Electronic Supplementary Information (ESI)

Ultrafast scale-up synthesis of calcium rod/layer MOFs and luminescence detection of water in organic solvents

Yonghong Xiao,[a] Zhi-Hong Lin,[a] Mian Li[ab] and Xiao-Chun Huang*[ab]

[a]*Department of Chemistry and Key Laboratory for Preparation and Application of Ordered Structural Materials of Guangdong Province, Shantou University, Guangdong 515063, China*

E-mail: xchuang@stu.edu.cn

[b]*Chemistry and Chemical Engineering Guangdong Laboratory, Shantou 515031, China*

Contents

1. Experimental Details

1.1 Materials

Analytical reagents were purchased from Xilong Scientific Co., Ltd. (Shantou, China) and Energy Chemical (Shanghai, China) and used without further purification. Extra-dry ethanol (99.5%), N, N-Dimethylformamide (99.9%) and tetrahydrofuran (99.5%) were purchased from Energy Chemical (Shanghai, China). Ultrapure water was prepared by reverse osmosis technique.

1.2 Instruments and methods

Single crystal X-ray diffraction (SC-XRD) tests were performed on Rigaku XtaLab Pro MM007HF DW X diffractometer at ambient temperature. **Powder X-ray diffraction (PXRD)** data were collected on Rigaku MiniFlex600 diffractometer using Cu K_α radiation. Patterns were scanned over 5-50° (2θ) with a scan speed of 10°/min and a step width of 0.02°, respectively. **Temperature-dependent powder X-ray diffraction (TD-PXRD)** patterns were recorded on Rigaku Ultima IV X-ray diffractometer equipped with graphite-monochromatic Cu Kα radiation (λ = 1.54178 Å) in nitrogen flow at various temperatures. The morphology and size of calcium MOFs were investigated by using ZEISS Gemini 300 field-emission **scanning electron microscope (SEM)** with an acceleration voltage of 5 kV. **Thermogravimetric analysis (TGA)** was conducted on skimmer-type Rigaku Thermo mass TG-DTA/mass spectrometer system under helium atmosphere. The heating rate was 10 °C/min from room temperature to 800 °C. **Photoluminescence (PL)** spectra of as-synthesized CaMOFs were tested by using PTI QuantaMaster Model QM-TM spectrometer. **Infrared spectra (IR)** were measured from 400 cm^{-1} to 4000 cm^{-1} by Nicolet AVATAR 360 in transition mode. Samples were diluted in KBr pellets. **Elemental analysis (EA)** was conducted on Elementar Vario EL III CHNS elemental analyzer under argon atmosphere at a thermal decomposition temperature of 950 °C.

1.3 Synthetic procedures

Synthesis of $[Ca(DNBPDC)(DMF)_2]_n$ single crystal (denoted as CaDNBPDC, ROD-94)

The ligand, 2,2'-dinitro-4,4'-biphenyldicarboxylic acid (H_2DNBPDC) was prepared according to previous reported procedure.[1] 6 mg (0.02 mmol) of H_2DNBPDC, 40 μL of 0.5M $Ca(NO_3)_2$ (0.02 mmol) and 3 mL DMF were added into a 10×200 mm glass tube, which was flame sealed after addition. The tube was ultrasonicated for several minutes until all starting materials were completely dissolved. The mixture was heated up to 120 °C for 72 h then slowly cooled to room temperature in a temperature-programmed oven. Light yellow spindle-shaped crystals (yield ~70%, based on ligand) were harvested. The crystals were preserved in mother liquor before SC-XRD test.

Synthesis of $[Ca(DNBPDC)(DMF)_2]_n$ powder (denoted as CaDNBPDC, ROD-94)

In a 20 mL vial equipped with a magnetic stirring rod, 332 mg (1 mmol) of H_2DNBPDC and 5 mL DMF were added with constant stirring giving a transparent solution. 2 mL of 0.5 M $Ca(OAc)_2$ (1 mmol) was quickly injected into the former solution with stirring. After 1 min, the off-white precipitate was filtered, washed with DMF and TCM, then dried in air. Yield 96% (based on ligand). IR (KBr, cm^{-1}),3070(w), 2928(w), 1653(s), 1592(s), 1535(s), 1397(s), 1340(s), 1254(w), 1107(w), 818(w), 785(m), 720(w), 663(w). Anal. Calcd (%) for $C_{20}H_{20}N_4O_{10}Ca$: C, 46.50; H, 3.88; N, 10.68. Found: C, 45.70; H, 4.11; N, 10.75.

Synthesis of $[Ca(NH_2PTA)DMF]_n$ powder (denoted as $CaNH_2PTA$, ROD-95)[2]

In a 20 mL vial equipped with a magnetic stirring rod, 181 mg (1 mmol) of 2-aminoterephthalic acid (H_2NH_2PTA) and 5 mL DMF were added with constant stirring giving a transparent solution. 2 mL of 0.5 M $Ca(OAc)_2$ (1 mmol) was quickly injected into the former solution with stirring. After 1 min, the yellow-brown precipitate was filtered, washed with DMF and TCM, then dried in air. Yield 86% (based on ligand). IR (KBr, cm^{-1}): 3458(s), 3340(s), 2941(w), 1633(s), 1564(s), 1491(s), 1413(s), 1377(s), 1324(m), 1255(s), 1104(m), 835(m), 774(s), 672(m), 570(w), 509(w). Anal. Calcd (%) for $C_{11}H_{12}N_2O_5Ca$: C, 45.16; H, 3.75; N, 9.57. Found: C, 44.77; H, 4.12; N, 9.37.

Synthesis of $[Ca(NDC)DMF]_n$ powder (denoted as CaNDC)[3]

In a 20 mL vial equipped with a magnetic stirring rod, 216 mg (1 mmol) of 2,6-naphthalenedicarboxylic acid (H_2NDC) and 5 mL DMF were added with constant stirring giving a milky mixture. 2 mL of 0.5 M $Ca(OAc)_2$ (1 mmol) was quickly injected into the former solution with stirring. After 1 min, the white precipitate was filtered, washed throughoutly with DMF and TCM, then dried in air. Yield 87% (based on ligand). IR (KBr, cm^{-1}), 2939(w), 1646(s), 1609(s), 1553(m), 1497(s), 1404(s), 1366(s), 1185(m), 1079(m), 928(w), 786(s), 729(m), 660(m), 554(w), 436(s). Anal. Calcd (%) for $C_{15}H_{13}NO_5Ca$: C, 55.04; H, 3.98; N, 4.28. Found: C, 56.00; H, 4.01; N, 3.92.

Synthesis of $\{[Ca_2(PTA)_2(H_2O)_2(DMF)_2]\cdot H_2O\}_n$ powder (denoted as CaPTA)[4]

In a 20 mL vial equipped with a magnetic stirring rod, 166 mg (1 mmol) of terephthalic acid (H_2PTA) and 5 mL DMF were added with constant stirring giving a colorless, transparent solution. 2 mL of 0.5 M $Ca(OAc)_2$ (1 mmol) was quickly injected into the former solution with stirring. After 1 min, the white precipitate was filtered, washed throughoutly with DMF and TCM, then dried in air. Yield 90% (based on ligand). IR (KBr, cm^{-1}), 2928(s), 1666(s), 1556(s), 1397(s), 1112(w), 1031(w), 1023(w), 827(m), 753(m), 677(m), 513(w). Anal. Calcd (%) for $C_{22}H_{28}N_2O_{13}Ca_2$: C, 43.42; H, 4.61; N, 4.61. Found: C, 41.92; H, 4.87; N, 4.90.

Synthesis of $\{[Ca_3(BTC)_2(H_2O)_2(DMF)_2]\cdot 3H_2O\}_n$ powder (denoted as CaBTC)[2]

In a 20 mL vial equipped with a magnetic stirring rod, 210 mg (1 mmol) of trimesic acid (H_3BTC) and 5 mL DMF were added with constant stirring giving a colorless, transparent solution. 2 mL of 0.5 M $Ca(OAc)_2$ (1 mmol) was quickly injected into the former solution with stirring. After 1 min, the white precipitate was filtered, washed throughoutly with DMF and TCM, then dried in air. Yield 60% (based on ligand). IR (KBr, cm^{-1}), 2932(w), 1665 (s), 1621(s), 1540(s), 1434(s), 1378(s), 1116(m), 766(m), 723(m), 667(w), 523(w). Anal. Calcd (%) for $C_{24}H_{30}N_2O_{19}Ca_3$: C, 37.40; H, 3.90; N, 3.64. Found: C, 38.82; H, 4.22; N, 3.98.

