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# SUPPORTING INFORMATION

## Molecular-level Insights into Self-Assembly Driven Enantioselective Recognition Process

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**Materials:** All chemicals were purchased from TCI, Himedia, Merck (Sigma-Aldrich) and Alfa Aesar and were used without further purification. Double-distilled (18.3 m $\Omega$ ) deionized water (ELGA PURELAB Ultra) was used for hydrogel formation. HCl was diluted to a concentration of 1N and then used.

Instruments: FT-IR spectra were recorded on a Carry-660 FT-IR spectrometer. <sup>1</sup>H and <sup>13</sup>C NMR spectra in methanol-d4 and D2O were recorded on a Jeol-ECX-500 MHz spectrometer using tetra methyl silane as an internal standard. HRMS spectra were recorded on a Bruker impact-HD spectrometer. The morphology of the gel was characterized by using a field emission scanning electron microscope (FESEM) FEI Nova Nano SEM-450. Rheological measurements were performed using a stress-controlled rheometer (Anton Paar Quality Control Rheometer MCR 302 instrument) equipped with stainless steel parallel plates (25 mm diameter, 2.5 mm gap). The gels were prepared at different concentrations (1-3 equiv.) of Rmandelic acid w.r.t FA and left undisturbed for ~24h before performing the measurements. To perform the rheological measurements the gels were scooped out and placed on the plate and were rested for 1 min before starting the measurements. The amplitude sweep measurements were performed at a shear strain % of 1 to 100%. X-ray photoelectron spectroscopy (XPS) measurements were carried out on a NEXSA surface analysis model by Thermo Fisher Scientific using Al-K<sub> $\alpha$ </sub> (1486.6 eV) X-ray radiation. The XPS data were acquired with a spot size 400 µm having a standard lens mode. The obtained data from the instrument were plotted and deconvoluted using Avantage software. X-ray diffraction data was measured on Agilent Technologies X-ray (Cu Kα, l= 1.5406 Å at 298(2) K) diffractometer system. Data were collected by standard 'CrysalisPro' Software (online version) and the reduction was under taken with CrysalisPro Software (offline version).<sup>1</sup> All calculations and molecular structure were solved by direct methods using OLEX<sup>2</sup> and full-matrix least-squares (F<sup>2</sup>) on SHELXL-97.<sup>2, 3</sup> The positions of all non-hydrogen atoms were located and were refined anisotropically.

After that, hydrogen atoms were obtained from the residual density map and refined with isotropic thermal parameters. CCDC reference number of FA and FA/(S)-MA 2031638 and 2031639 respectively.

Synthetic Procedure of FA: To a methanolic solution of L-Arginine (491mg, 1.1 equiv., 80 mL), fluorene-2-carboxalehyde (500mg, 1 equiv.) was slowly added. The reaction mixture was refluxed for 6 hrs leading to formation of precipitates within the reaction solution. Further, the reaction mixture was treated with sodium borohydride (4 equiv.) with constant stirring at room temperature for 1 h. The solvent was evaporated after completion of the reaction using a rotary evaporator. The resulting residue was then dissolved in water and acidified with dil. HCl to pH 7-8 under stirring. The white precipitate thus formed was filtered through a sintered funnel and washed with water (20 mL). Finally, the product was air-dried for 1 h to afford white powder as the ligand (FA). Yield = 72%. Expected mass  $[M + H^+] = 352.1972$ , recorded mass  $[M + H^+] = 352.1972$ . Specific rotation at 1mg/mL, 28 °C= 7.903. <sup>1</sup>H-NMR (500 MHz, Methanol-*d4*):  $\delta$  7.80 (t, J= 7.6 Hz, 2H), 7.63 (s, 1H), 7.54 (d, J= 6.85 Hz, 1H), 7.45 (d, J= 7.7 Hz, 1H), 7.35 (t, J= 6.9 Hz, 1H), 7.29 (t, J= 7.7 Hz, 1H), 4.14 (d, J= 12.7 Hz, 1H), 4.0 (d, J= 13.1 Hz, 1H), 3.43- 3.40 (m, 1H), 3.21- 3.12(m, 2H), 1.87- 1.68 (m, 4H). <sup>13</sup>C-NMR (125 MHz, methanol-*d4*):  $\delta$  176.1, 158.7, 145.3, 144.9, 143.6, 142.3, 133.9, 129.6, 128.3, 128, 127.6, 126.2, 121.2, 121.1, 63.1, 52.5, 42.1, 37.6, 29.5, 26.2.

