

Solvent Dependent Structures of Melamine: Porous or Nonporous?

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(5) Supporting Information

ABSTRACT: Two hydrogen-bonded organic frameworks (HOFs) of the melamine (MA), MA-1-H₂O and MA-2-DMF, have been crystallized in H₂O and DMF (*N*,*N*-Dimethylformamide), respectively. Structurally, MA-1-H₂O and MA-2-DMF are condensed and porous, respectively. The porous MA-2-DMF sustains the porous feature under the solvent exchange by acetone, dichloromethane (CH₂Cl₂), and toluene, while it transforms into the condensed one under methanol (MeOH) and ethanol (EtOH). Furthermore, MA-2-DMF and MA-2-Toluene can undergo reversible single



crystal-single crystal transformation with retention of the porous hydrogen-bonded organic frameworks.

INTRODUCTION

Hydrogen-bonded organic frameworks (HOFs) have regained extensive interest recently because of their potential applications as organic porous materials for carbon dioxide capture,¹ hydrocarbon adsorption and separation,¹⁻¹⁰ chiral resolution,¹¹ and luminescent properties.^{12,13} Compared with other types of porous materials such as zeolites and metal–organic frameworks,^{14–18} HOFs can not only be easily prepared by crystallization, but more importantly can be straightforwardly recycled and processed through a solution chemistry approach, making them unique porous materials for their useful applications and device fabrications.

Because the hydrogen bonding interactions within HOFs are much weaker than those ionic bonds with zeolites and coordination bonds within MOFs, it is challenging to stabilize the HOFs and thus establish their permanent porosities. A large number of organic building molecules with typically rigid backbones^{19,20} and specific functional groups such as guanidinium-sulfonate (GS),^{21–24} pyridyl and carboxyl groups,¹ pyrazole,⁹ pyridone,²⁵ and benzimidazolone²⁶ have been examined for their construction of HOFs, but with limited success to establish their gas/vapor sorption capacities. During our studies based on Wuest's pioneer $work^{27-30}$ to construct robust HOFs for gas separation and enantioselective separation of chiral alcohols, we have realized that 2,4-diaminotriazinyl (DAT) group is indeed a very powerful building unit to assemble and stabilize porous HOFs because of their multiple sites for the hydrogen bonding. The success of this DAT unit for the construction of porous HOFs has motivated us to explore new simple organic building units to develop the HOF chemistry.³¹ Given the fact that melamine has very similar structure features at the DAT motif, we have been wondering

about the possibility to make use of melamine as the molecular building unit to construct porous HOFs. Literature studies show that melamine sometimes does co-crystallize with other organic molecules to form "porous" structures, but none of them has been realized to have flexible/robust structures even for solvent exchanges.^{32–34} Herein we report the solvent dependent crystal structures of melamine in which both crystallographically condensed and porous structures have been discovered. More importantly, we realized that these two phases can be reversibly transformed with each other, while the porous one can sustain the solvent exchange.

EXPERIMENTAL SECTION

All reagents and solvents were used as received from commercial suppliers without further purification. ¹H NMR spectra were recorded on a Varian Mercury 500 MHz spectrometer. The coupling constants were reported in hertz. Thermogravimetric analyses (TGA) were measured using a Shimadzu TGA-50 analyzer under a nitrogen atmosphere at a heating rate of 5 °C min⁻¹. Powder X-ray diffraction (PXRD) patterns were recorded by a Rigaku Ultima IV diffractometer operated at 40 kV and 44 mA with a scan rate of 1.0 deg min⁻¹. The crystallographic measurement of MA-1-H₂O, MA-2-DMF, and MA-2-Toluene were collected at 100 (2) K on a Rigaku X-ray diffractometer system equipped with a Mo-target X-ray tube ($\lambda = 0.71073$ Å) operated at 3000 W power (50 kV, 40 mA) with Saturn 724 CCD detector. The data sets were corrected by empirical absorption correction using spherical harmonics, implemented in the SCALE3 ABSPACK scaling algorithm. The structure was solved by direct methods and refined by full matrix least-squares methods with the SHELX-97 program package. The H atoms on C atoms were

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generated geometrically. X-ray crystallographic data is summarized in Table 1.

