

# **Self-assembly of semiaromatic poly(amic acid) into flower-like microparticles via one-step precipitation polymerization**

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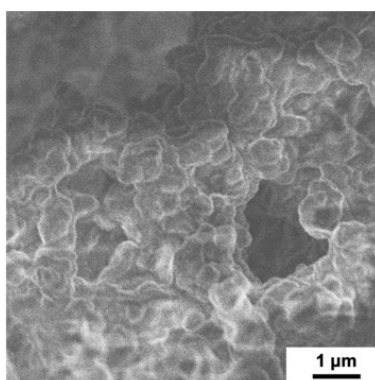
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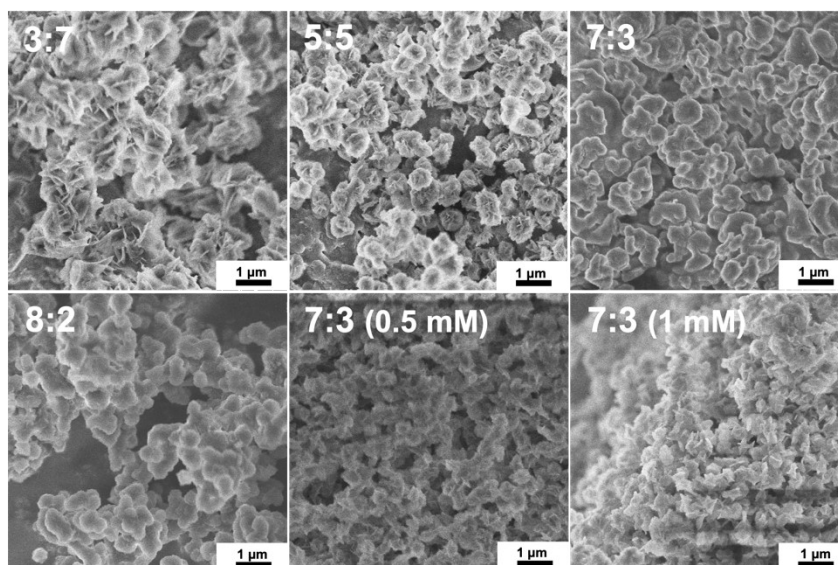
**Table S1.** Result of solubility test for DA-6 (5 mg/ mL).

Solvent	Solubility		
	r.t.	Upon heating	Recovered to r.t.
EtOH	-	-	
THF	-	+	+
Ether	-	-	
DMF	+		
DMSO	+		
DMAc	+		
Acetone	-	-	
Hexane	-	-	
Toluene	-	-	
Ethyl acetate	-	-	
Chloroform	-	-	
Dichloromethane	-	+	-
Chlorobenzene	-	-	
Pyridine	+		
NMP	+		
Acetonitrile	-	-	
Methyl ethyl ketone	-	-	
Acetophenone	-	-	
Cyclohexanone	-	+	+

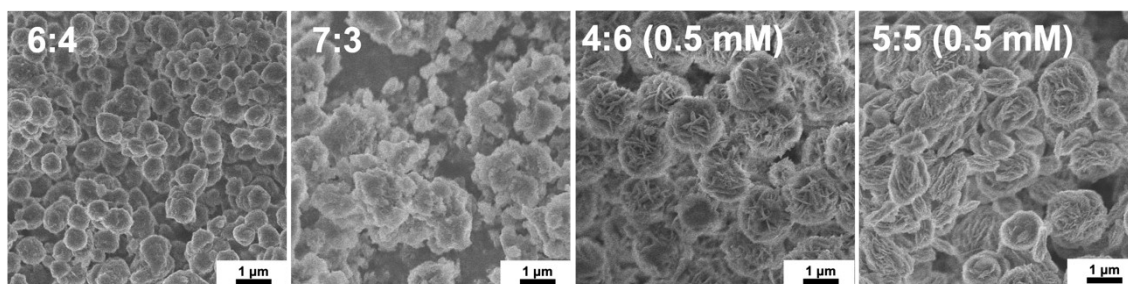
-:insoluble, +:soluble, -/recovered to r.t.: precipitate if recovered to r.t., +/-recovered to r.t.: do not precipitate if recovered to r.t.



