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# Dynamic self-assembled polymer: HCl responsive inversion of supramolecular polymer handedness†

Arpita Paikar and Debasish Haldar \*

A series of discotic tricarboxyamides with varied amino acid side arm functionality and their HCl responsive diverse self-assembly behavior and formation of dynamic polymer is studied. Discotic trisamide **1** obtained from Boc protected L-Lys self-assembled into a long fibrillar aggregate-like supramolecular P helical polymer. However, on addition of HCl, the supramolecular helical handedness of the assemblies is completely inverted. Irrespective of the chiral centres configuration, supramolecular chiral bias is arising from molecular conformations. FE-SEM reveals the formation of right handed entangled polymeric fibers. However, left handed entangled fibers have appeared on addition of 0.12 M HCl. The trisamide **2** containing Boc protected L-Trp exhibits disk like morphology both in the presence and absence of 0.12 M HCl and does not show a change of supramolecular handedness. This demonstrates how remarkably distinct morphologies originate from stimuli responsive building blocks assembled in a subtly different manner.

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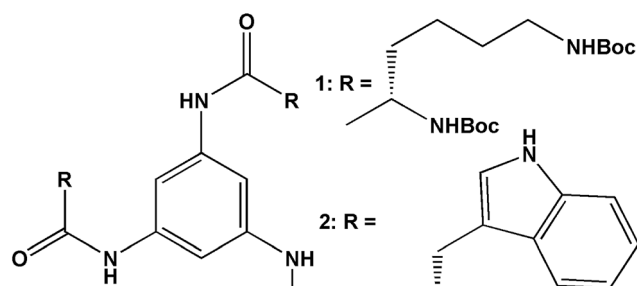
## Introduction

Supramolecular polymers are highly interesting for their dynamic character. Tuning the nature, directionality and spatial arrangement of stimuli responsive building blocks in self-assembled systems provides chiral supramolecular polymers with new features compared to the bulk materials.<sup>1</sup> Understanding the chirality of supramolecular systems<sup>2–4</sup> is highly important for biological sciences as well as material sciences mainly in the fields of chiral recognition and sensing,<sup>5</sup> optoelectronic materials,<sup>6</sup> chiroptical switches,<sup>7,8</sup> asymmetric catalysts<sup>9,10</sup> and separation processes,<sup>11</sup> metal coordination,<sup>12</sup> redox reactions,<sup>13</sup> host–guest interactions,<sup>14</sup> anions,<sup>15,16</sup> solvent polarity,<sup>17</sup> concentrations variations,<sup>18,19</sup> light irradiation,<sup>20,21</sup> high pressure<sup>22</sup> and temperature<sup>23</sup> on large discotic,<sup>24</sup> dendritic,<sup>25,26</sup> and gelator molecules.<sup>27</sup> Feringa, van Esch and coworkers have reported the cyclohexane based C<sub>3</sub> symmetric discotic hydrogelator containing three amino acid side groups.<sup>28–31</sup> Meijer and coworkers have discussed analogous systems with 1,3,5-benzene tricarboxylic acid core.<sup>32–36</sup> For these compounds, tuning experimental conditions promotes the selection of a predominant assembly.<sup>37</sup> Changing the stereochemistry of the building blocks as well as experimental condition can control the supramolecular chiral amplification.<sup>38</sup> However, the examples of designer supramolecular assemblies that can adopt diverse well-defined

chiral nanostructures by simply changing the molecular conformation are relatively rare.<sup>39</sup>

Herein, we report the conformational preferences in the trisamide system over the configuration of the chiral center to determine the helical handedness of supramolecular polymers. We have designed and synthesized a series of trisamides containing Boc protected L-lysine (Lys), L-tryptophan (Trp) and benzene-1,3,5-triamine and investigated their structures and stimuli responsive behavior. Interestingly the trisamide **1** containing (Boc)<sub>2</sub>-L-Lys formed one-dimensional supramolecular helical polymer. On protonation, the supramolecular helical handedness of the polymeric assemblies has inverted. However, the analogue **2** containing (Boc)-L-Trp exhibits disk like morphology both in presence and absence of 0.12 M HCl and does not show change of supramolecular handedness.

