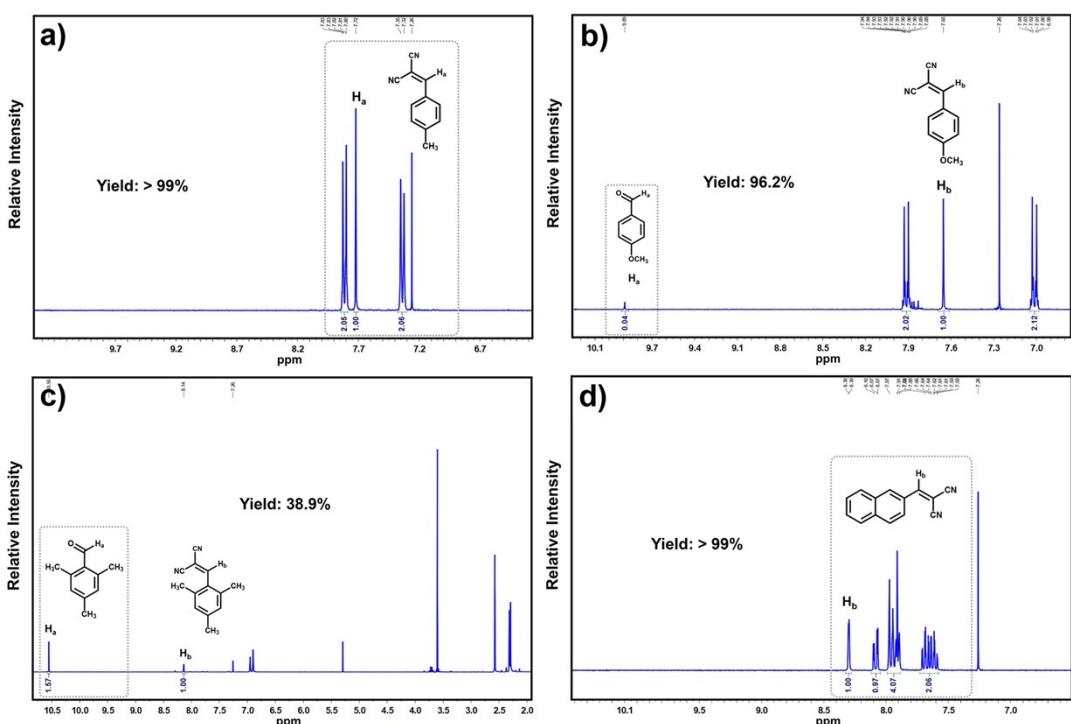


Fig. S50. ^1H NMR spectra of the Knoevenagel condensation by **JLU-MOF117** with a) 4-methylbenzaldehyde, b) 4-anisaldehyde, c) 2,4,6-trimethylbenzaldehyde, and d) 2-naphthaldehyde as reactants.

