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# Control by one drop of solvent: selective preparation of guest release/trap-triggered interconvertible molecular crystals

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Interconvertible molecular crystals  $1_{close}$  and  $1_{open}$  composed of 4pyridyl-1,3-indanedione dimer 1 were selectively obtained. Thermal removal of solvent molecules in  $1_{open}$  afforded  $1_{close}$ . Further dipping of  $1_{close}$  in a specific solvent reproduced  $1_{open}$ . No crystallinity loss was observed even though both processes involved drastic change of molecular packing arrangements.

Porous materials, represented by the metal organic frameworks or porous coordination polymers (MOFs or PCPs),<sup>1</sup> covalent organic frameworks (COFs)<sup>2</sup> and hydrogen-bonded organic frameworks (HOFs),<sup>3</sup> are widely studied because of their high potential towards the practical uses such as gas sorption, molecular separation, sensing and ion/electron conduction. They possess structural rigidity and high stability against the adsorption/desorption process of the guest molecules. On the other hand, organic crystals having solvent incorporated spaces, which are formed exclusively by the soft van der Waals type interactions, in most case, easily undergo a structural collapse on removal of the guest solvent molecules. Very few examples retain their crystallinity during the process to exhibit permanent porosity<sup>4,5</sup> or even show drastic structure change<sup>6,7</sup> (Fig. 1a). Nevertheless, organic crystals are attractive as they are easily renewed by the simple recrystallization process and especially the ones showing drastic structure change by guest trapping/releasing possess a great potential to provide a platform for the new type of "structurally soft" functional materials though such systems are still quite rare<sup>6,7</sup>. Herein, we report a new molecular crystalline system composed of Xshaped 4-pyridyl-1,3-indanedione dimer (1). In the system, 1 selectively gives the two specific packing structures, tightly packed  $\mathbf{1}_{close}$  and solvent-included  $\mathbf{1}_{open}$  (Fig. 1b). The structural selectivity from  $\mathbf{1}_{close}$  to  $\mathbf{1}_{open}$  was easily switched by simple addition of a drop of various kinds of "trigger" solvent in the crystallization condition. Interestingly, thermal removal of the



Fig. 1 a) Three types of solvent-incorporated molecular crystals and their behaviour in solvent trapping-releasing process. b) Formation of two crystal structures  $\mathbf{1}_{dose}$  and  $\mathbf{1}_{open}$  and their interconversion by solvent trapping-releasing.

solvent molecules from  $\mathbf{1}_{open}$  afforded a densely-packed crystal  $\mathbf{1}_{close}$  via dynamic structure transformation without any crystallinity loss though  $\mathbf{1}_{open}$  is stabilized by exclusively weak intermolecular interactions. Whereas, dipping  $\mathbf{1}_{close}$  in a specific solvent afforded again  $\mathbf{1}_{open}$  without getting dissolved.

Synthesis of the target dimer **1** was achieved as illustrated in Scheme 1. 4-pyridyl-1,3-indanedione monomer, which existed in the twitter-ionic form in DMSO- $d^6$  as observed in its <sup>1</sup>H NMR spectrum (Fig. S1, ESI) was prepared with following the reported procedure<sup>8</sup>. This time we successfully obtained single

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crystals of the monomer and revealed its twitter-ionic structure by X-ray analysis (Fig. S3, ESI). An oxidative homocoupling of the Na enolate of the monomer by  $PhI(OAc)_2$  gave 4-pyridyl-1,3indanedione dimer **1** in a quantitative yield<sup>9</sup>.

1 was easy to form two kinds of crystals with the totally different packing structures depending on the crystallization conditions. We used vapour diffusion method using  $CH_2Cl_2/EtOH$  as the general condition (ESI). When 1 was crystallized by the above condition, a well-packed structure  $\mathbf{1}_{close}$  was obtained (Fig. 2). In its crystal structure, the 4-pyridyl-1,3-indanedione skeleton was a crystallographically independent unit and the C6-C6 distance, which connected the two 4-pyridyl-1,3-indanedione units, was 1.58 Å, slightly longer than the typical C(sp<sup>3</sup>)-C(sp<sup>3</sup>) bond distance because of steric hindrance<sup>10</sup>. All the molecules of **1** were in a distorted H-shape and they engaged 1-dimensionally with each other along the c axis with forming  $\text{CH}{\cdots}\pi$  interactions between C13 and C14 (3.52 Å) (Figs. 2b, c). They interacted with each other by the  $\pi$ - $\pi$  stacking at the phenyl rings of their indanedione skeletons and  $CH\cdots\pi$  interactions between C5 and C11, with the intermolecular distances of 3.44 Å and 3.44 Å, respectively (Fig. 2b). Due to these interactions, the crystal was closely packed and did not have any spaces to accommodate other molecules such as solvents.