Starting from commercially available 1,3,5-trinitrophenol (picric acid), 1,3,5-triaminobenzene was synthesized in single step in 86% yield, by refluxing with ammonium formate and Zn dust in methanol at 60 °C for 1 h (Scheme 1, ESI†). The



Scheme 1 The trisamides **1** and **2**.

Department of Chemical Sciences, Indian Institute of Science Education and Research Kolkata, Mohanpur, West Bengal 741252, India. E-mail: [deba\\_h76@yahoo.com](mailto:deba_h76@yahoo.com); [deba\\_h76@iiserkol.ac.in](mailto:deba_h76@iiserkol.ac.in); Fax: +91 3325873020; Tel: +91 3325873119

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Fig. 2 The UV-Vis titration of (a) trisamide 1 and (b) trisamide 2 methanol solution ( $1.984 \times 10^{-4}$  M) and ( $3.451 \times 10^{-5}$  M) respectively with gradual addition of 0.12 M HCl. Inset: colour change by addition of HCl.

broad. The amide I bands are now split in 1 : 2 ratio and appear at 1689 and 1640  $\text{cm}^{-1}$  (Fig. 3a). The intensity of amide II band at 1523  $\text{cm}^{-1}$  is very low (Fig. 3a). Fig. S5, ESI<sup>†</sup> shows the FT-IR spectra of 2.

Moreover, to investigate the effect of HCl on trisamide 1, the NMR titration experiments were performed. To a solution of compound 1  $1.082 \times 10^{-2}$  (M) in  $\text{CD}_3\text{OD}$  the 0.57 (M) HCl solution was gradually added by an amount of 10  $\mu\text{L}$  at room temperature and the NMR spectra were recorded. The results of the NMR titrations of trisamide 1 have been shown in the Fig. 3b. The NMR titrations data show that the NH protons get exposed to the acid and there are significant shift of NH protons for the trisamide 1 with gradually increase in HCl concentration. From the stack plot, the core amide NH exhibits upfield shift suggest the strong hydrogen bond formation. Whereas the protons of central aromatic core show no shift *i.e.* the stacking structure is not changing. The Boc protected NHs of Lys exhibits downfield shift as well as broadening suggest that these NHs are open to acid. Fig. S6, ESI<sup>†</sup> shows the acid dependence NH chemical shifts of 2.

In order to gain more information on the structural changes of these trisamides in presence of acid, we performed circular dichroism (CD) measurements. The positive cotton effect observed (185 nm) in the CD spectra of trisamide 1 in methanol



Fig. 3 (a) FT-IR spectra of trisamide 1 in absence of HCl (black) and in presence of HCl (red). (b) The acid dependence of NH chemical shifts of trisamide 1 at varying concentrations of HCl. Star: core CONH, circle: core aromatic Hs, square: Lys side chain NHs, triangle: Lys NHs.

( $1.474 \times 10^{-5}$  M) is suggesting a supramolecular right handed helix (Fig. 4a) which is consistent with the handedness of the helices formed by the tricarboxamide containing *L*-methionine methyl ester with 3 : 0 conformation in the crystalline state.<sup>41</sup> On addition of HCl, ( $4 \times 10^{-5}$  M) the helical handedness of the supramolecular polymer reverses to negative cotton effect (Fig. 4a). The CD spectra is suggesting a left handed supramolecular polymer which is consistent with the handedness of the helices formed by the tricarboxamide containing  $\beta$ -alanine methyl ester with 2 : 1 conformation in crystal.<sup>40</sup> This suggest that only 2.713 equivalent HCl is enough for the chirality change of tricarboxamide 1. However the trisamide 2 in methanol ( $1.474 \times 10^{-5}$  M) has a positive band at 185 nm which does not change after addition of HCl ( $4 \times 10^{-5}$  M) (Fig. 4b). There also a negative band at 200 nm and 217 nm which does not shift in presence of acid (Fig. 4b). The CD experiments also performed with trisamide containing achiral  $\beta$ -alanine and  $\gamma$ -aminobutyric acid in absence and presence of HCl. The  $\gamma$ -aminobutyric acid containing trisamide shows a positive band at 210 nm which does not shift in presence of acid (Fig. S7, ESI<sup>†</sup>). Another trisamide containing  $\beta$ -alanine shows a negative band at 200 nm and after addition of HCl it remain in the same conformation (Fig. S8, ESI<sup>†</sup>). To examine the supramolecular