Synthesis of $[Ca(BPDC)H_2O]_n$ powder (denoted as CaBPDC)[5]

In a 20 mL vial equipped with a magnetic stirring rod, 244 mg (1 mmol) of 4,4'-biphenyldicarboxylic acid (H_2BPDC) and 5 mL DMAc were added with constant stirring giving a milky liqui. 2 mL of 0.5 M $Ca(OAc)_2$ (1 mmol) was quickly injected into the former solution with stirring. After 1 min, the white precipitate was filtered, washed throughoutly with DMAc and TCM, then dried in air. Yield 93% (based on ligand). IR (KBr, cm^{-1}), 2925(w), 2683(w), 1658(s), 1584(s), 1515(s), 1438(s), 1397(s), 1177(m), 994(w), 851(m), 770(s), 668(m), 521(s). Anal. Calcd (%) for $C_{14}H_{10}O_5Ca$: C, 56.38; H, 3.36; N, 0.00. Found: C, 52.12; H, 3.94; N, 1.56.

Synthesis of $[Ca(DHPTA)(DMF)_2]_n$ powder (denoted as CaDHPTA)[2]

In a 20 mL vial equipped with a magnetic stirring rod, 198 mg (1 mmol) of 2,5-dihydroxyterephthalic acid (H_2DHPTA) and 5 mL DMF were added with constant stirring giving a dark brown solution. 2 mL of 0.5 M $Ca(OAc)_2$ (1 mmol) was quickly injected into the former solution with stirring. After 1 min, the yellowish precipitate was filtered, washed with DMF and TCM, then dried in air. Yield 93% (based on ligand). IR (KBr, cm^{-1}), 2927(w), 2863(w), 1677(s), 1443(s), 1339(m), 1226(m), 1097(m), 924(w), 872(m), 829(m), 768(s), 673(s), 604(w), 526(w). Anal. Calcd (%) for $C_{14}H_{18}N_2O_8Ca$: C, 43.98; H, 4.71; N, 7.33. Found: C, 42.82; H, 3.98; N, 7.65.

1.4 Single crystal X-ray crystallography

Single crystal of **CaDNBPDC** was transferred to a microscope slide along with a little mother liquor. A small piece of crystal (about $0.1\times0.1\times0.2$ mm^3) was picked and washed completely in vaseline, then mounted using a Dual-Thickness MicroLoop™ (**MiTeGen, Ithaca, New York, USA**).

Data collection was performed on Rigaku Oxford XtaLAB Pro diffractometer equipped with micro focus sealed X-ray tube (Cu K_α radiation, λ = 1.54178 Å) and PILATUS 200K detector at 298 K. Data reduction, absorption correction was conducted on CrysAlisPro (Rigaku,

V1.171.39.7e, 2015). Absorption correction was based on implanted spherical harmonics in SCALE3 ABSPACK.

The structure was solved by intrinsic phasing method using *SHELXT*[6] program implanted in *Olex2*[7]. Refinement with full matrix least squares techniques on F^2 was performed by using *SHELXL*[8]. Non-hydrogen atoms were refined anisotropically and all hydrogen atoms were generated based on riding mode. Crystallographic data and structure refinement results are given in Table S1. Selected bond lengths and angles are shown in Table S2.

1.5 Topological analysis

Systre[9] program was used to identify the underlying net of MOFs. *ToposPro*[10] program was used for computing point symbol and vertex symbol[11] of the net and *Topcryst*[10] database (The Samara Topological Data Center) can be checked for occurrences of nets in crystal structures. More information of **vbb** net is available in the *RCSR*[12] database (see http://rcsr.net/nets/vbb).

1.5.1 Topological information of vbb net

Point symbol for net: $\{4^3.6^2.8\}$

Extended point symbol: [4.4.4.8.6.6]

4-c net; uninodal net

Coordination sequences

C1: 1 2 3 4 5 6 7 8 9 10

Num 4 9 18 36 58 85 118 156 198 245

Cum 5 14 32 68 126 211 329 485 683 928

TD10 = 928

1.5.2 Systre net file of vbb (*.cgd)

```
CRYSTAL
  NAME vbb
  GROUP Fddd:2
  CELL 2.12405 2.09325 6.75151 90.0000 90.0000 90.0000
  NODE 1 6  0.36121 0.13535 0.05082
  EDGE  0.36121 0.13535 0.05082   0.13879 0.36465 -0.05082
  EDGE  0.36121 0.13535 0.05082   -0.11121 0.11465 0.05082
  EDGE  0.36121 0.13535 0.05082   0.11121 -0.11465 -0.05082
  EDGE  0.36121 0.13535 0.05082   0.38879 0.61465 0.05082
  EDGE  0.36121 0.13535 0.05082   0.36121 0.11465 0.19918
# EDGE_CENTER  0.25000 0.25000 -0.00000
# EDGE_CENTER  0.12500 0.12500 0.05082
# EDGE_CENTER  0.23621 0.01035 -0.00000
# EDGE_CENTER  0.37500 0.37500 0.05082
# EDGE_CENTER  0.36121 0.12500 0.12500
END
```

1.5 Solvent stability test of $CaNH_2PTA$ (ROD-95)

At room temperature, 5 mg $CaNH_2PTA$ was weighed and placed in 10 mL screw-cap scintillation vial, 5 mL solvent was added. After being oscillated for 1 min at room temperature, the supernatant (about 2 mL) was taken out for PL test and the rest mixture was capped and placed still for 3 days. The resulting solid was filtered and used for PXRD test.

1.6 Procedures of moisture sensing

$CaNH_2PTA$ (ROD-95) was synthesized via the method reported in ***1.3 Synthetic procedures***. At room temperature, 5 mg $CaNH_2PTA$ was weighed and placed in 5 mL EP centrifuge tube,

3 mL mixed solvent (water and ultra-dry DMF/EtOH/THF) was added. After being oscillated for 30 s at room temperature, the clear liquid was filtered by syringe with filter membrane. The solution was further transferred to a cuvette, sealed, and the steady state fluorescence test was performed. 360 nm was selected as the excitation wavelength, and the range of emission wavelength was 380 nm to 600 nm.

1.7 Preparation of fluorescent test paper

The absorbent paper (purchased from Taizhou Jin'ao Paper Co. Ltd.) was cut into square chips with a length of about 1 cm. The chips were soaked in 5 mL DMF solution containing 1 mmol ligand, stirred, and then 2 mL 0.5 M calcium acetate aqueous solution was quickly injected into the DMF solution. At this time, a large amount of light yellow $CaNH_2PTA$ (ROD-95) was generated. After standing for 10 min, the free calcium MOF and DMF solution were removed with a burette. Fresh DMF was added for washing. After 5 cycles, the test papers were taken out with a tweezer and dried.

2. Supporting Figures and Tables

Table S1 Crystallographic data for CaDNBPDC (ROD-94)

CCDC No. 1979626; DOI: 10.5517/ccdc.csd.cc24fyxg

Empirical formula	$C_{20}H_{20}N_4O_{10}Ca$
Formula weight	516.48
Crystal system	Monoclinic
Space group	*C*2/c
a (Å)	12.9400(3)
b (Å)	13.0550(2)
c (Å)	27.6303(6)
α (°)	90
β (°)	95.917(2)
γ (°)	90
V (Å^3)	4642.77(16)
Z	8
Temperature (K)	293(2)
Density (calculated)	1.478 g/cm^3
Absorption coefficient	2.902 mm^{-1}

$F(000)$	2144
Crystal size	0.2 x 0.1 x 0.1 mm^3
Theta range for data collection	3.216 to 74.102°
Index ranges	-16<=*h*<=14, -9<=*k*<=16, -33<=*l*<=34
Reflections collected	12374
Independent reflections	4571 [R_{int} = 0.0275]
Completeness to theta = 67.684°	99.7 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4571 / 51 / 363
Goodness-of-fit on F^2	1.095
Final *R* indices [I>2sigma(I)][a]	R_1 = 0.0668, wR_2 = 0.1765
R indices (all data)[a]	R_1 = 0.0687, wR_2 = 0.1773
Extinction coefficient	0.00019(5)
Largest diff. peak and hole	0.734 and -0.520 e.$Å^{-3}$

[a] $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR_2 = \{[\Sigma w(F_o^2-F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$; $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, where $P = [\max(F_o^2, 0) + 2F_c^2]/3$ for all data.