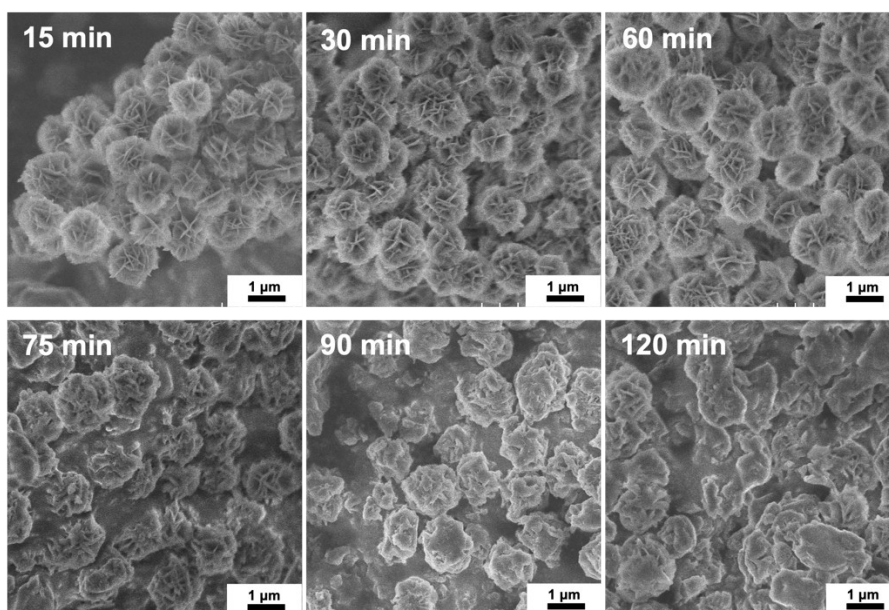
**Figure S1.** FE-SEM images of DA-6/PMDA prepared from THF.



**Figure S2.** FE-SEM images of DA-6/PMDA prepared in the mixed solvents of cyc/tol = 3:7, 5:5, 7:3, 8:2 and the influence of monomer concentration was studied in the condition of cyc/tol = 7:3.



**Figure S3.** FE-SEM images of DA-6/PMDA prepared in the mixed solvents of cyc/ace = 6:4, 7:3, and in an elevated concentration of 0.5 mM/ 100 mL in cyc/ace = 4:6 and 5:5.

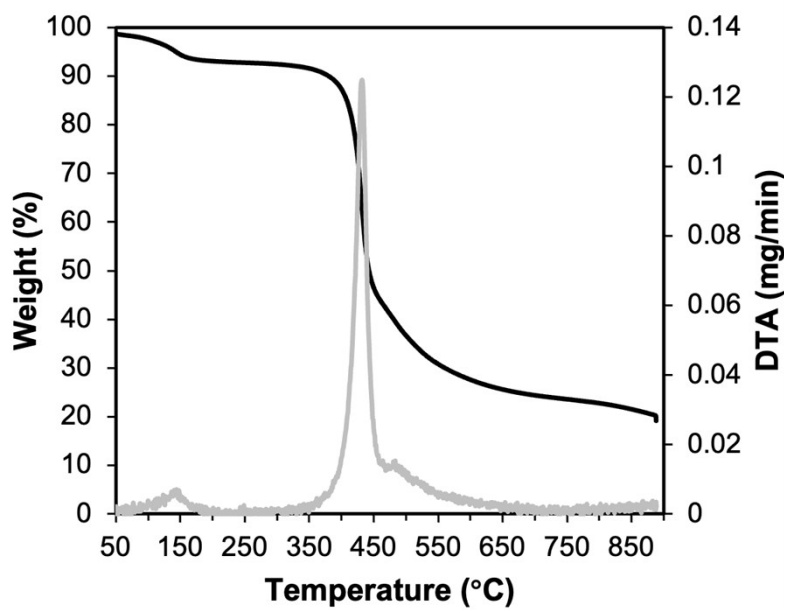


**Figure S4.** The morphology evolution of FLPs was studied by observing particle morphology at 5-minute intervals up to 120 min in the cyc/ace = 4:6. FLPs were visible from the beginning, but their morphology became increasingly blurred after 60 minutes of reaction time.