concentrated under vacuum to about 20–30 mL, cooled in an ice-water bath, covered with a layer of ethyl acetate (about 50 mL) and acidified with a dilute solution of  $\text{KHSO}_4$  to pH 2–3 (Congo red). The aqueous phase was extracted with ethyl acetate and this operation was done repeatedly. The ethyl acetate extracts were pooled, washed with water and dried over anhydrous  $\text{Na}_2\text{SO}_4$  and evaporated under vacuum. The pure material was obtained as a waxy solid. Yield: 2.98 g (8.64 mmol, 86.4%).  $^1\text{H}$  NMR ( $\text{DMSO}-d_6$ , 400 MHz,  $\delta$  in ppm): 12.32 (b, 1H, COOH), 7.0172–6.9981 (d, 1H, NH,  $J = 3.82$ ), 6.7824–6.7576 (t, 1H, NH), 4.0496–3.9962 (m, 1H, Lys  $\text{C}^\alpha\text{H}$ ), 2.9179–2.8683 (m, 2H, Lys  $\text{C}^\omega\text{H}$ ), 1.6202–1.5076 (m, 2H, Lys  $\text{C}^\beta\text{H}$ ), 1.3702 (s, 18H, BOC Hs), 1.3397–1.2309 (m, 4H, Lys  $\text{C}^\gamma\text{H}$ ).  $^{13}\text{C}$  NMR ( $\text{DMSO}-d_6$ , 125 MHz,  $\delta$  in ppm): 173.2634, 155.6395, 78.9982, 66.3907, 53.4861, 30.4427, 29.0924, 28.2330, 22.9325.

**(b) Boc-Trp-OH.** A solution of L-tryptophan (2.04 g, 10 mmol) in a mixture of dioxane (20 mL), water (10 mL), and 1 N NaOH (10 mL) was stirred and cooled in an ice-water bath. Di-*tert*-butylpyrocarbonate (2.6 g, 12 mmol) was added and stirring was continued at room temperature for 6 h. Then, the solution was concentrated in vacuum to about 10–15 mL, cooled in an ice-water bath, covered with a layer of ethyl acetate (about 50 mL), and acidified with a dilute solution of  $\text{KHSO}_4$  to pH 2–3. The aqueous phase was extracted with ethyl acetate and this operation was done repeatedly. The ethyl acetate extracts were pooled, washed with water, and dried over anhydrous  $\text{Na}_2\text{SO}_4$  and evaporated in a vacuum. The pure material was obtained as a solid. Yield: 2.31 g (7.56 mmol, 75.6%).  $^1\text{H}$  NMR (500 MHz,  $\text{DMSO}-d_6$ ,  $\delta$  in ppm): 12.53 [b, 1H, COOH], 10.89 [b, 1H, NH, indole], 7.5 [s, 1H, amide NH], 7.33–7.32 [d, 1H, CH,  $J = 5$ ], 7.15–7.14 [d, 1H, CH,  $J = 5$ ], 7.06–7.0 [t, 2H, CH], 4.72 [m, 1H,  $\text{C}^\alpha\text{H}$ ], 3.12 [m, 1H,  $\text{C}^\beta\text{H}$ ], 2.99 [m, 1H,  $\text{C}^\beta\text{H}$ ], 1.3 [s, 9H, BOC]  $^{13}\text{C}$  NMR (125 MHz,  $\text{DMSO}-d_6$ ,  $\delta$  in ppm): 174.03, 155.45, 136.13, 127.22, 123.69, 120.94, 118.39, 118.19, 111.43, 110.2, 78.05, 54.56, 28.21, 28.1, 27.81, 26.85.