**Enantioselective Recognition Experiment:** To perform the enantioselective recognition of mandelic acid, FA (5mg, 0.014 mmol) was added in 200µL water wherein 20µL of 1N HCl was added to dissolve FA completely. Finally, 1 equiv. (R)-MA/(S)-MA was added to the above solution which immediately resulted in gel/precipitate formation respectively.

**Thermo-reversibility and Self-Healing:** The FA/(R)-MA gel formed at 1:1 ratio was heated slowly and the transition to solution state was monitored. The temperature at which gel to sol

transition occurred was noted. Thereafter this solution was cooled to room temperature leading to regaining the gel state.

The self-healing behaviour of FA/(R)-MA was confirmed by cutting the gel into two halves. Methyl orange was added to one of the halves to distinguish them. Finally, the two gels were placed together to note their behaviour.

Synthesis Procedure for FDA: FDA was synthesized following the same procedure as reported above using D-Arginine. Yield = 68% Expected mass  $[M + H^+] = 352.1972$ , recorded mass  $[M + H^+] = 352.1972$ . Specific rotation at 1mg/mL, 28 °C= -7.125. <sup>1</sup>H-NMR (500 MHz, Methanol-*d4*):  $\delta$  7.75 (dd, J= 7.55 and 3.4 Hz, 2H), 7.54- 7.51 (m, 2H), 7.36- 7.31 (m, 2H), 7.27- 7.24 (m, 1H), 3.87- 3.85 (m, 3H), 3.65 (d, J= 12.35 Hz, 1H), 3.15- 3.09 (m, 3H), 1.66- 1.60 (m, 3H). <sup>13</sup>C-NMR (125 MHz, methanol-*d4*):  $\delta$  181.7, 158.6, 144.9, 144.7, 142.8, 142.2, 139.4, 128.6, 127.9, 127.8, 126.6, 126.1, 120.8, 120.7, 64.3, 53.6, 42.3, 37.6, 31.6, 26.7.

**Computational Details:** We performed DFT calculations employing Becke's three-parameter hybrid with Lee-Yang-Parr correlation functional  $(B3LYP)^{4, 5}$  in conjunction with 6-31G(d,p) basis set. Grimme's D3 dispersion with Becke-Johnson damping  $(D3BJ)^6$  was used during all calculations to account for the electron correlation effects due to dispersion interactions. The experimental solvent effect was mimicked using solvation effect of water ( $\varepsilon = 78.35$ ) through polarizable continuum (CPCM) solvation model. The calculations were performed on a 4:1 model of the gelator FA and mandelic acid (MA), consisting of four units of FA and one unit of MA and five explicit water molecules. The models were constructed using the crystal structure of FA/(S)-MA as a guide. Using a similar 4:1 model both for FA/(S)-MA and FA/(R)-MA, we were able to make a direct comparison between them in terms of the molecular-level interactions of the mandelic acid enantiomers, (R)-/(S)-MA with the gelator. As described in the crystal structure, the guanidine nitrogen and amine nitrogen appear protonated. However, it is unlikely that such protonation disrupts the hydrogen bonding network between FA and