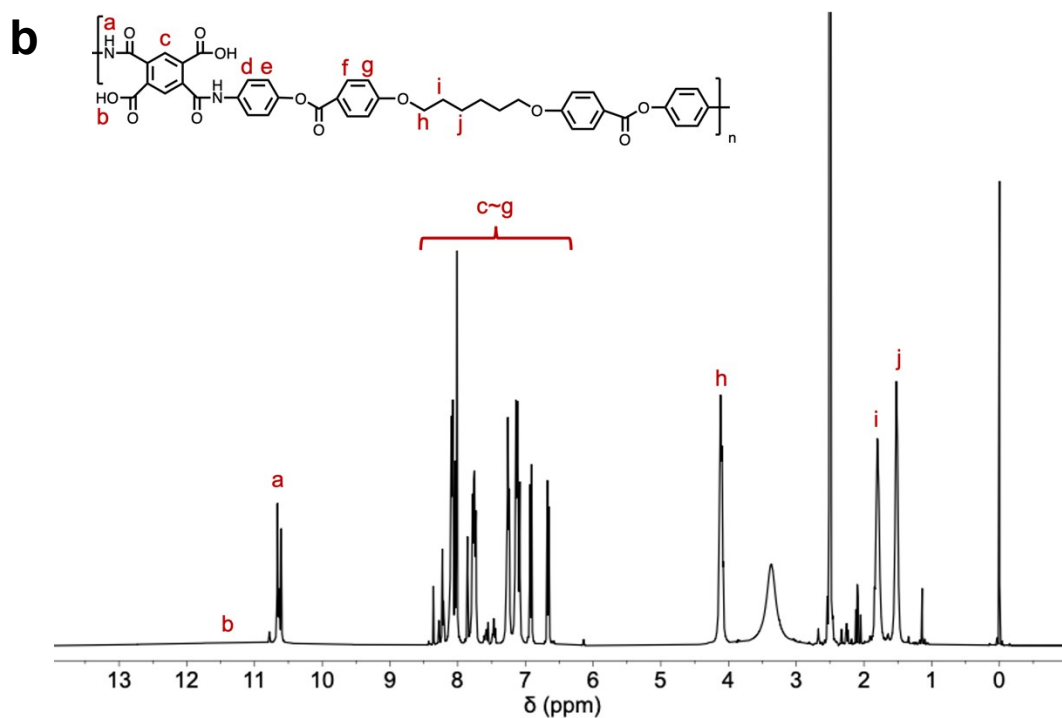
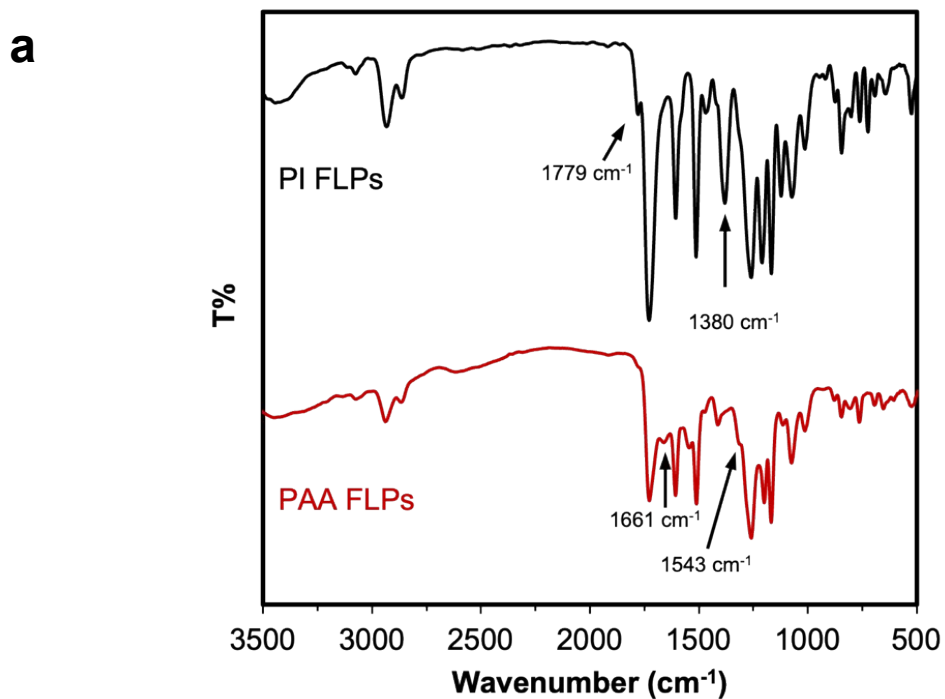
**Table S2.** Inherent viscosities of PAA particles prepared in the mixed solvent of cyc/tol = 7:3 and cyc/ace = 8:2, and PAA of the same chemical composition prepared from solution polymerization