Table 1. Crystallographic Data for the MA-1- H_2O , MA-2-DMF, and MA-2-Toluene

Identification code	MA-1-H ₂ O	MA-2-DMF	MA-2-Toluene
Empirical formula	$C_3H_6N_6$	$\begin{array}{c} C_3H_6N_6\cdot\\ C_3H_7NO\end{array}$	$2C_{3}H_{6}N_{6}\cdot C_{7}H_{8}$
Formula weight	126.14	199.21	344.38
Temperature/K	100(2)	100(2)	100(2)
Crystal system	monoclinic	tetragonal	tetragonal
Space group	$P2_1/n$	$I4_1/a$	<i>I</i> 4 ₁
a/Å	7.2789(3)	12.150(2)	12.236(6)
b/Å	7.4799(3)	12.150(2)	12.236(6)
c/Å	10.332(4)	24.405(2)	24.574(3)
$\alpha/^{\circ}$	90.00	90.00	90.00
$\beta/^{\circ}$	108.495(4)	90.00	90.00
$\gamma/^{\circ}$	90.00	90.00	90.00
Volume/Å ³	533.46(4)	3602.7(9)	3679.5(6)
Ζ	4	16	8
$D_{\rm cal}~({\rm g~cm^{-3}})$	1.570	1.469	1.243
$\mu \ (\mathrm{mm}^{-1})$	0.117	0.069	0.086
F(000)	264	1056	1456
Reflections collected	1947	9049	9511
Data/restraints/ parameters	954/0/106	2053/0/100	1881/1/101
GOF	1.769	1.016	1.981
Final R indexes $[I > 2\sigma(I)]$	$\begin{array}{l} R_1 = 0.0856, \mathrm{w}R_2 \\ = 0.1802 \end{array}$	$\begin{array}{l} R_1 = 0.0850, \mathrm{w}R_2 \\ = 0.1756 \end{array}$	$R_1 = 0.1025, wR$ = 0.1758
Final <i>R</i> indexes [all data]	$\begin{array}{l} R_1 = 0.0869, \mathrm{w}R_2 \\ = 0.1818 \end{array}$	$\begin{array}{l} R_1 = 0.1000, \mathrm{w}R_2 \\ = 0.1835 \end{array}$	$R_1 = 0.1036, wR$ = 0.1954
Largest diff. peak/ hole/e Å ⁻³	0.53/-0.53	0.28/-0.27	0.20/-0.45

Synthesis of MA-1-H₂O. 50 mg Melamine (MA) was dissolved in 6 mL H₂O under heating. The resulting solution was cooled to room temperature and filtered. The filtrate was allowed to evaporate at room temperature for 2 days. Colorless block-shaped crystals were obtained in 91% yield.

Synthesis of MA-2-DMF. 50 mg Melamine (MA) was dissolved in 6 mL DMF solvent under heating. The resulting solution was cooled to room temperature and filtered. The filtrate was allowed to evaporate at room temperature for 1 day. Large colorless square-shaped crystals were obtained in 89% yield.

Solvent Soaking Experiment. A small amount of as-synthesized **MA-2-DMF** crystals were soaked in 10 mL different solvent (CH_2Cl_2 , acetone, toluene, EtOH, and MeOH), replacing the soaking solution every 24 h for 3 days. After soaking, the samples were filtered and placed in inert atmosphere until further analysis. The isolated samples are named as **MA-2-Solvent** (solvent = CH_2Cl_2 , acetone, toluene, EtOH, and MeOH).

RESULTS AND DISCUSSION

Single crystals of MA-1-H₂O and MA-2-DMF were easily recrystallized from H₂O and DMF, respectively (Figure S1). The single crystal of MA-2-Toluene was obtained by solvent exchange of MA-2-DMF with toluene. Their phase purities were confirmed by the powder X-ray diffraction patterns which match well with their simulated ones from X-ray single crystal structures (Figure S2). Thermogravimetric analysis (TGA) indicates that there is no solvent molecule within MA-1-H₂O. The weight losses are 36.7% for MA-2-DMF and 26.7% for MA-2-Toluene between 70 and 120 °C, corresponding to the lost DMF molecules (calc. 36.6%) and toluene molecules (calc. 26.8%), respectively, which are encapsulated in their void



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Figure 1. TGA curves of MA-1-H₂O (red), MA-2-DMF (black), MA-2-toluene (blue), MA-3-Acetone (green), MA-2-CH₂Cl₂ (purple), MA-2-EtOH (pink), and MA-2-MeOH (brown).



Figure 2. X-ray crystal structure of $MA-1-H_2O$ showing (a) local hydrogen bonding environments of one MA molecule (yellow); (b) packing of the 3D framework.

spaces of the crystal structures (Figure 1). The decomposition temperature for MA-1-H₂O (190 °C) is much lower than that of MA-2-DMF and MA-2-Toluene (250 °C) (Figure 1). NMR studies of the dissolved MA-2-DMF and MA-2-Toluene show that there exist an equivalent DMF solvent molecule and half an equivalent toluene per melamine molecule within their frameworks, respectively (Figure S3). The chemical formulas of MA-1-H₂O, MA-2-DMF, and MA-2-Toluene are determined to be $C_3H_6N_6$, $C_3H_6N_6$ ·C₃H₇NO, and $C_3H_6N_6$ ·0.5C₇H₈, respectively, based on their single crystal structures, TGA and NMR data.