(R)-/(S)-MA –OH, as –OH is far away from the protonated sites. Furthermore, using a protonated FA unit in our 4:1 theoretical model would disrupt the overall charge balance. Therefore, we constructed our 4:1 model using the non-protonated structures, i.e. carboxylic groups were used instead of carboxylate–iminium zwitterionic forms. Five explicit water molecules, four hydrogen-bonded to the –NH group of four gelator units and one hydrogen-bonded to the –COOH group of the MA were used in our 4:1 model to mimic the effects of water on hydrogen bonds. The models were constructed from the crystal structure of FA/(*S*)-MA and the position of the FA units were kept frozen during the optimisation to see the difference in H-bonding interactions between MA and gelator FA for FA/(*S*)-MA and FA/(*R*)-MA structures. Although a full simulation of the morphology was not feasible at the DFT level, we tried to draw a reasonable conclusion on the morphology difference between FA/(*R*)-MA and FA/(*S*)-MA based on our DFT results. In this regard, we performed a DFT-level geometry optimisation of our 4:1 FA:MA models by fixing all the N-atoms at their crystal position of FA/(*S*)-MA structure. All calculations were performed using Gaussian 09 (Revision D.01) suite of quantum chemistry program.<sup>7</sup>



**Fig. S1** Molecular structure of **FA**, **FDA**, (*R*)- mandelic acid ((*R*)-MA), (*S*)mandelic acid ((*S*)-MA), (*R*)- $\alpha$ -methoxyphenyl acetic acid ((*R*)-OMe-MA), (*S*)- $\alpha$ methoxyphenyl acetic acid ((*S*)-OMe-MA), (*R*)-2-chloromandelic acid ((*R*)-Cl-MA) and (*S*)-2-chloromandelic acid ((*S*)-Cl-MA).



Scheme S1. Synthesis scheme of chiral gelator FA.



Fig. S2 <sup>1</sup>H NMR spectra of FA in methanol-d4



Fig. S3 <sup>13</sup>C NMR spectra of FA in methanol-d4



Fig. S4 HRMS spectra of FA

![](_page_7_Figure_2.jpeg)

**Fig. S5** Crystal structure of FA asymmetric unit and showing intermolecular H-bonding and interactions with water.

Identification code	FA_A_Lt_Cu
Empirical formula	$C_{20}H_{26}N_4O_3$
Formula weight	370.45
Temperature/K	150.00(10)
Crystal system	orthorhombic
Space group	P21212
a/Å	8.3715(6)
b/Å	33.178(3)
c/Å	7.1858(4)
$\alpha/^{\circ}$	90.00
в/°	90.00
γ/°	90.00
Volume∕ų	1995.9(3)
Ζ	4
$\rho_{calc}g/cm^3$	1.233
$\mu/mm^{-1}$	0.685
F(000)	792.0
Crystal size/mm <sup>3</sup>	$0.320 \times 0.210 \times 0.120$
Radiation	CuKα (λ = 1.54184)
20 range for data collection/°	12.32 to 133.8
Index ranges	-9 ≤ h ≤ 8, -30 ≤ k ≤ 39, -5 ≤ l ≤ 8
Reflections collected	2470
Independent reflections	1811 [R <sub>int</sub> = 0.0443, R <sub>sigma</sub> = 0.0543]
Data/restraints/parameters	1811/42/223
Goodness-of-fit on F <sup>2</sup>	1.520
Final R indexes [I>=2σ (I)]	R <sub>1</sub> = 0.1243, wR <sub>2</sub> = 0.3434
Final R indexes [all data]	R <sub>1</sub> = 0.1351, wR <sub>2</sub> = 0.3646
Largest diff. peak/hole / e Å <sup>-3</sup>	0.60/-0.34
Flack parameter	0.3(4)

Table S1 Crystallographic data for FA

![](_page_8_Picture_2.jpeg)

Fig. S6 Photograph of (a) gel formed by FA and (R)-MA and (b) precipitates formed by FA and (S)-MA

![](_page_9_Figure_0.jpeg)

Fig. S7 Rheological (Amplitude sweep) measurement of hydrogel formed by FA: (R)-MA (1:1)

**Rheological measurements:** To confirm the gel nature, the storage modulus (G') dominates over the loss modulus (G") at lower strain, while G" crosses over G' and dominates at a higher strain indicative of the gel to sol transition. The point of intersection is termed as yield stress ( $\gamma$ %), and higher  $\gamma$ % signifies higher strain bearing capability and therefore a more mechanically stable gel.