Table S2 Selected bond lengths [Å] and angles [°] for CaDNBPDC (ROD-94)

Ca(1)-O(7)	2.284(3)	O(7)-Ca(1)-O(10)	83.16(11)
Ca(1)-O(2)#1	2.289(3)	O(2)#1-Ca(1)-O(10)	177.40(11)
Ca(1)-O(8)#2	2.319(3)	O(8)#2-Ca(1)-O(10)	91.44(11)
Ca(1)-O(1)#3	2.355(3)	O(1)#3-Ca(1)-O(10)	79.71(11)
Ca(1)-O(10)	2.363(3)	O(7)-Ca(1)-O(9)	86.16(10)
Ca(1)-O(9)	2.371(3)	O(2)#1-Ca(1)-O(9)	86.14(10)
Ca(1)-C(18)	3.213(5)	O(8)#2-Ca(1)-O(10)	91.44(11)
O(7)-Ca(1)-O(2)#1	94.66(10)	O(1)#3-Ca(1)-O(10)	79.71(11)
O(7)-Ca(1)-O(8)#2	103.72(11)	O(7)-Ca(1)-O(9)	86.16(10)
O(2)#1-Ca(1)-O(8)#2	87.70(10)	O(2)#1-Ca(1)-O(9)	86.14(10)
O(7)-Ca(1)-O(1)#3	162.09(11)	O(8)#2-Ca(1)-O(9)	168.77(11)
O(2)#1-Ca(1)-O(1)#3	102.59(10)	O(1)#3-Ca(1)-O(9)	90.19(11)
O(8)#2-Ca(1)-O(1)#3	81.99(11)	O(10)-Ca(1)-O(9)	95.10(11)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y,-z+3/2 #2 -x+1,-y+1,-z+1 #3 x-1/2,-y+3/2,z-1/2 #4 x+1/2,-y+3/2,z+1/2

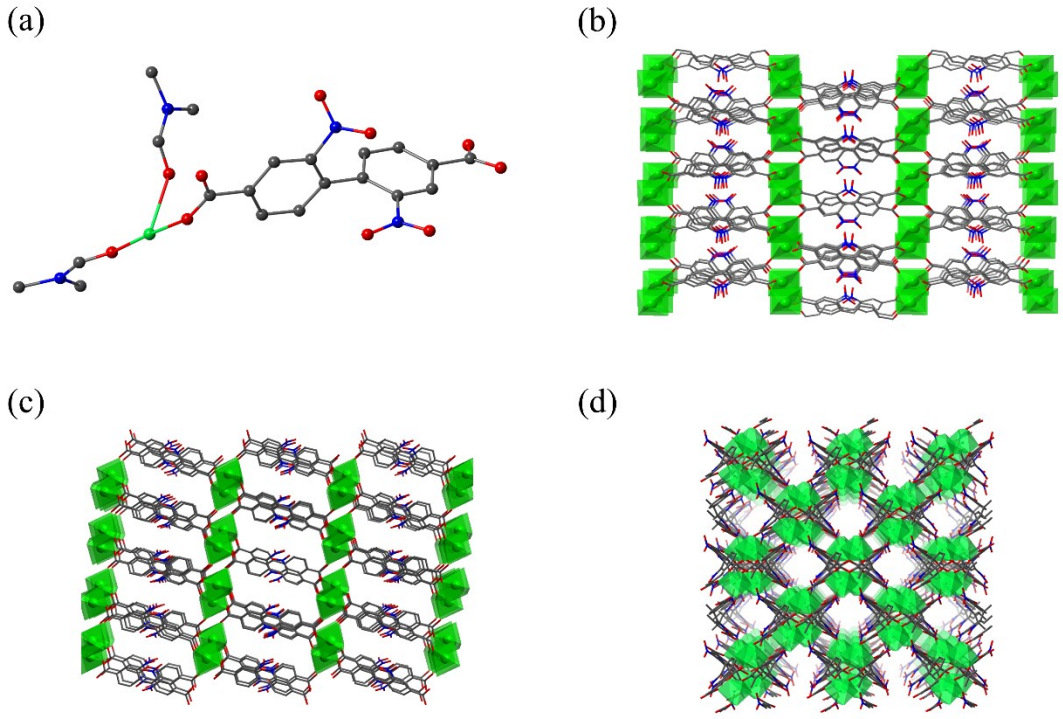

Fig. S1 Structure of ROD-94. (a) The asymmertric unit of CaDNBPDC; (b, c, d) 3D presentation views along a,b,c-axis (All DMF molecules were omitted for clarity).Color codes: Ca, green sphere and plolyhdra; C, dark grey; O, red; N, blue.

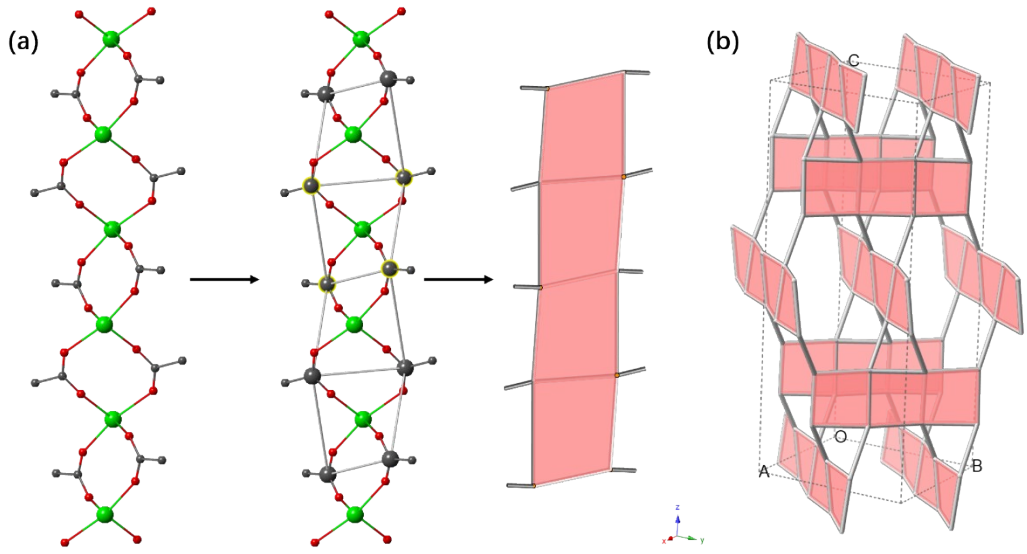

Fig. S2 Topology (underlying net) of ROD-94. (a) Ladder-shape rod SBU (shown in red); (b) vbb net with 2-way rods.

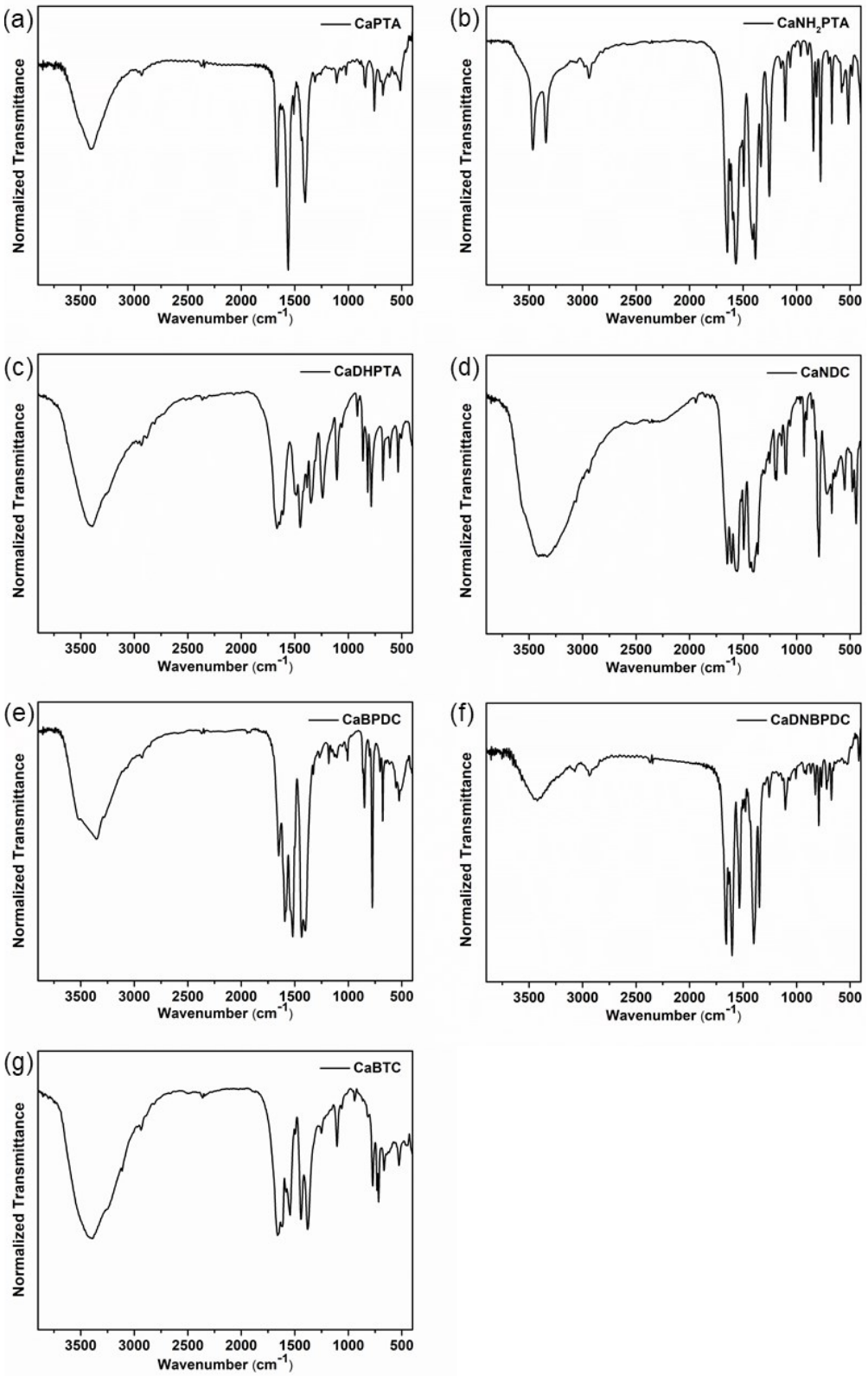

Fig. S3 IR spectra of as-synthesized 7 types of Ca MOFs (a) CaPTA (b) $CaNH_2PTA$ (c) CaDHPTA (d) CaNDC (e) CaBPDC (f) CaDNBPDC (g) CaBTC