X-ray Crystal Structure of MA-1-H₂O. The similar structures of MA-1-H₂O have been reported in previous literature.³⁵⁻³⁷ The crystallization methods in the literature and here are essentially identical (using water as solvent to



Figure 3. X-ray crystal structure of **MA-2-DMF** showing (a) the local hydrogen bonding environments of MA molecules; (b) threedimensional hydrogen-bonded organic framework; (c) packing of the framework in which the solvent DMF molecules (CPK models) reside in the channels of 3D hydrogen-bonded organic framework along *a* axis.

recrystallize melamine). The reported structure of melamine crystallizes in $P2_1/a$ space group with slightly different unit cell parameters (a = 10.54, b = 7.45, c = 7.25, $\beta = 112.03$) compared with that of MA-1-H₂O (Table 1), attributed to the different crystallography setting. As shown in Figure 2a of MA-1-H₂O, each MA molecule connects with four neighboring MA molecules through 8-fold hydrogen bonds to form a three-dimensional HOF (Figure 2b). The dihedral angles between planes of neighboring MA aromatic rings in MA-1-H₂O are 34° or 180°. MA-1-H₂O is a condensed structure in which there is no solvent molecule inside the pores. The hydrogen bonds and dihedral angles between planes of neighboring MA in MA-1-H₂O and reported melamine are comparable.

X-ray Crystal Structure of MA-2-DMF. The X-ray crystal structure of MA-2-DMF is completely different from that of MA-1-H₂O. Each MA molecule forms 9-fold hydrogen bonds with six nearby MA molecules and one DMF molecule (Figure 3a). Close examination on the structure of MA-2-DMF reveals that there exist two sets of double-layered MA molecules



Figure 4. X-ray crystal structure of **MA-2-Toluene** showing (a) the local hydrogen bonding environments of MA molecules; (b) packing of the framework in which the solvent toluene molecules (CPK models) reside in the channels of 3D hydrogen-bonded organic framework along the *a* axis.





orthogonal to each other (Figure 3b). The dihedral angles between planes of neighboring MA aromatic rings in MA-2-DMF are 90° or 180°. Packing diagram shows that the DMF molecules reside in the two-dimensional channels of about 5.0 Å in diameter along a and b axes (Figure 3c).

X-ray Crystal Structure of MA-2-Toluene. The X-ray crystal structure of MA-2-Toluene crystallizes in a space group $(I4_1)$, which is close to that of MA-2-DMF $(I4_1/a)$. The local environment of the MA molecule in MA-2-Toluene is similar to that of the MA molecule in MA-2-DMF. The hydrophobic toluene molecule interacts with MA molecule by a π - π

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motif	donor-H…acceptor	H…A (Å)	D–H…A (Å)	angle (deg)		
MA-1-H ₂ O						
II	N3–H3A…N5 ⁱ	2.22	3.06	162		
Ι	N2-H2A····N4 ⁱⁱ	2.08	3.01	175		
Ι	N1-H1B····N6 ⁱⁱⁱ	2.20	3.08	163		
symmetry codes	(i) $1/2 - x$, $-1/2 + y$, $1/2$	z - z; (ii) $-1 - x, 1 - y, -z$	z; (iii) -x, -y, -z			
MA-2-DMF						
Ι	N5–H5B…N1 ⁱ	2.19	3.05	178		
Ι	N4-H4B····N3 ⁱⁱ	2.12	2.98	178		
II	N5–H5A…N2 ⁱⁱⁱ	2.32	3.09	148		
II	N6-H6A…N2 ^{iv}	2.40	3.22	161		
symmetry codes	(i) $1/2 + x$, y, $1/2 - z$; (ii)	(i) $1/2 + x$, y, $1/2 - z$; (ii) $-1/2 + x$, y, $1/2 - z$; (iii) $5/4 - y$, $1/4 + x$, $1/4 - z$; (iv) $3/4 - y$, $1/4 + x$, $1/4 + z$				
MA-2-Toluene						
Ι	N4-H4B····N9 ⁱ	2.08	3.00	168		
Ι	N2-H2B···N11 ⁱⁱ	1.99	2.90	177		
Ι	N1-H1A…N12 ⁱⁱⁱ	2.87	2.97	88		
Ι	N3-H3A···N10 ^{iv}	2.07	2.97	177		
II	N1 -H1B…N8	2.58	2.90	103		
II	N7 ⁱⁱⁱ -H7A…N8	2.25	3.12	165		
symmetry codes	(i) y, $1/2 - x$, $1/4 + z$; (ii)) 1 + y, 1/2 - x, 1/4 + z;	iii) $1/2 - y$, x , $-1/4 + z$; (iv) 1	/2 - y, -1 + x, -1/4 + z		

Table 2. Summary of Hydrogen Bonding Motifs Revealed in MA HOFs and Hydrogen Bonding Interactions Details and Geometries

Scheme 2. Schematic Illustration of Crystal-Crystal Transformations of the Melamine Based HOFs



stacking interaction (3.2 Å in distance). The packing diagram shows that the toluene molecules also reside in channels along the a and b axes in the 3D hydrogen-bonded organic frameworks (Figure 4b).