![](_page_9_Figure_3.jpeg)

**Fig. S8** Rheological (Amplitude sweep) measurement of hydrogel formed by FA: (R)-MA (a) 1:1.7, (b) 1:2.5, (c) 1:2.7, (d) 1:3

Ratio (FA:(R)-MA)	Yield Strength γ(%)
1:1	35.947
1:1.7	15.711
1:2.5	14.801
1:2.7	13.802
1:3	11.806

Table S2 Yield strength values of gel formed with various ratios of FA:(R)-MA

#### Unknown concentration calculation

Three batches of different concentrations ranging from 1 equiv. to 3 equiv. with respect to FA were prepared and the amplitude sweep measurements were performed. The unknown concentration was calculated based on the yield strength values  $\gamma(\%)$  in all three batches individually. The unknown concentration was found to be 2.06 equiv., 1.92 equiv. and 1.83 equiv. wih respect to FA. The error bar was calculated from the obtained values of the unknown concentration.

![](_page_10_Figure_4.jpeg)

Fig. S9 Graph between different ratios of FA: (R)- MA with respect to the yield strength

![](_page_11_Figure_0.jpeg)

Fig. S10 Gel to sol transition of 1:1 FA: (R)-MA gel in response to temperature

#### Thermo-reversibility:

The gel (1:1) (FA:(R) -MA) was found to exhibit transition to a sol in response to heat at 50 °C and again to a gel on cooling.

#### Self-assembly behaviour:

Self-assembly behaviour was established by placing two gels in contact to each other, which eventually fused with each other within 30 minutes without needing any external heat

![](_page_11_Picture_6.jpeg)

Fig. S11 Two FA: (R)-MA gel that heal when placed together. (methyl orange is added to color one of the gels)

![](_page_12_Figure_0.jpeg)

Fig. S12 <sup>1</sup>H NMR spectra of FA on addition of (R)-MA displaying broadening of peaks

![](_page_12_Figure_2.jpeg)

Fig. S13 XPS survey spectrum of FA

![](_page_13_Figure_0.jpeg)

Fig. S14 XPS survey spectrum of FA/(R)-MA xerogel

![](_page_13_Figure_2.jpeg)

Fig. S15 XPS survey spectrum of FA/(S)-MA precipitates

![](_page_14_Figure_0.jpeg)

Fig. S16 FTIR spectra of FA, FA/(R)-MA and FA/(S)-MA

#### **FTIR measurements:**

FTIR measurements were performed for FA, MA, xerogel (FA/(R)-MA) and precipitates (FA/(S)-MA) (Fig. S16). The stretching frequencies of carbonyl FA was observed at 1690 and 1643 cm<sup>-1</sup> which shifted to 1650 cm<sup>-1</sup> for xerogel while the precipitates displayed a peak at 1645 cm<sup>-1</sup> thereby confirming the role of carboxylic acid group in hydrogen bonding.<sup>8</sup>

![](_page_14_Figure_4.jpeg)

**Fig. S17** Crystal Structure of (a) FA/(S)-MA showing H-bonding interactions between FA, (S)-MA and water units

#### Single crystal structure analysis of FA/(S)-MA

The single crystal diffraction analysis revealed the monoclinic crystallization of integrated system (FA/(S)MA) with P2<sub>1</sub> space group. The asymmetric unit consists of mandelate ion and one FA unit with (S) configuration as shown in Figure 3a. It was found that both the guanidine moiety and amine group attached with chiral carbon of FA unit appeared as protonated form whereas the carboxylic acid remained as carboxylate anion. Similarly, the mandelic acid was found to be participate as mandelate in the crystal system. Similar type of structure has been reported by Yoichi litaka et al. and D. Xu et al. in L-Arginine Phosphate Monohydrate and L-arginine 4-nitrophenolate 4-nitrophenol dehydrate systems respectively.<sup>9-10</sup> As shown in the Fig. S17, the carboxylate moiety of mandelic acid was directly involved in intermolecular H-bonding with the guanidium moiety. In addition, carboxylate group also formed the hydrogen bonds with the protonated amine group of adjacent FA units. On the other hand -OH group of mandelic acid was H-bonded with carboxylate group of FA unit.