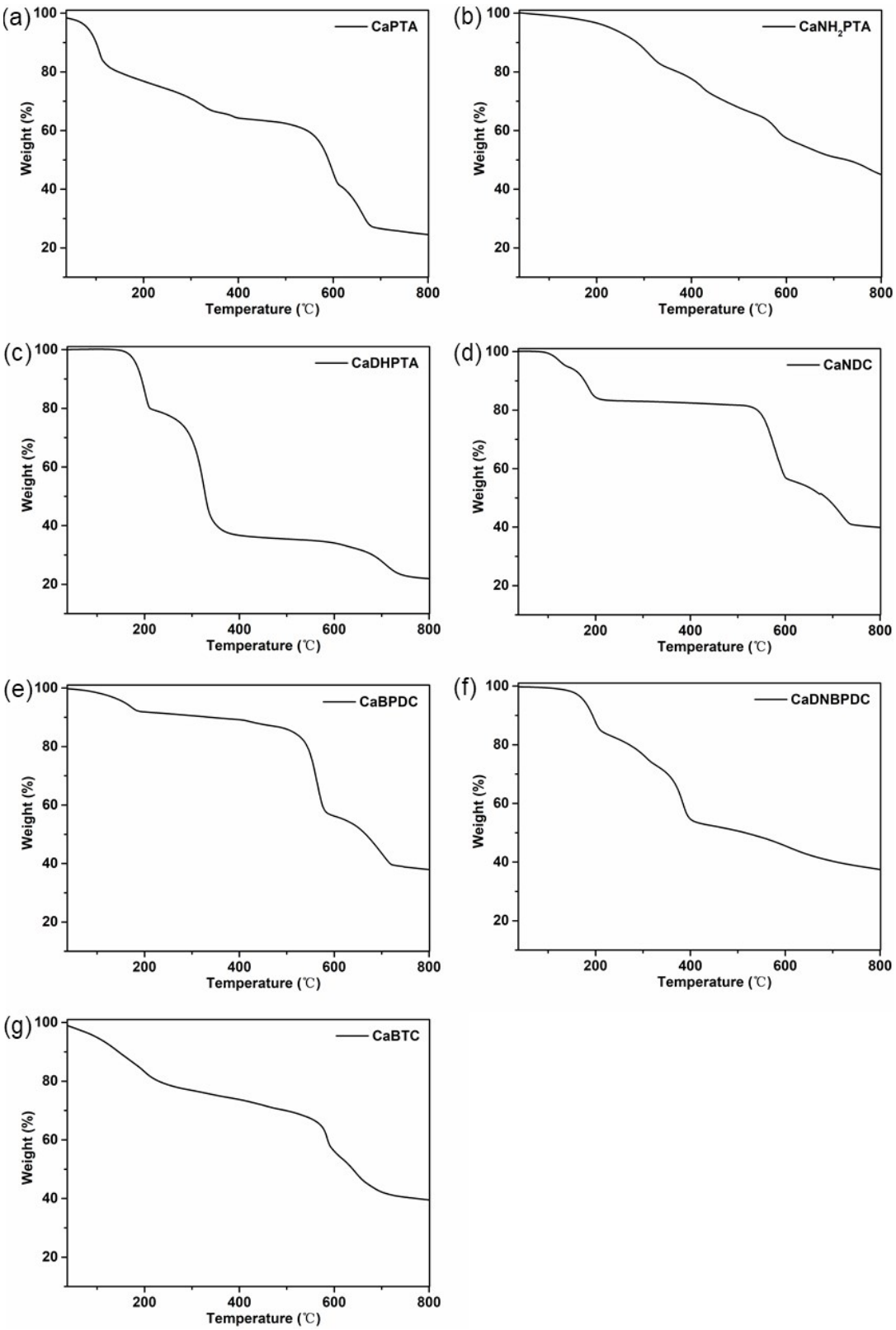

Fig. S4 TGA diagrams of as-synthesized 7 types of Ca MOFs (a) CaPTA (b) $CaNH_2PTA$ (c) CaDHPTA (d) CaNDC (e) CaBPDC (f) CaDNBPDC (g) CaBTC

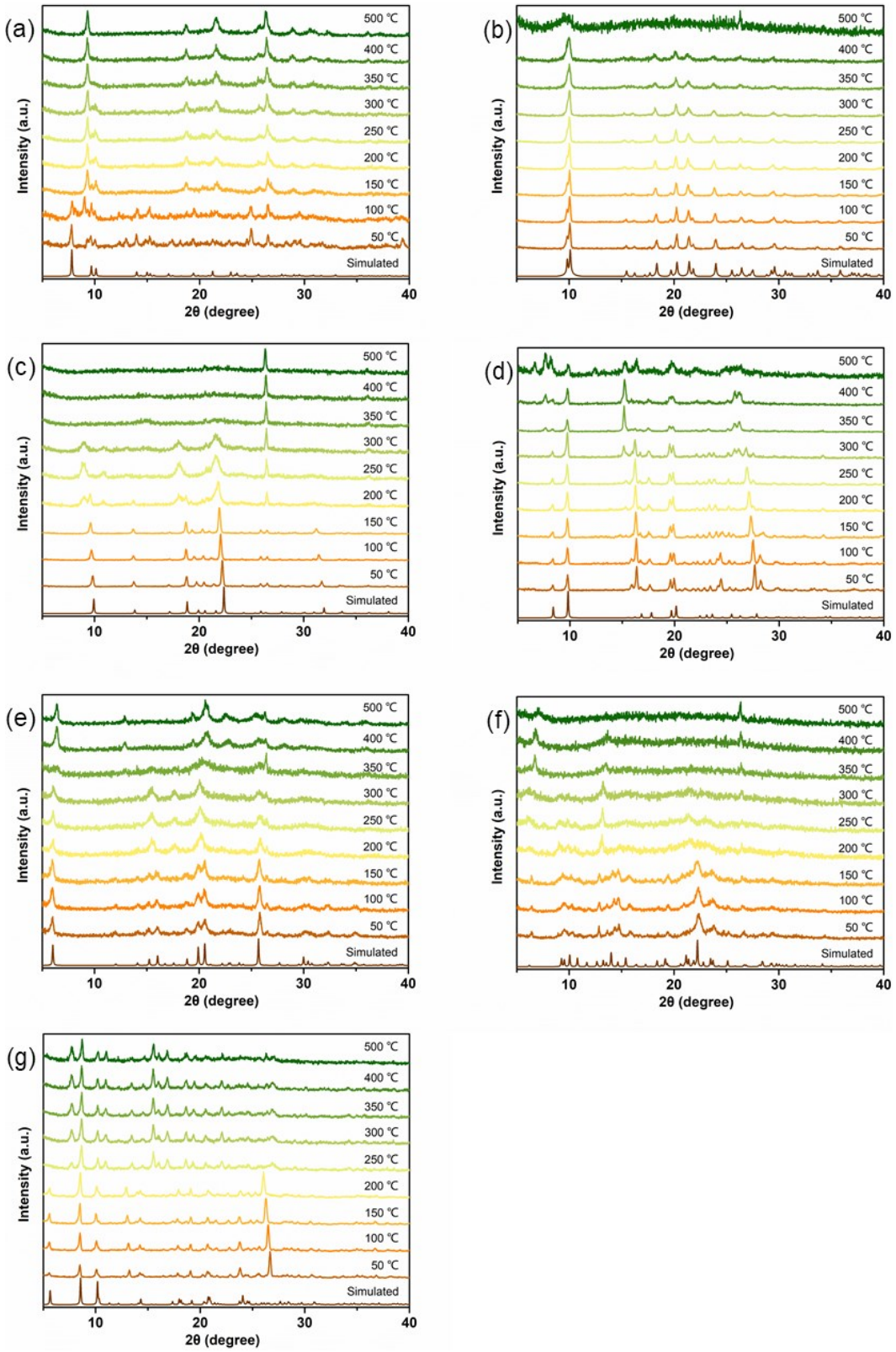

Fig. S5 VT-PXRD patterns of as-synthesized 7 types of Ca MOFs (a) CaPTA (b) $CaNH_2PTA$ (c) CaDHPTA (d) CaNDC (e) CaBPDC (f) CaDNBPDC (g) CaBTC