Basic Hydrogen Bonding Motifs. The detailed X-ray crystal structural studies of these HOFs of MA enable us to rationalize some basic hydrogen bonding motifs to connect MA molecules. As summarized in Scheme 1, there are two basic hydrogen bonding motifs (I) and (II). In motif I, the neighboring MA molecules are parallel with each other; while in motif II, the two nearby MA molecules are in orthogonal positions. Some more structural features of the hydrogen bonding interactions are listed in Table 2 as well.

Crystal Transformation by Solvent Soaking. Solventinduced crystal transformation experiments were conducted at room temperature by soaking the crystals of **MA-2-DMF** in different solvents for 3 days: acetone, CH₂Cl₂, toluene, EtOH, and MeOH. The solid samples isolated after solvent soaking are named as **MA-2-Solvent** (solvent = acetone, CH₂Cl₂, toluene, EtOH, and MeOH). During the soaking experiments, no obvious dissolution-precipitation process was observed in any solvent.

In toluene, the crystal sample of MA-2-DMF can keep its crystallinity very well. Optical microscopy revealed that single crystals did not exhibit any change in transparency or crystal integrity after the soaking experiment. Single crystal X-ray diffraction analysis revealed that the lattice parameters of MA-**2-toluene** had changed to be a = 12.236 Å, b = 12.236 Å, c =24.574 Å in I41, which is also slightly larger than the one of MA-2-DMF (Table 1). The NMR data indicated that the solvent exchange process can be finished in 48 h (Figure S3a). More surprisingly, after immersion of the single crystal of MA-2-toluene in DMF for 3 days, the NMR data showed that the toluene molecules in MA-2-toluene can be reversely exchanged by DMF (Figure S3b). Single crystal X-ray diffraction of the crystal sample revealed that the framework reverted back to the original $I4_1/a$ unit cell. This reversible single crystal to single crystal transformation (SCSCT) is very rare in HOF materials.

In acetone and CH_2Cl_2 , although the crystal sample of MA-2-DMF becomes opaque in 5 h, the acetone and CH_2Cl_2 exchanged MA-2 still keep the high crystallinity and have the same basic similar structures as MA-2-DMF, as confirmed from their PXRDs which match well with that of MA-2-DMF (Figure S2). The NMR data also indicate that the DMF guests in MA-2-DMF can be fully exchanged with acetone or CH_2Cl_2 (Figure S4). However, soaking MA-2-DMF in MeOH and EtOH leads to completely different structures. Given the fact that the PXRD patterns of MA-2-MeOH and MA-2-EtOH are quite similar to that of MA-1-H₂O, structures of MA-2-MeOH and MA-2-EtOH might be close to that of the condensed MA-1-H₂O as well. Apparently, the much stronger hydrogenbonded interactions with MeOH/EtOH with melamine lead to such framework transformations. The NMR data suggested that no solvent molecules are included in the MA-2-MeOH and MA-2-EtOH because of the nonporous nature of the structures (Figure S4).

In summary, two hydrogen-bonded organic frameworks (HOFs) of the melamine (MA), MA-1-H₂O and MA-2-DMF, have been crystallized in H₂O and DMF, respectively. The Xray crystal structural studies indicate that the formation of different HOF isomers is attributed to the different hydrogenbonded patterns of melamine molecules: structurally, MA-1-H₂O and MA-2-DMF are condensed and porous, respectively. The porous MA-2-DMF sustains the porous feature under the solvent exchange by acetone, dichloromethane (CH₂Cl₂), and toluene, while it transforms into the condensed state under methanol (MeOH) and ethanol (EtOH). Furthermore, MA-2-DMF and MA-2-Toluene can undergo reversible single crystal-single crystal transformation with retention of the porous hydrogen-bonded organic frameworks (Scheme 2). We are working on the construction of even more robust melamine based HOFs for their recognition of small molecules and their applications on gas separations.

ASSOCIATED CONTENT

S Supporting Information

X-ray crystallographic files (CIF), PDF of check-cif, power Xray diffraction analysis (PXRD), and NMR data of MA-1-H₂O, MA-2-DMF MA-2-Toluene, MA-2-Acetone, MA-2-CH₂Cl₂, MA-2-MeOH, and MA-2-EtOH. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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