Identification code	FA_SMA_Rt_Cu
Empirical formula	$C_{28}H_{32}N_4O_5$
Formula weight	504.58
Temperature/K	293(2)
Crystal system	monoclinic
Space group	P21
a/Å	10.8104(5)
b/Å	5.7783(3)
c/Å	21.3807(10)
α/°	90.00
в/°	91.903(5)
γ/°	90.00
Volume/ų	1334.82(11)
Ζ	2
$ ho_{calc}g/cm^3$	1.255
µ/mm⁻¹	0.712
F(000)	536.0
Crystal size/mm <sup>3</sup>	$0.393 \times 0.098 \times 0.079$
Radiation	CuKα (λ = 1.54184)
20 range for data collection/°	15.88 to 133.72
Index ranges	$-12 \le h \le 10$ , $-6 \le k \le 6$ , $-25 \le l \le 25$
Reflections collected	4483
Independent reflections	2860 [R <sub>int</sub> = 0.0263, R <sub>sigma</sub> = 0.0416]
Data/restraints/parameters	2860/1/335
Goodness-of-fit on F <sup>2</sup>	0.988
Final R indexes [I>=2σ (I)]	$R_1 = 0.0343$ , $wR_2 = 0.0796$
Final R indexes [all data]	$R_1 = 0.0384$ , $wR_2 = 0.0815$
Largest diff. peak/hole / e Å <sup>-3</sup>	0.12/-0.13
Flack parameter	-0.2(2)

Table S3 Crystallographic data for FA/(S)-MA

![](_page_16_Picture_0.jpeg)

Fig. S18 Photograph of gel formed by (a) FA/(S)-OMe-MA and (b) FA/(R)-OMe-MA

![](_page_16_Picture_2.jpeg)

Fig. S19 Photograph of gel formed by (a) FA/(S)-Cl-MA and (b) FA/(R)-Cl-MA

![](_page_16_Picture_4.jpeg)

Fig. S20 SEM images of gel formed by (a) FA/(R)-OMe-MA and (b) FA/(S)-OMe-MA

![](_page_17_Figure_0.jpeg)

Fig. S21  $^{1}$ H NMR spectra of OMe-MA in D<sub>2</sub>O

![](_page_17_Figure_2.jpeg)

Fig. S22  $^{1}$ H NMR spectra of FA/(R)-OMe-MA and (c) FA/(S)-OMe-MA in D<sub>2</sub>O

![](_page_18_Figure_0.jpeg)

Fig. S23 XPS profile of (a) N1s and (b) O1s of FA/(R)-OMe-MA and (c) FA/(S)-OMe-MA

![](_page_18_Figure_2.jpeg)

Fig. S24 XPS survey spectrum of FA/(R)-OMe-MA xerogel

![](_page_19_Figure_0.jpeg)

Fig. S25 XPS survey spectrum of FA/(S)-OMe-MA xerogel

![](_page_19_Figure_2.jpeg)

![](_page_19_Figure_3.jpeg)

**Fig. S26** Hydrogen bonding interaction between (R)-/(S)-methoxyphenylacectic acid (OMe-MA) and the gelator FA.

Both (R)-and (S)-OMe-MA retain very similar interaction between the -OMe group and the gelator carboxylic moiety, which likely results in the formation of similar morphology at the microscopic level (Fig. S20).

![](_page_20_Figure_0.jpeg)

**Fig. S27** Interaction between the –Cl group of (R)- and (S)-chloro-mandelic acid (CI-MA) and the gelator unit.

The DFT-optimised geometry shows that the FA gelator unit interacts with the -Cl group of (R)- or (S)-Cl-MA through fluorene C–H bond at a very similar distance, while maintaining similar types of Hbonds between MA and FA. This scenario is different from what we observed in the case of the original (R)- or (S)-MA, where distinct C-H… $\pi$  interactions resulted in different morphology. Therefore, in case of (R)- or (S)-Cl-MA, due to the similar nature of interactions between the FA and MA units, we obtained a similar morphology in the formed gel (Fig. S28).