Table S3 Yield calculation for $CaNH_2PTA$ (ROD-95) synthesized in different durations

Reaction time (min)	Mass of product (mg)	Yield (%)
1	241.2	83.9
2	248.3	84.6
5	245.4	83.9
10	248.9	85.3
30	250.0	85.6

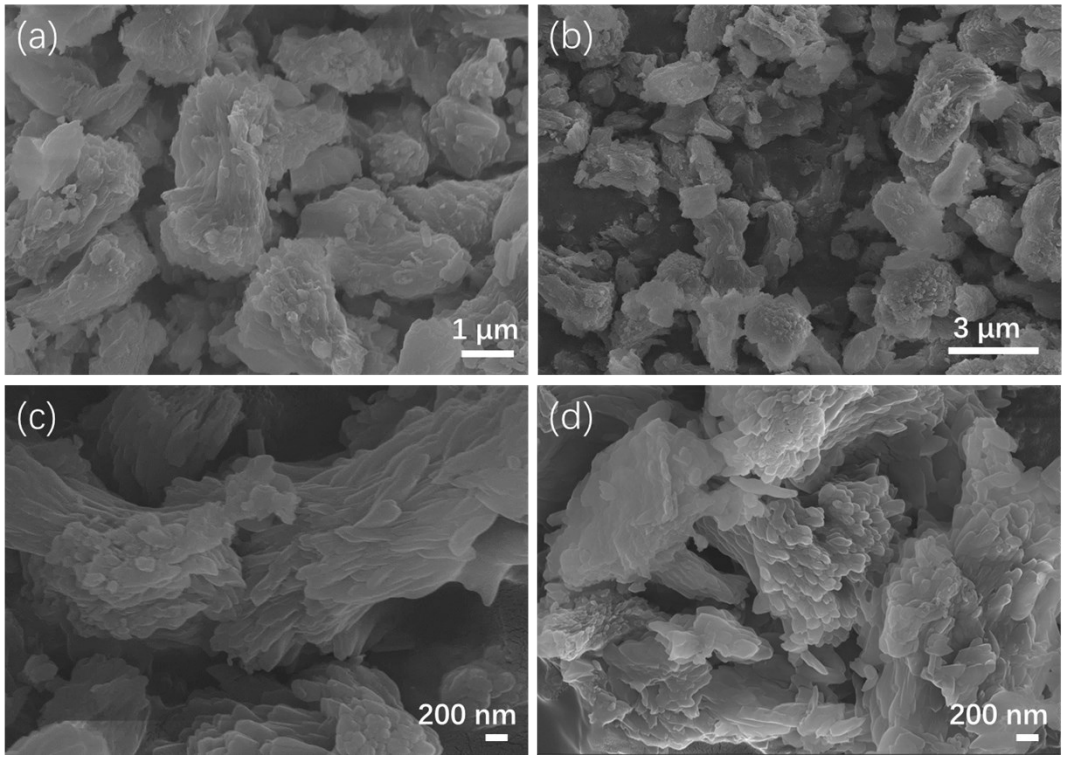

Fig. S6 SEM images of $CaNH_2PTA$ (ROD-95) synthesized within (a) 1 min, (b) 2 min, (c) 5 min and (d) 10 min

Table S4 Yield calculation for $CaNH_2PTA$ (ROD-95) synthesized with different DMF concentrations

DMF volume (mL)	Mass of product (mg)	Yield (%)
5	246.2	84.2
8	249.3	85.3
10	242.4	82.9
15	242.9	83.2
20	248.2	84.9

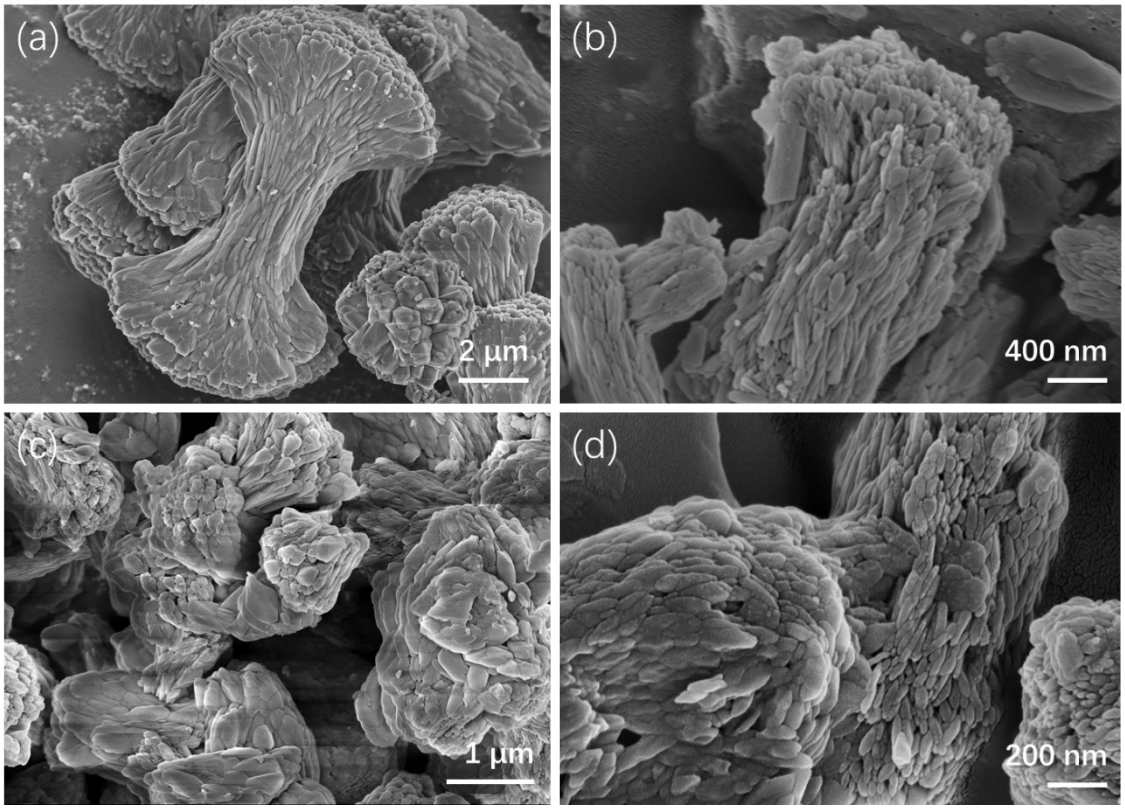

Fig. S7 SEM images of $CaNH_2PTA$ (ROD-95) synthesized in (a) 5 mL, (b) 10 mL, (c) 15 mL and (d) 20 mL DMF

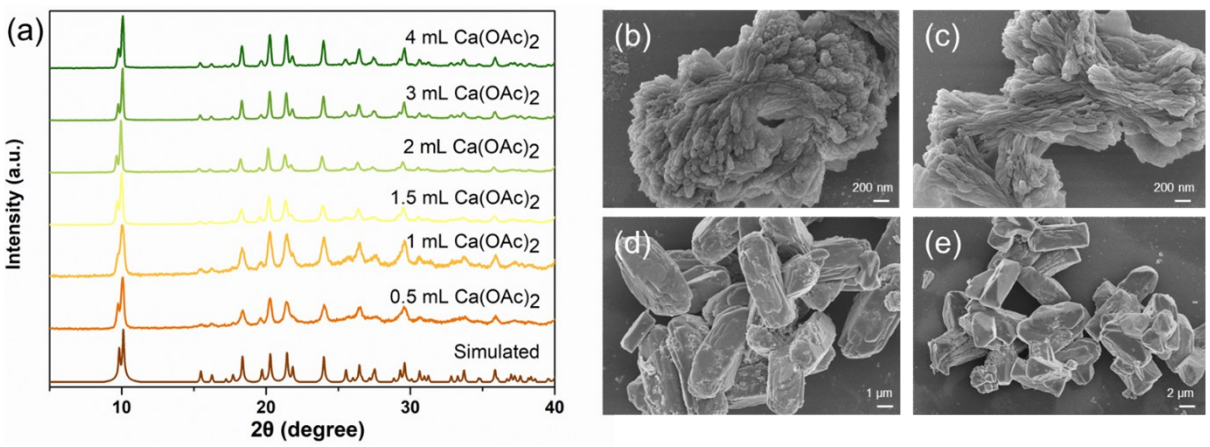

Fig. S8 (a) PXRD patterns of products synthesized with different addition amounts of $Ca(OAc)_2$ solution (b-e) SEM images of $CaNH_2PTA$ (ROD-95) synthesized with 0.5 mL, 1 mL, 3 mL and 4 mL $Ca(OAc)_2$ solution

Table S5 Yield calculation for $CaNH_2PTA$ (ROD-95) synthesized with different addition amounts of $Ca(OAc)_2$ solution