**(c) 1,3,5-Triaminobenzene.** 1.145 g (5 mmol) of picric acid was dissolved in 50 mL MeOH taken in a 100 mL round bottom flask. To this solution 3.78 g (60 mmol) ammonium formate and 1.96 g (30 mmol) Zn dust were added and the mixture was refluxed at 60 °C for 1 h. The reaction mixture was cooled to room temperature and filtered. The filtrate was evaporated under vacuum to provide tri-aminobenzene as black solid. Yield: 528.8 mg (4.29 mmol, 85.8%).  $^1\text{H}$  NMR ( $\text{DMSO}-d_6$ , 400 MHz,  $\delta$  in ppm): 8.300 (s, 3H, aromatic protons), 5.418 (b, 6H,  $\text{NH}_2$ ).  $^{13}\text{C}$  NMR ( $\text{DMSO}-d_6$ , 100 MHz,  $\delta$  in ppm): 166.35, 125.33.

**(d) Trisamide 1.** 325 mg (2.64 mmol) triaminobenzene was dissolved in 25 mL dry DMF. 2.73 g and (7.91 mmol)  $\text{Boc}_2\text{-Lys-OH}$ , 1.65 g (8 mmol) DCC and 1.08 g (8 mmol) HOBt were added and stirred for 48 h at room temperature. Then the solution was added to 200 mL water, taken in a separating funnel and shaken for 15 min and 150 mL ethyl acetate was added and shaken for another 10 min. The organic layer was collected and DCU was filtered off. The organic layer was washed with 2 M HCl (3 × 50 mL), brine (2 × 50 mL), then 1 M sodium carbonate (3 × 50 mL) and brine (2 × 50 mL) and dried over anhydrous sodium sulfate and evaporated under vacuum to yield trisamide 1 as a yellow solid. Purification was performed

by silica gel column (100–200 mesh size) using ethyl acetate and hexane (3 : 1) as eluent. Yield: 1.562 g (1.41 mmol, 53.4%).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz,  $\delta$  in ppm): 6.568 (s, 3H, triamine NH protons), 6.0317 (s, 3H, TAB), 5.4141 (s, 3H, NH), 4.1120 (s, 3H, NH Boc), 3.0823–3.0747 (d, 3H,  $J = 1.92$   $\text{C}^\alpha\text{H}$  Lys), 1.8325–1.7632 and 1.6800–1.6012 (2m, 18H,  $\text{C}^{\beta+\gamma+\omega}\text{H}$  of Lys), 1.3781 (s, 54H, Boc).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz,  $\delta$  in ppm): 175.5228, 156.6967, 156.1335, 80.3308, 79.6014, 54.4205, 40.3461, 40.3461, 34.0851, 32.3809, 30.0484, 22.9715.