In contrast, addition of a drop of another solvent in the same crystallization setting for  $\mathbf{1}_{\text{close}}$  afforded a totally different packing of the single crystals  $\mathbf{1}_{open}$ .  $\mathbf{1}_{open}$  possessed 1dimensional (1D) channels that trapped solvent molecules within them. Various kinds of solvents such as hexane, pentane, decane, cyclohexane, t-BuOH, (CH<sub>2</sub>Cl)<sub>2</sub> and benzene were applied as the "trigger" to switch the packing structure from  $\mathbf{1}_{close}$  to  $\mathbf{1}_{open}$  (vide infra). Fig. 3 shows the packing structure of  $\mathbf{1}_{\text{open}}$  prepared by addition of hexane.  $\mathbf{1}$  in  $\mathbf{1}_{\text{open}}$  also had the  $\mathcal{C}_2$ symmetrical structure and the central C6-C6 distance was 1.57 Å, which was almost the same as that in  $\mathbf{1}_{close}$ . 1D-channels were formed along the c axis with an open-window size of 5.3 Å  $\times$  7.0 Å and the total open volume of 421 Å, that is around 18% of the unit cell volume. The channels were occupied by the severely disordered solvent molecules and their positions and occupancy factors were impossible to be determined precisely by the X-ray crystallographic analysis, suggesting that the solvent molecules dynamically moved within the channel. It is noteworthy that the single crystal with the same packing could also be obtained by a slow evaporation of the CH<sub>2</sub>Cl<sub>2</sub> solution of 1 (Fig. S4, ESI), indicating that the quantitative balance between EtOH and the additive was a key factor for the selective preparation of  $\mathbf{1}_{close}$ and  $\mathbf{1}_{open}$  (vide infra). In the crystal structure of  $\mathbf{1}_{open}$ , several short contacts were observed at C4…N1 (3.29 Å) and C7…O2 (3.39 Å), which can be assigned to the CH…N and CH…O



**Fig. 2** Crystal structures of  $\mathbf{1}_{close}$ . a) Displacement ellipsoid plot of  $\mathbf{1}$  in the crystal structure of  $\mathbf{1}_{close}$  at 50% probability. b) Closed packing pattern viewed from the *b* axis. The red and black dotted lines: CH… $\pi$  interactions; blue arrow:  $\pi$ - $\pi$  interaction. c) 1-dimensionally engaged assembling pattern along the c axis. The pale blue arrow at the bottom corresponds to the vertical one in b). In b), Gray: C; blue: N; red: O. Hydrogen atoms are omitted for clarity.

interactions, respectively. As indicated by their bond lengths, they are much weaker hydrogen bonds than the conventional NH…N and OH…O bonds<sup>11,12</sup>. Although a small overlapping of the pyridine rings (C10…C14: 3.35 Å) was also observed (Figs. 3b, c), practically the  $\mathbf{1}_{open}$  crystals recrystallized by either the vapour diffusion using CH<sub>2</sub>Cl<sub>2</sub>/EtOH with the small portion of the specific solvents or by simple slow evaporation of CH<sub>2</sub>Cl<sub>2</sub> possessed no visible strong interactions unlike the MOFs, COFs or HOFs. Nevertheless, these  $\mathbf{1}_{open}$  crystals were quite stable under air and showed no solvent loss at the ambient under air and showed no solvent loss at the ambient temperature.

To comprehend the molecular trapping ability of  $\mathbf{1}_{open}$ , the amount of trapped solvents was determined by the TGA and the <sup>1</sup>H NMR spectral analysis using hexane-trapped crystalline powder (Fig. S5, ESI). This crystalline powder of 1<sub>open</sub> was obtained by the addition of an excess amount of hexane in the saturated CH<sub>2</sub>Cl<sub>2</sub> solution of **1**. The success of the sample preparation was confirmed by powder X-ray diffraction (PXRD) measurement (Fig. S5, ESI). The TGA revealed the high thermal stability of 1 up to around 250 °C. During this measurement, 6.5% weight-loss due to the elimination of inner solvent was observed between 70 °C and 108 °C, and then the TGA track became flat until it exhibited a large weight loss due to the decomposition of 1 around 250 °C. When the inner solvent was assumed to be only hexane, the 6.5% weight-loss indicated that 36 mol% of hexane was incorporated in the channel (Fig. S6a, ESI). This was supported by their <sup>1</sup>H NMR data, which indicated a hexane content of 35 mol% (Fig. S6b, ESI). This was variable, depending on the crystallization process, especially the amount of hexane added (Fig. S7, ESI). However, even a long soaking (for more than a month) of either the single crystals or the crystalline powder of 1<sub>open</sub> in hexane at the ambient temperature did not change the trapped hexane content and it did not exceed more than 40 mol% of the total amount of the indanedione dimer. This also indicated the dynamic motion of