Volume of $Ca(OAc)_2$ solution (mL)	Mass of product (mg)	Yield (%)
0.5	82.3	28.2
1	176	60.2
1.5	235.5	80.6
2	246.2	84.2
3	246.8	84.4
4	247.4	84.6

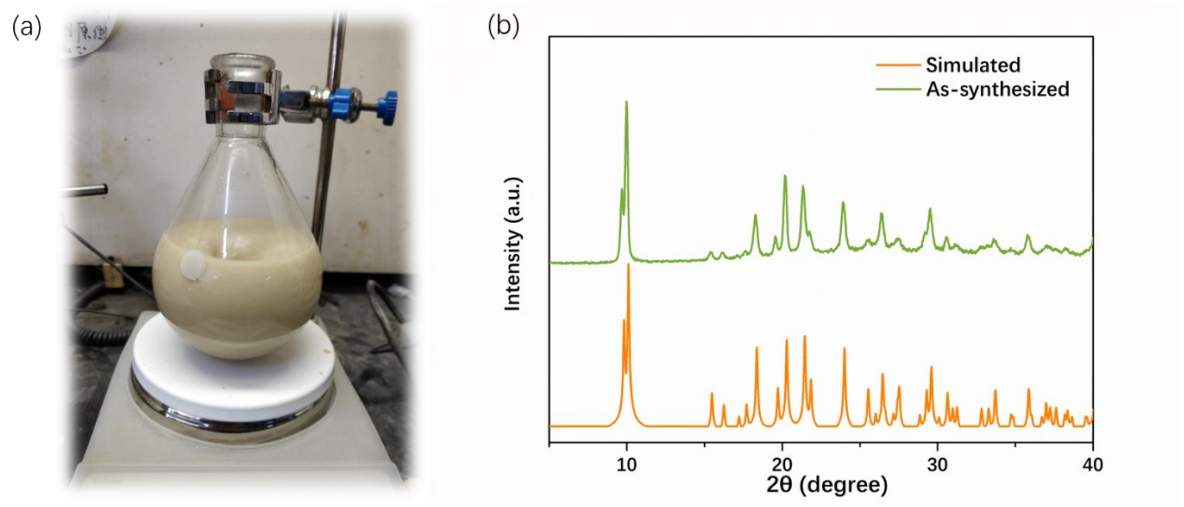

Fig. S9 (a) The photograph and (b) PXRD patterns of $CaNH_2PTA$ (ROD-95) synthesized in a batch upscaled 60 times.

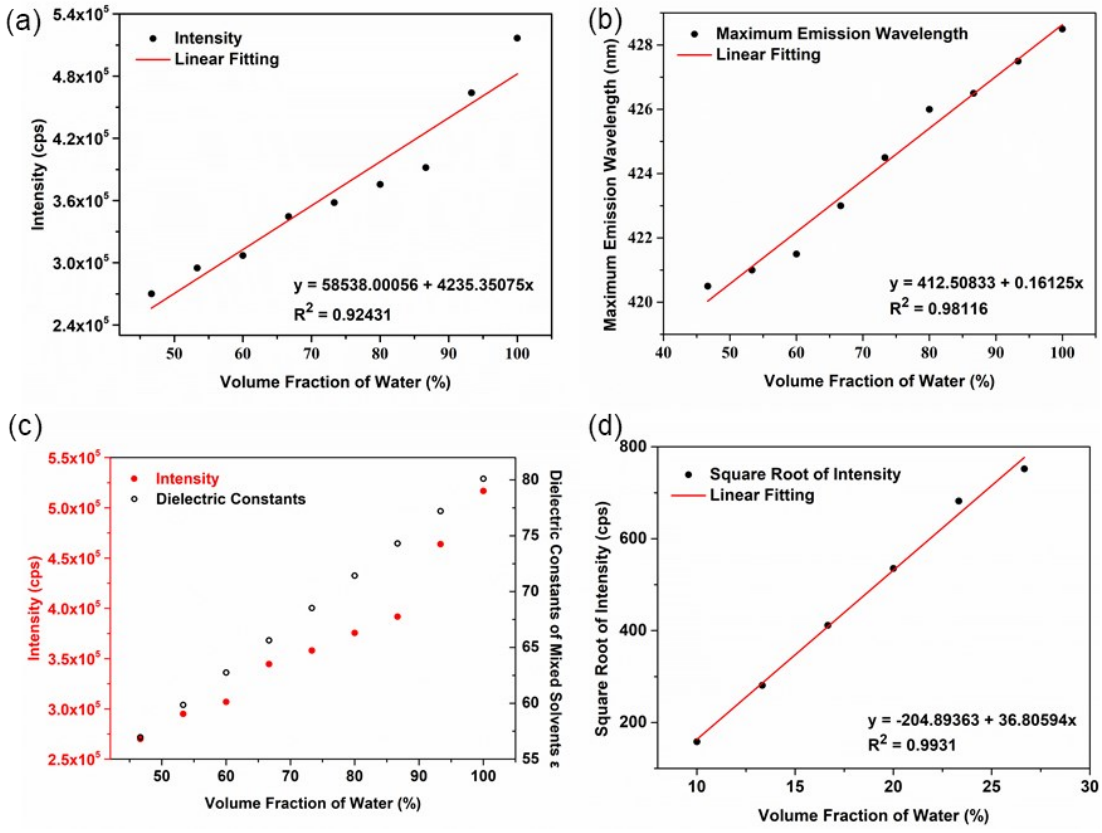

Fig. S10 Diagrams of (a) intensity and (b) maximum emission wavelength against water ratio in DMF-H_2O system; (c) intensity against dielectric constants and (d) square roots of intensity against volume fraction of water in DMF-H_2O system.

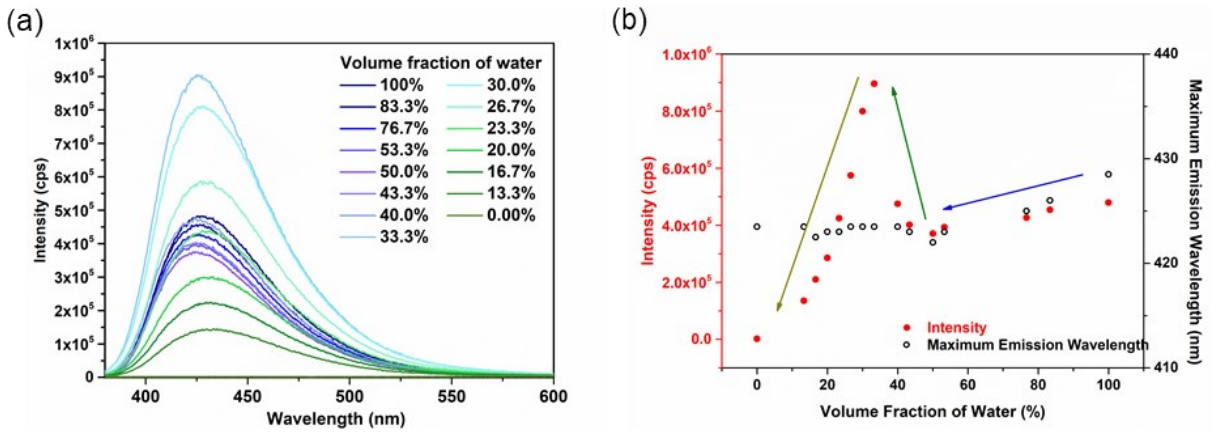

Fig. S11 (a) The PL spectrum for the supernatant of $CaNH_2PTA$ (ROD-95) and water-EtOH mixture with different water ratio; (b) Diagram of maximum emission intensity and wavelength as a function of water concentration

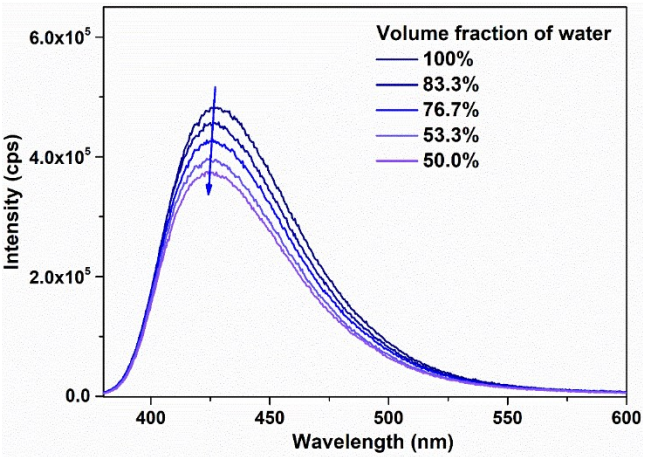

Fig. S12 The PL spectrum for the supernatant of $CaNH_2PTA$ (ROD-95) and water-EtOH mixtures with 50% to 100% of water