	$\eta_{inh}^*$ [dL/g]
Cyc/tol = 7:3	0.14
Cyc/ace = 8:2	0.26
DA-6/PMDA PAA	1.11

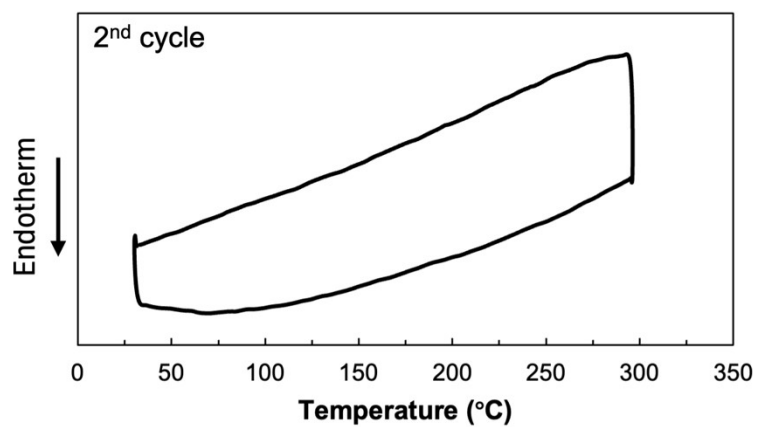
\*The inherent viscosities ( $\eta_{inh}$ ) of the PAAs were measured in NMP at a solid content of 0.5 dL/g at 30 °C using an Ostwald viscometer



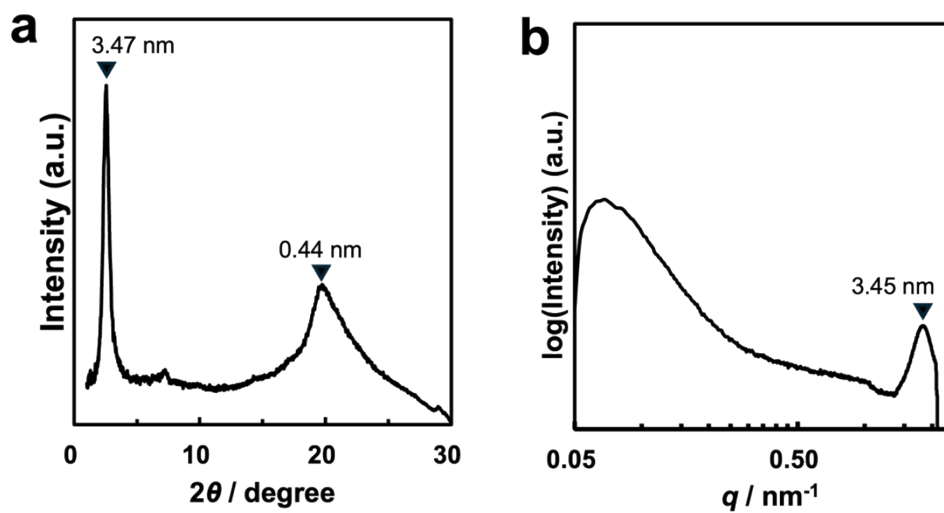
**Figure S5.** TGA/DTA of PAA FLPs.