![](_page_20_Figure_3.jpeg)

Fig. S28 SEM images of gel formed by (a) FA/(R)-Cl-MA and (b) FA/(S)-Cl-MA

![](_page_21_Picture_0.jpeg)

Fig. S29 Photograph of gel/precipitates formed at different ratios of (R): (S)-MA

![](_page_21_Picture_2.jpeg)

Fig. S30 Photograph of precipitates embedded into the gel formed and precipitates formed at different ratios

**Effect of mixture of enantiomers:** We investigated the gelation phenomena in the presence of a mixture of enantiomers, wherein retention of the pure gel up to 85:15 mixture of (R)- and (S)-mandelic acid, respectively (Fig. S29) was found. Thereafter, self-assembly occurred through both gelation and precipitation. Ultimately, in a ratio of 70:30 the discrimination was spotted where the complete precipitation induced assembly process dominated (Fig. S30).

![](_page_22_Figure_0.jpeg)

**Fig. S31** <sup>1</sup>H NMR spectra of FDA in methanol-*d4* 

![](_page_22_Figure_2.jpeg)

Fig. S32 <sup>13</sup>C NMR spectra of FDA in methanol-d4

![](_page_23_Figure_0.jpeg)

Fig. S33 HRMS spectra of FDA

![](_page_23_Picture_2.jpeg)

Fig. S34 Photograph of (a) gel formed by FDA/(S)-MA and (b) precipitates formed by FDA/(R)-MA

![](_page_24_Figure_0.jpeg)

**Fig. S35** Crystal structure of FDA/(R)-MA showing H-bonding interactions between FDA, (R)-MA and water units.

#### Single crystal structure analysis of FDA/(R)-MA

The single crystal XRD showed that (FDA/(R)-MA) crystallized in a monoclinic crystal system with space group P2<sub>1</sub>. The asymmetric unit consists of one FDA unit with R configuration and mandelate ion (Fig. 3b). Like FA/(S)-MA system, both the guanidine moiety and the amine (-NH) attached with chiral carbon were found to be in protonated form whereas the carboxylic group of the FDA unit and mandelic acid remained in deprotonated form. As shown in the Fig. S35, the carboxylate moiety of mandelic acid and guanidinium moiety of FDA unit were found to be involved in strong intermolecular interactions as observed in FA/(S)-MA system.

Identification code
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FDA\_RMA\_RT\_Cu

Empirical formula	$C_{28}H_{32}N_4O_5$
Formula weight	504.58
Temperature/K	293(2)
Crystal system	monoclinic
Space group	P21
a/Å	10.8308(8)
b/Å	5.7790(4)
c/Å	21.3692(13)
$\alpha/^{\circ}$	90.00
<i>в/</i> °	91.933(7)
γ/°	90.00
Volume/ų	1336.77(16)
Ζ	2
$ ho_{calc}g/cm^3$	1.254
$\mu/mm^{-1}$	0.711
F(000)	536.0
Crystal size/mm <sup>3</sup>	0.223 × 0.095 × 0.068
Radiation	CuKα (λ = 1.54184)
20 range for data collection/°	12.44 to 133.66
Index ranges	-12 ≤ h ≤ 12, -6 ≤ k ≤ 4, -19 ≤ l ≤ 25
Reflections collected	4185
Independent reflections	2815 [R <sub>int</sub> = 0.0373, R <sub>sigma</sub> = 0.0559]
Data/restraints/parameters	2815/1/335
Goodness-of-fit on F <sup>2</sup>	0.990
Final R indexes [I>=2σ (I)]	R <sub>1</sub> = 0.0389, wR <sub>2</sub> = 0.0887
Final R indexes [all data]	$R_1 = 0.0462, wR_2 = 0.0921$
Largest diff. peak/hole / e Å-³	0.14/-0.14
Flack parameter	0.0(3)

### Table S4 Crystallographic data for FDA/(R)-MA

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