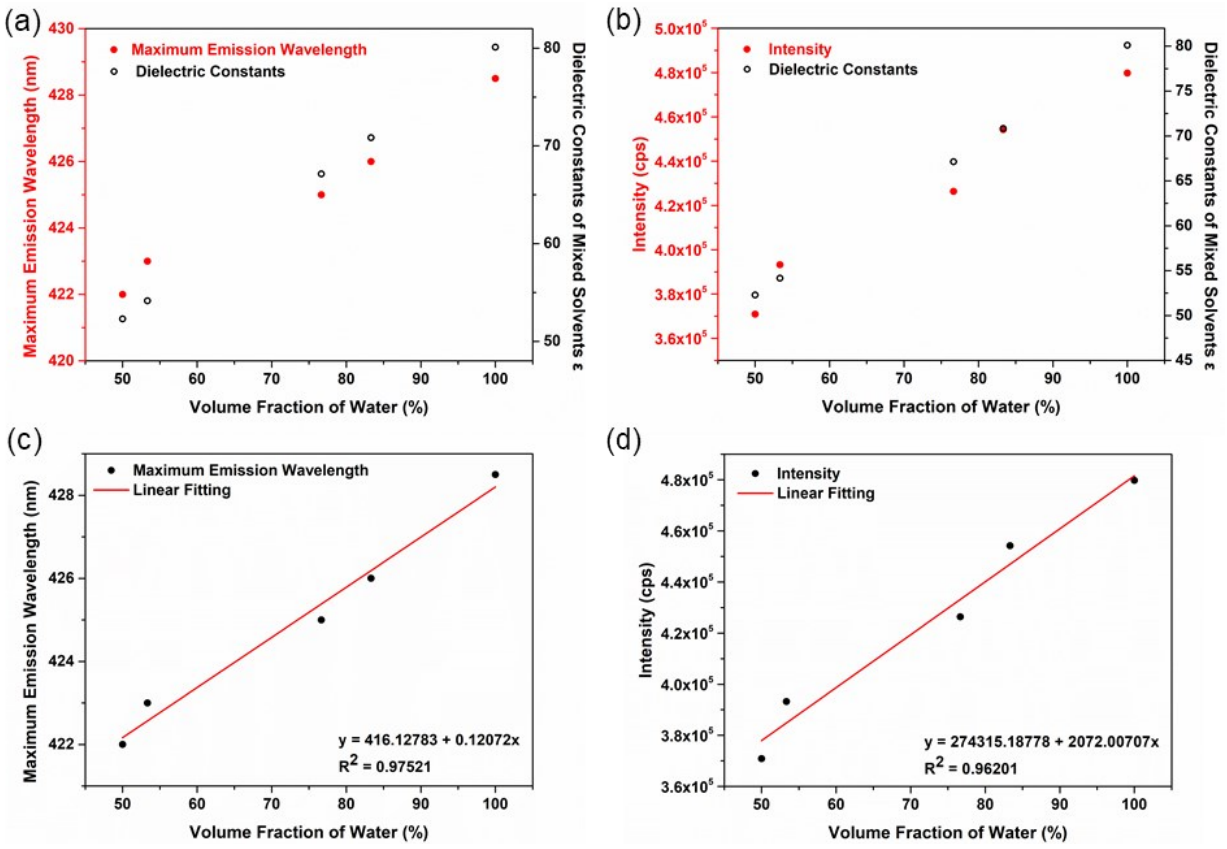

Fig. S13 Diagrams of (a) maximum emission wavelength and (b) intensity against water ratio and (c) maximum emission wavelength and (d) intensity against dielectric constants in water-EtOH system with 50% to 100% of water

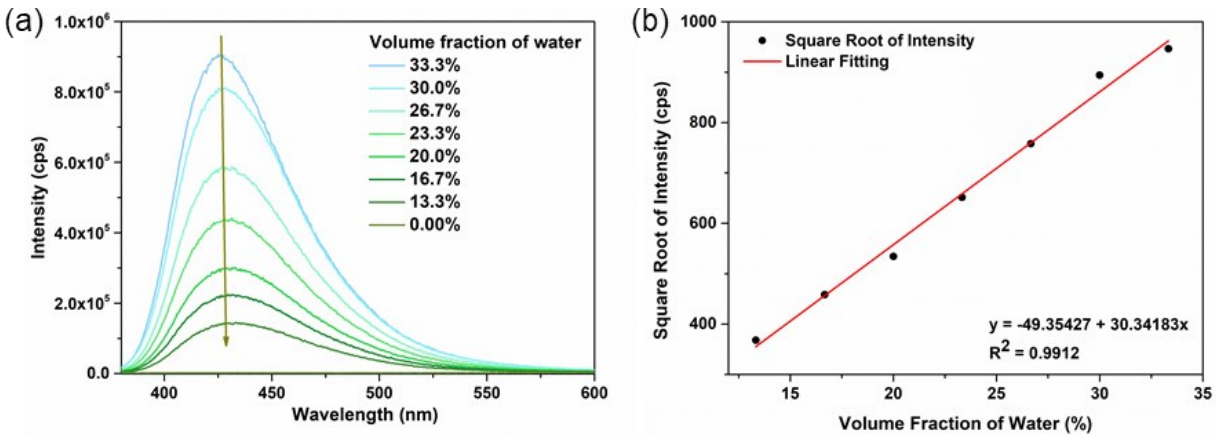

Fig. S14 Diagrams of (a) PL spectrum in low concentration area and (b) square roots of intensity against volume fraction of water in EtOH-H_2O system

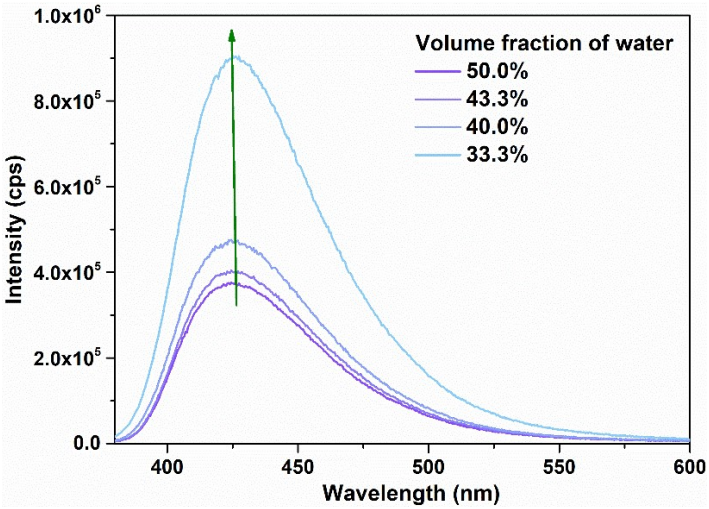

Fig. S15 Diagram of PL spectrum of sudden-change area in EtOH-H_2O system

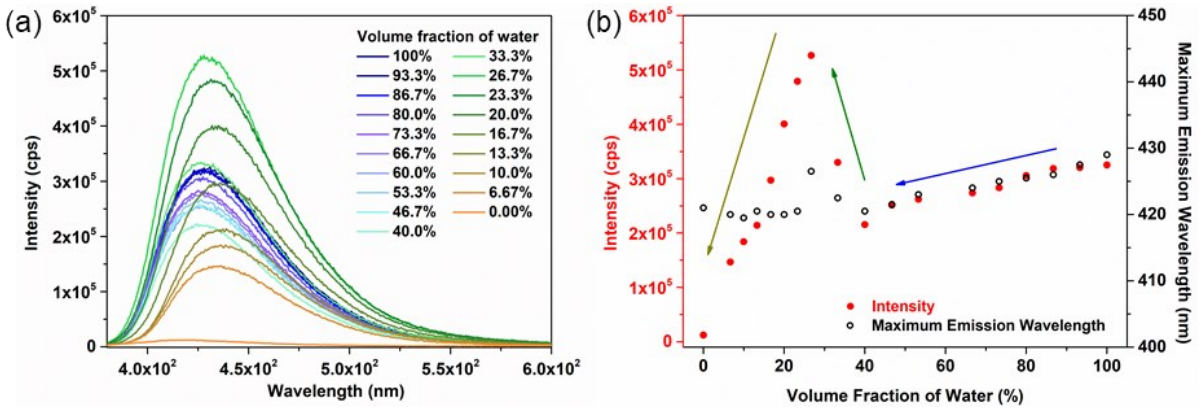

Fig. S16 (a) The PL spectrum for the supernatant of $CaNH_2PTA$ (ROD-95) and water-THF mixture with different water ratio; (b) Diagram of maximum emission intensity and wavelength as a function of water concentration