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**Fig. 3** Crystal structures of  $\mathbf{1}_{open}$  obtained via the addition of hexane. a) Displacement ellipsoid plot of  $\mathbf{1}$  in the crystal structure of  $\mathbf{1}_{open}$  at 50% probability. b) Packing structure viewed from the *c* axis and *c*) from the *a* axis. In b) and c), Gray: C; blue: N; red: O. Green dotted line: CH…N interaction; blue dotted line: CH…O interaction; red dotted line:  $\pi$ - $\pi$  interaction. Hydrogen atoms are omitted for clarity.

the solvent molecules within the channel and the possibility of the presence some empty spaces in  $\mathbf{1}_{open}$  to be used for small molecule entrapment. It was confirmed by the CO<sub>2</sub> gas sorption experiment which clearly showed a further gas encapsulation in the 1-D channel of  $\mathbf{1}_{open}$  (Fig. S8, ESI). Meanwhile, neither the TGA nor the <sup>1</sup>H NMR data of  $\mathbf{1}_{close}$  showed the presence of any solvent molecules, thus corresponding well with the single crystal structure of  $\mathbf{1}_{close}$ .

Further investigation of **1**<sub>open</sub> was performed using variabletemperature powder X-ray diffraction (VT-PXRD) analysis of the crystalline powder of  $\mathbf{1}_{\text{open}}\text{,}$  which was prepared by the same method as used in TGA analysis. The experimental results involved the gradual change of the PXRD patterns to reveal that the structure change from the initial  $\mathbf{1}_{\text{open}}$  structure to the  $\mathbf{1}_{\text{close}}$ one on heating, while retaining the crystallinity (Fig. 4a). Until the temperature reached 92 °C, the observed peaks were the ones corresponding to  $\mathbf{1}_{\text{open}}.$  At the temperatures above 92 °C, however, new patterns assignable to  $\mathbf{1}_{\text{close}}$  were observed and their intensity gradually increased while the peak corresponding to 1<sub>open</sub> became weaker. At 107 °C, the observed PXRD pattern was same as that of  $\mathbf{1}_{close}$ , and the pattern of  $\mathbf{1}_{open}$ could not be found. On analysing the TGA results and the changes in the PXRD patterns, it is clear that  $\mathbf{1}_{open}$  transformed into  $\mathbf{1}_{close}$  by the loss of solvent molecules. During this process, no intermediate peaks were observed. Even crystal to crystal transformation in van der Waals interaction-based molecular crystal is still rare,<sup>5-7</sup> only few molecules are reported which involve the drastic host-packing change.<sup>6,7</sup> Further heating resulted in another crystalline phase with drastic crystallinity loss at more than 246 °C (Fig. S9, ESI). The crystallinity of the sample was finally lost at more than 280 °C, indicating the decomposition of1 as observed in the TGA. The heating experiment using single crystals of  $\mathbf{1}_{\text{open}}$  also showed the crystalline to crystalline transformation, although the resulting crystals were heavily cracked and were no longer applied to single crystal X-ray analysis. On the other hand,  $\mathbf{1}_{close}$  retained its single crystal nature even above 200 °C.

The structural features of both the crystals inspired us to test the potential of  ${\bf 1}$  as a source of reversible guest



**Fig. 4** PXRD patterns of **1**. a) VT-PXRD data of the crystal powder of  $\mathbf{1}_{open}$ . The heating rate was 3 °C/min. The patterns of  $\mathbf{1}_{open}$  and  $\mathbf{1}_{close}$  are simulated from each single crystal data. b) Changes in PXRD patterns of the crystal powder of  $\mathbf{1}_{close}$  after soaking in hexane and pentane. The initial  $\mathbf{1}_{close}$  powder was prepared by heating  $\mathbf{1}_{open}$  at the rate of 3 °C/min until 170 °C.