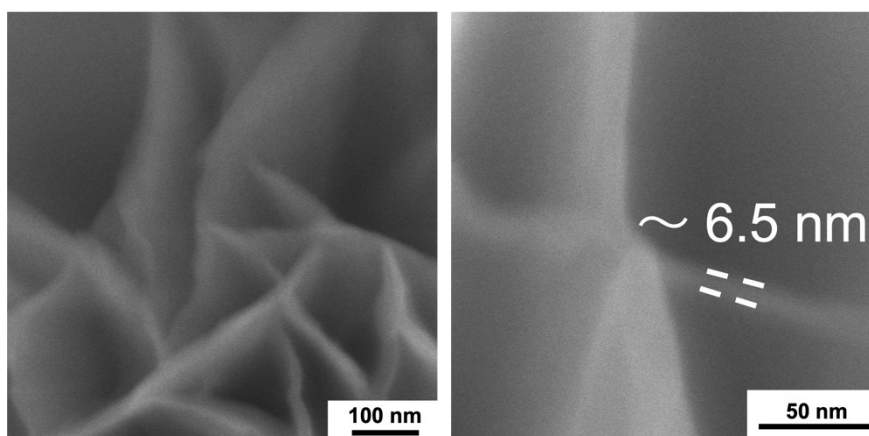
**Figure S6.** (a) FT-IR spectra of PAA FLPs prepared in cyc/ace = 5:5 as an example and PI. (b)  $^1\text{H}$  NMR (400 MHz) spectra of DA-6/PMDA PAA particles in  $\text{DMSO-}D_6$  prepared in cyc/ace = 2:8 as the representative. The carboxyl proton was not obvious, which could be attributed to COOH-NH- interactions and 3D conformation and concentration of PAA.<sup>1</sup>



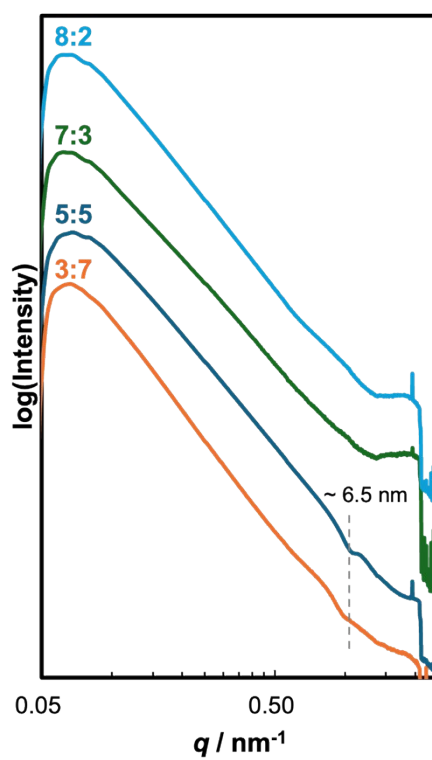
**Figure S7.** DSC thermogram of PI FLPs prepared in cyc/ace = 2:8 shows no phase transition behavior between 30~300 °C.