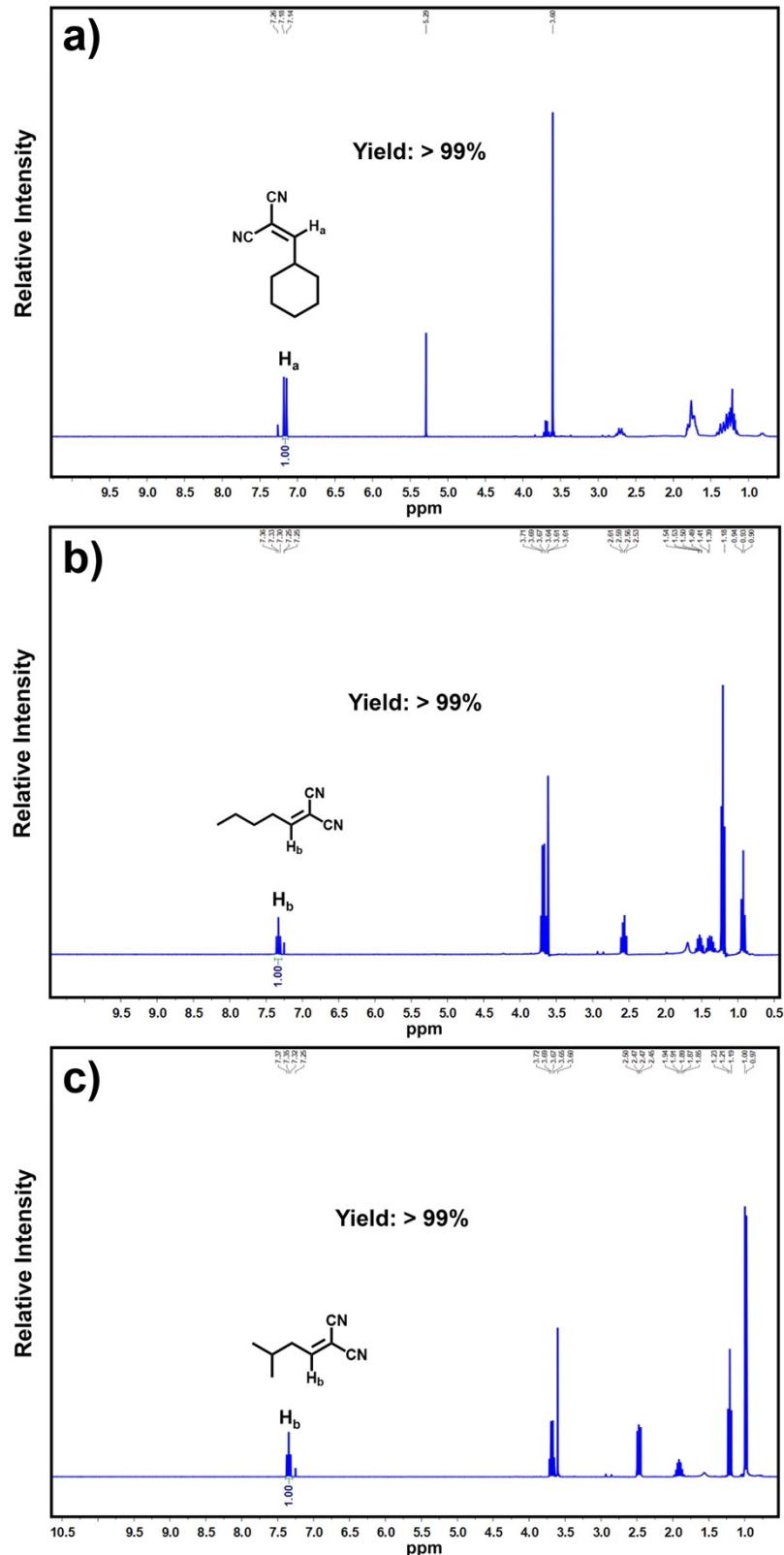


Fig. S51. ¹H NMR spectra of the Knoevenagel condensation by **JLU-MOF117** with a) cyclohexanecarboxaldehyde, b) pentanal, and c) isovaleraldehyde as reactants.

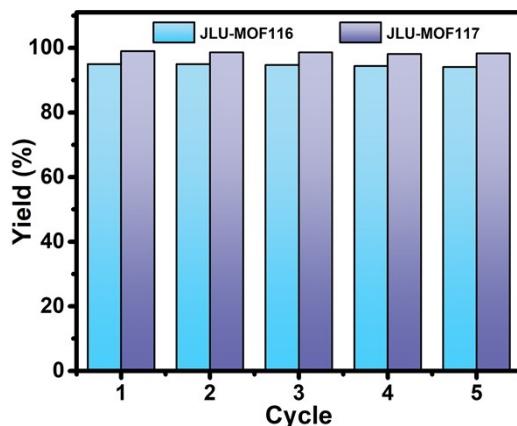


Fig. S52. The catalytic performances of **JLU-MOF116** and **117** for Knoevenagel condensation within five cycles.

Table S7. Knoevenagel condensation catalytic efficiencies of **JLU-MOF116** and **117** in comparison with other MOF materials.

Compound	Cat. (mol%)	Solvent	Temp. (°C) & Time (h)	Yield (%)	TON	TOF (min ⁻¹)	Ref.
JLU-MOF117	0.25	Ethanol	40 °C/1.5 h	99	396	4.40	This work
JLU-MOF116	0.25	Ethanol	40 °C/2 h	95	380	3.17	This work
NUC-21	0.3	Solvent free	70 °C/1 h	97	323	5.38	14
[Co ₂ (bptc)(H ₂ O) ₂]·5DMA	2	Solvent free	60 °C/6 h	> 99	50	0.14	15
MOF3	0.25	DMF	RT/6 h	97	388	1.08	16
NUC-25	0.4	Solvent free	80 °C/24 h	99	248	0.17	17
[Zn(kN-H ₃ L)(H ₂ O) ₃]·3H ₂ O	3	THF	50 °C/4 h	94	31	0.13	18
NUC-38Yb	0.3	Ethanol	45 °C/24 h	96	1280	0.89	19
NUC-28	0.3	Ethanol	45 °C/24 h	96	320	0.22	20
UiO-66-NH-RNH ₂	1	Toluene	RT/2 h	97	97	0.81	21
[Cu ₂ (μ-H ₃ ddba) ₂ (phen) ₂]	2	H ₂ O	25 °C/1 h	> 99	50	0.83	22

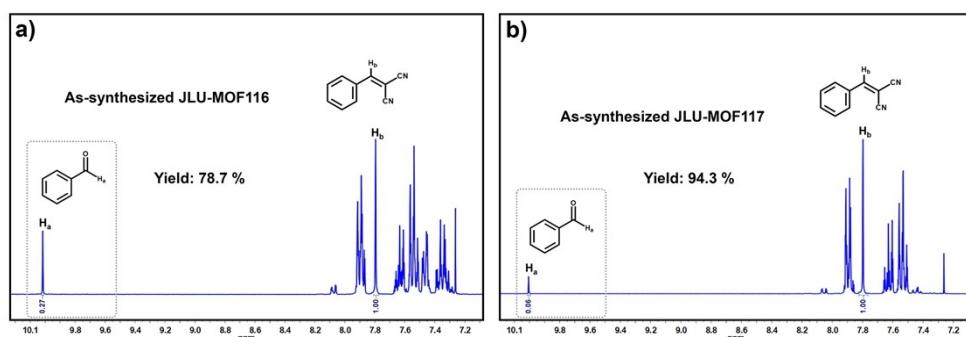


Fig. S53. The Knoevenagel condensation reactions by 0.25 mol as-synthesized **JLU-MOF116** and **JLU-MOF117**, respectively.

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