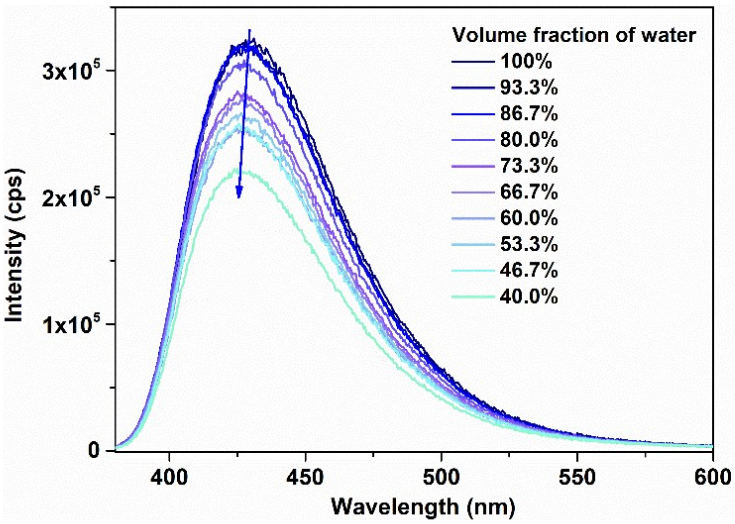

Fig. S17 The PL spectrum for the supernatant of $CaNH_2PTA$ (ROD-95) and water-THF mixtures with 40% to 100% of water

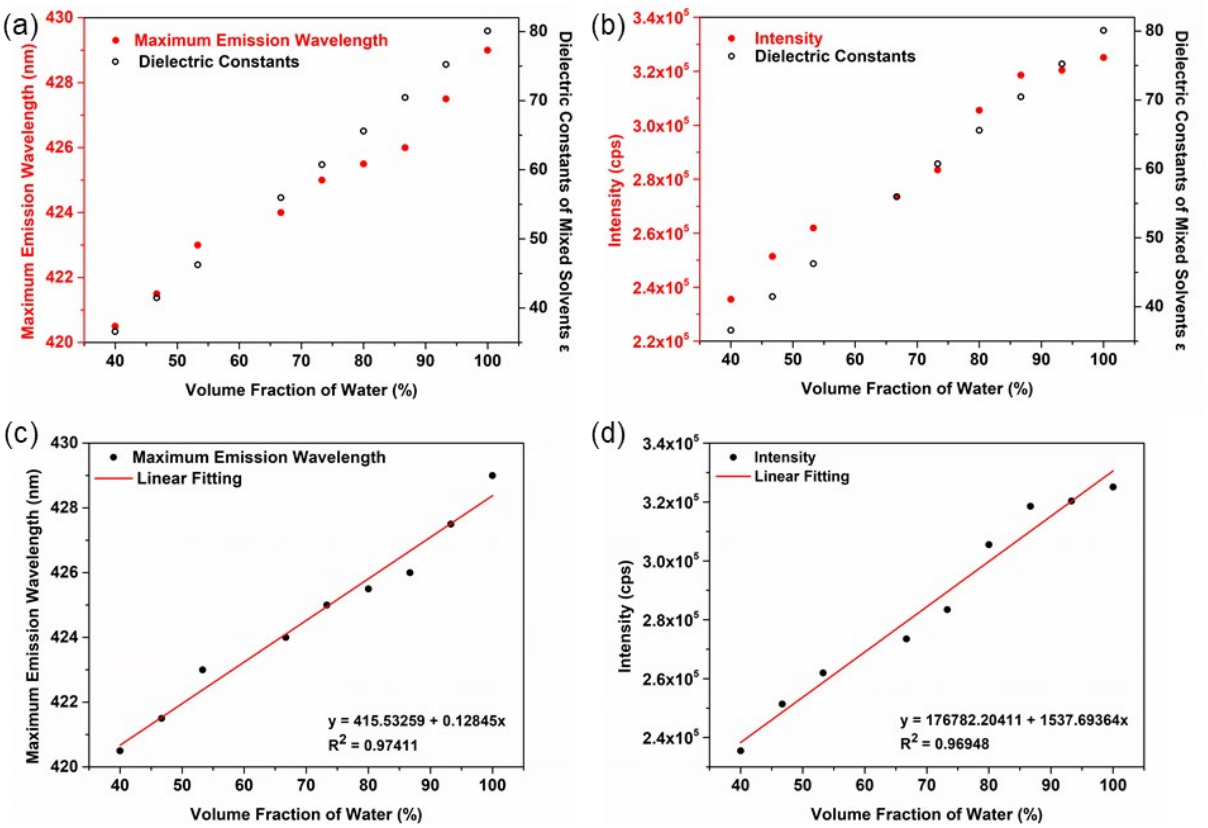

Fig. S18 Diagrams of (a) maximum emission wavelength and (b) intensity against water ratio and (c) maximum emission wavelength and (d) intensity against dielectric constants in water-THF system with 40% to 100% of water

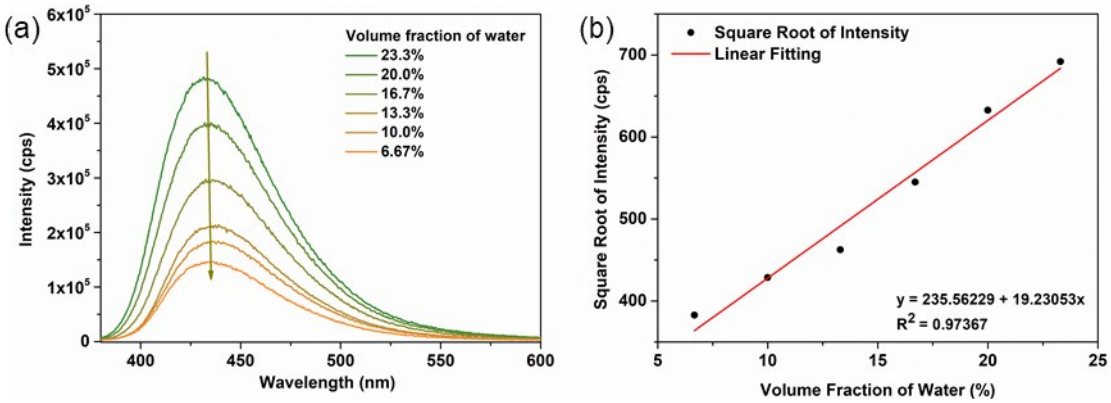

Fig. S19 Diagrams of (a) PL spectrum in low concentration area and (b) square roots of intensity against volume fraction of water in THF-H_2O system

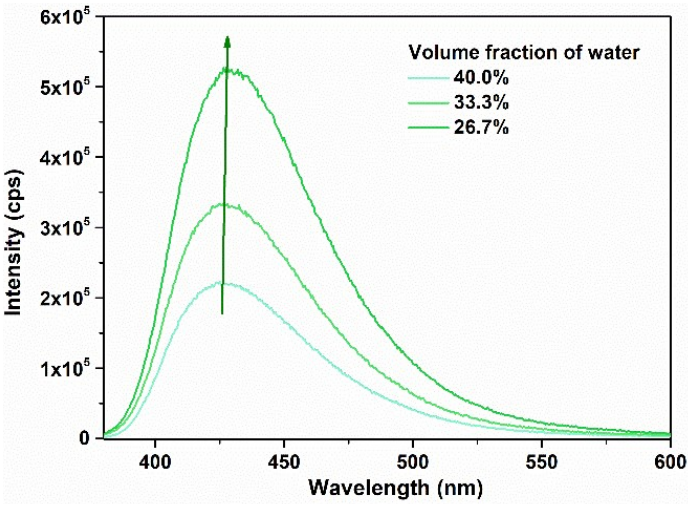

Fig. S20 Diagram of PL spectrum of sudden-change area in THF-H_2O system

3. References

1. P. V. Dau and S. M. Cohen, *CrystEngComm*, 2013, **15**, 9304.
2. P.-C. Liang, H.-K. Liu, C.-T. Yeh, C.-H. Lin and V. t. z. Zima, *Cryst. Growth Des.*, 2011, **11**, 699.
3. C. A. Williams, A. J. Blake, C. Wilson, P. Hubberstey and M. Schröder, *Cryst. Growth Des.*, 2008, **8**, 911.
4. M. Mazaj and N. Zabukovec Logar, *Cryst. Growth Des.*, 2015, **15**, 617.
5. C. Volkringer, J. Marrot, G. Férey and T. Loiseau, *Cryst. Growth Des.*, 2008, **8**, 685.
6. G. M. Sheldrick, *Acta Crystallogr. A*, 2015, **71**, 3.
7. O. V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard and H. Puschmann, *J. Appl. Crystallogr.*, 2009, **42**, 339.
8. G. M. Sheldrick, *Acta Crystallogr. A*, 2008, **64**, 112.
9. O. Delgado-Friedrichs and M. O'Keeffe, *Acta Crystallogr. A*, 2003, **59**, 351. Access at: http://gavrog.org/
10. V. A. Blatov, A. P. Shevchenko and D. M. Proserpio, *Cryst. Growth Des.*, 2014, **14**, 3576. Access at: https://topospro.com/ and https://topcryst.com/
11. V. A. Blatov, M. O'Keeffe and D. M. Proserpio, *CrystEngComm*, 2010, **12**, 44.
12. M. O'Keeffe, M. A. Peskov, S. J. Ramsden and O. M. Yaghi, *Acc. Chem. Res.*, 2008, **41**, 1782. Access at: http://rcsr.net/