trapping/releasing crystalline system involving significant structure change, while retaining crystallinity. In general, the lattice stabilization energy is quite high in the case of the tightlypacked molecular crystals containing no solvent molecules, and therefore, a guest insertion in the non-porous packing structure is difficult to occur unless the framework has permanent porosity<sup>4,5,13</sup> (Fig. S10, ESI). However, the present system exhibited reversible guest trapping/releasing property. The crystalline powder of  $\mathbf{1}_{close}$  prepared by heating of  $\mathbf{1}_{open}$  at the rate of 3 °C/min until 170 °C was left in hexane or pentane at both 25 °C and 60 °C in a sealed condition. After 1 day, both the samples exhibited no structural change. However, after 2 days, a partial change in the PXRD pattern of the sample left at 60 °C was observed, resulting in the representative peak of  $\mathbf{1}_{\mathsf{open}}$ phase (Fig. 4b). The change in the diffraction pattern clearly indicated the recovery of the porous structure by the re-take of the solvent molecules. Interestingly, when pentane was used instead of hexane, a clearer peak change was observed. This meant that the porous  $\mathbf{1}_{open}$  had preferences when it traps solvent molecules. Other solvents such as cyclohexane and benzene were never trapped (Fig. S11, ESI). It should be mentioned that molecule **1** is totally insoluble both in hexane and pentane, indicating that the patterns corresponding to  $\mathbf{1}_{open}$ , which appeared after soaking, are not formed by the simple recrystallization on the crystalline powder surface.

The investigations so far clearly indicated the importance of solvents for the packing selectivity. We focused on what the key is to switch the structure between  $\mathbf{1}_{close}$  and  $\mathbf{1}_{open}$ . Table S1 (ESI) shows the solvents, which were used as the additive for crystallization of  $\mathbf{1}$  along with the empirical parameters of solvent polarity,  $E_{T}(30)$ .<sup>14</sup> The experimental results showed that the presence of relatively polar solvents, such as EtOH, CH<sub>3</sub>CN and cyclohexanol, resulted in  $\mathbf{1}_{close}$ . In contrast, less polar solvents such as hexane, pentane, decane, cyclohexane, *t*-BuOH, CH<sub>2</sub>Cl<sub>2</sub>, (CH<sub>2</sub>Cl)<sub>2</sub>, benzene, and toluene resulted in  $\mathbf{1}_{open}$ , which trapped the corresponding solvents in the channel. This result was reasonable considering that the inner-channel was surrounded by the hydrophobic carbon skeletons. Indeed, <sup>1</sup>H

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NMR measurement results of the powder crystals of  $\mathbf{1}_{open}$  obtained with various "trigger" solvents revealed the successful trapping of them (Fig. S12, ESI). EtOAc was an exceptional solvent that resulted in  $\mathbf{1}_{close}$  despite their low polarity and smaller molecular size than the channel diameter. One possible reason is that EtOAc might form a specific complex in the system to disturb the easy trapping within the 1D channel of  $\mathbf{1}_{open}$ .

Further investigation revealed that the 1D channel in  $1_{open}$  possessed the solvent preference among hexane, benzene, and cyclohexane (Fig. S13, ESI). <sup>1</sup>H NMR data of the CDCl<sub>3</sub> solution of the single crystals recrystallized from the three solvent mixture (1:1:1 molar ratio) showed that hexane was the most preferred one and that the preference of benzene and cyclohexane was not significant (31% total solvent content, hexane/benzene/cyclohexene = 62:26:12). From these results, it is assumed that the chain-like molecules have a higher preference, which fit in the cylinder-like 1D channel, than the other cyclic molecules, as also observed in the pillar[*n*]arenes<sup>15</sup>.

#### Conclusions

We found that 4-pyridyl-1,3-indanedione dimer 1 formed two kinds of crystals, densely-packed  $\mathbf{1}_{\text{close}}$  and solvent-incorporated 1<sub>open</sub>. They were selectively prepared by utilizing the "trigger solvent" from the same  $CH_2Cl_2/EtOH$  solvent system. Despite that  $\mathbf{1}_{\text{open}}$  possessed no strong intermolecular interactions within the structure,  $\mathbf{1}_{open}$  was stable under air. The thermal removal of the solvent molecules from the crystalline powder sample of  $\mathbf{1}_{\text{open}}$  resulted in crystal  $\mathbf{1}_{\text{close}}\text{,}$  which again afforded the initial open state on dipping the sample powder in hexane or pentane. Even though these processes involved significant packing structure change, the system retained high crystallinity. Because of simple structure and the thermal stability of 1, its delivertization might be easy. We expect that pore environment and the emergence of various physical properties, as realized in MOFs and other porous materials, is easily tuned by the simple molecular-level modification in this indanedione dimer-based system.

#### **Conflicts of interest**

There are no conflicts to declare.

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