**Figure S8.** WAXD profile (a) and SAXS profile (b) of DA-6/PMDA bulk PI film.

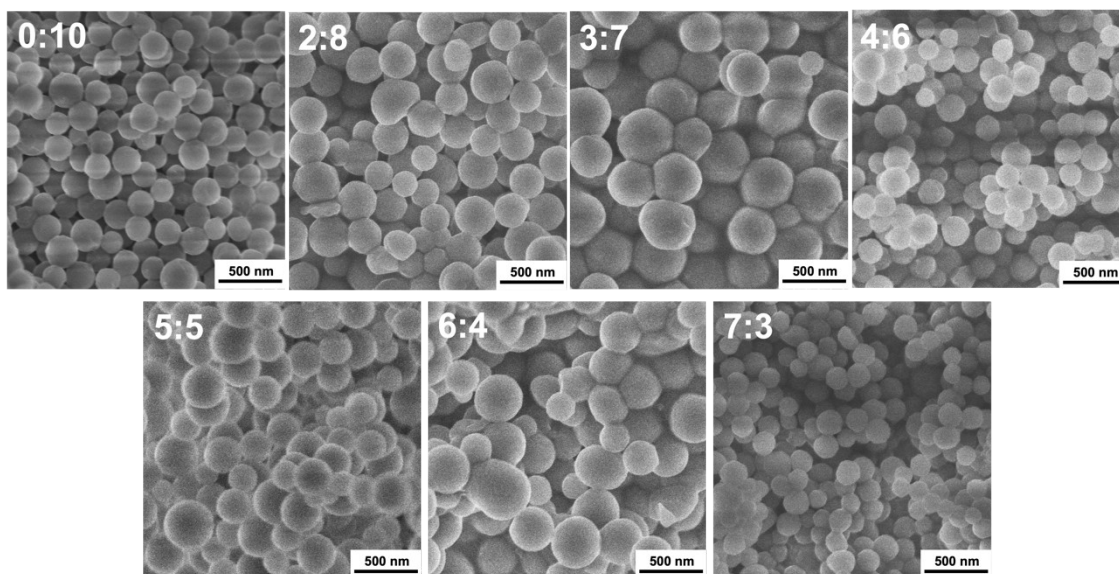


**Figure S9.** FE-SEM images of the layer thickness of the flower-like particles.



**Figure S10.** SAXS profile of DA-6/PMDA particles prepared in cyc/tol. Particles with fractal surface in cyc/tol = 3:7 and 5:5 exhibited oscillatory valley around 6.5 nm.

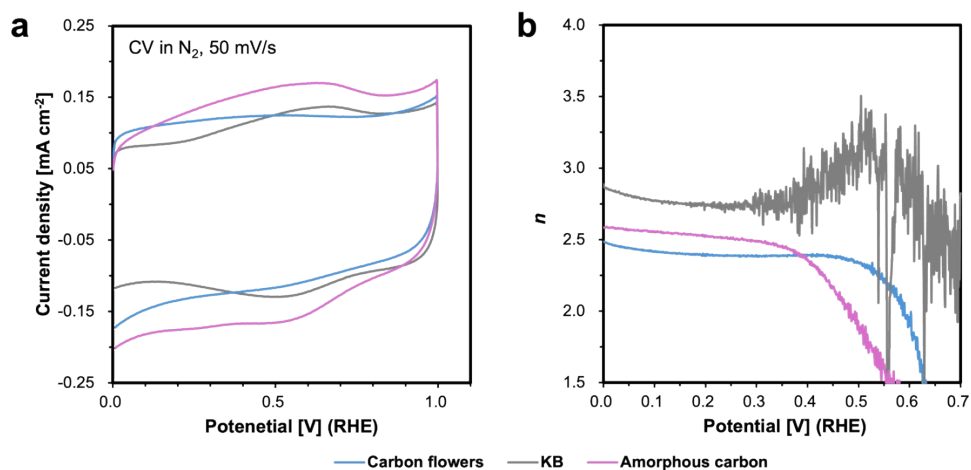




**Figure S11.** ODA/PMDA prepared in cyc/ace as the comparison.

**Table S3.** Half wave potential ( $E_{1/2}$ ) and the on-set potential ( $E_{0.1 \text{ mA}}$ ) defined as the potential at which a current density of 0.1 mA is achieved, are determined by the RRDE voltammogram of carbon flowers, amorphous carbon and KB.

	<b>Carbon flowers</b>	<b>Amorphous carbon</b>	<b>KB</b>
$E_{1/2}$ (V vs. RHE)	0.25	0.20	0.08
$E_{0.1 \text{ mA}}$ (V vs. RHE)	0.46	0.37	0.18



**Figure S12.** CV curves in  $N_2$  (a) and electron transfer number (b) of carbon flowers, amorphous carbon, and KB.

**Reference:**

1 İ. Yazgan, *Polym. Bull.*, 2020, **77**, 1191–1203.