**(e) Trisamide 2.** 325 mg (2.64 mmol) triaminobenzene was dissolved in 25 mL dry DMF. 2.41 g and (7.91 mmol) Boc-Trp-OH, 1.65 g (8 mmol) DCC and 1.08 g (8 mmol) HOBt were added and stirred for 48 h at room temperature. Then the solution was added to 200 mL water, taken in a separating funnel and shaken for 15 min and 150 mL ethyl acetate was added and shaken for another 10 min. The organic layer was collected and DCU was filtered off. The organic layer was washed with 2 M HCl (3 × 50 mL), brine (2 × 50 mL), then 1 M sodium carbonate (3 × 50 mL) and brine (2 × 50 mL) and dried over anhydrous sodium sulfate and evaporated under vacuum to yield trisamide 1 as a yellow solid. Purification was performed by silica gel column (100–200 mesh size) using ethyl acetate and hexane (4 : 1) as eluent. Yield: 1.936 g (1.97 mmol, 74.6%).  $^1\text{H}$  NMR ( $\text{DMSO}-d_6$ , 400 MHz,  $\delta$  in ppm): 10.79 (s, 3H, indole NH), 7.6088–7.5897 (d, 3H, triamine NH protons,  $J = 3.82$ ), 7.3740 (s, 3H, TAB), 7.3244–7.3053 (d, 3H, Trp aro protons,  $J = 3.82$ ), 7.1183 (s, 3H, Boc NH), 7.0687–6.9465 (m, 9H, Trp aro protons), 4.1565–4.1011 (m, 3H,  $\text{C}^\alpha\text{H}$  Trp), 3.0859–3.0382 and 2.9122–2.8531 (2m, 6H,  $\text{C}^\beta\text{H}$  of Trp), 1.3092 (s, 27H, Boc).  $^{13}\text{C}$  NMR ( $\text{DMSO}-d_6$ , 100 MHz,  $\delta$  in ppm): 174.8672, 156.2366, 136.7765, 127.5184, 123.7809, 122.7225, 120.2054, 119.3092, 111.7387, 80.5606, 55.1986, 28.7687.

### NMR experiments

All NMR studies were carried out on a Bruker AVANCE 500 MHz and Jeol 400 MHz spectrometer at 278 K. Compound concentrations were in the range 1–10 mM in  $\text{CDCl}_3$  and  $\text{DMSO}-d_6$ .

### FT-IR spectroscopy

All reported solution FT-IR spectra were obtained with a Perkin Elmer Spectrum RX1 spectrophotometer.

### UV-Vis spectroscopy

UV-Vis absorption spectra were recorded on a UV-Vis spectrophotometer (Hitachi).

### Mass spectra

Mass spectra were recorded on a Q-ToF Micro YA263 high resolution (Waters Corporation) mass spectrometer by positive-mode electrospray ionization.

### CD spectroscopy

Circular dichroism absorption spectra were recorded Jasco J-815 CD spectrophotometer.



## Field emission scanning electron microscopy

Morphologies of all reported compounds were investigated using field emission-scanning electron microscopy (FE-SEM). A small amount of solution of the corresponding compound was placed on a clean silicon wafer and then dried by slow evaporation. The material was then allowed to dry under vacuum at 30 °C for two days. The materials were gold-coated, and the micrographs were taken in an FE-SEM apparatus (Jeol Scanning Microscope-JSM-6700F).

## Atomic force microscopy

The morphology of the reported compound was investigated by atomic force microscopy (AFM). A drop of the sample solution in methanol were placed on a clean microscope cover glass and then dried by slow evaporation. The material was then allowed to dry under vacuum at 30 °C for two days. Images were taken with an NTMDT instrument, model no. AP-0100 by semicontact-mode.

## Conclusions

In summary, we have shown the addition of HCl reverses the helical handedness of the supramolecular polymer obtained from the self-assembly of chiral amino acid modified C<sub>3</sub>-symmetric trisamide. The intermolecular three fold hydrogen bonds break and amide functional groups re-organized in the opposite side of central aromatic core by virtue of interaction with HCl. Irrespective of chiral centres configuration, supramolecular chiral bias is arising from molecular conformations. FE-SEM reveals the formation of right handed entangled polymeric fibers. However, left handed entangled fibers have appeared on addition of 0.12 M HCl. The trisamide 2 containing Boc protected L-Trp exhibits disk like morphology both in presence and absence of 0.12 M HCl and does not show change of supramolecular handedness. The acid responsive supramolecular chirality inversion strategy has potential for other building blocks that form multiple competing chiral supramolecular structures.

## Conflicts of interest

Authors do not have any conflict of interest.

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