

Supplementary Information

Harmonizing conjugated and non-conjugated emission for amorphous blue organic afterglow through copolymerization and aggregation engineering

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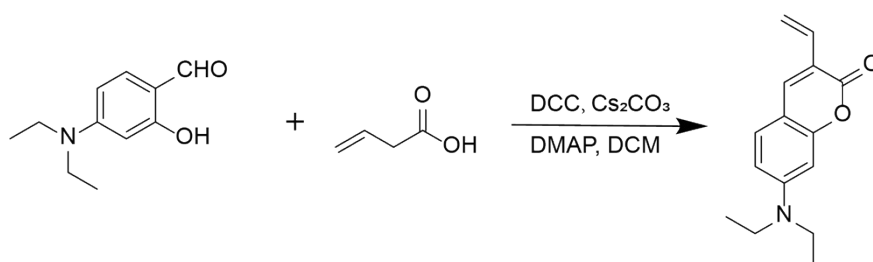
1 Materials and synthesis

Materials: The materials used in the experiments, including acrylamide, N-vinylcarbazole (**VCz**), 4-vinylphenylboronic acid (**VPhB**), 3-butenoic acid, dicyclohexylcarbodiimide (**DCC**), 4-diethylaminosalicylaldehyde, 3,6-dibromo-9-vinylcarbazole (**VCzBr₂**), 4-dimethylaminopyridine (**DMAP**), cesium carbonate, azobisisobutyronitrile (**AIBN**) and N,N-dimethylformamide (**DMF**) were purchased from Energy Chemical without further treatments. **AIBN** was recrystallized from ethanol then was used to initiate the free radical polymerization reaction. Manipulations involving air-sensitive reagents were performed in an atmosphere of dry argon.

Preparation of polyacrylamide (PAM): **PAM** was prepared by using the free radical polymerization of acrylamide initiated by **AIBN**. In a typical procedure, acrylamide (1.42 g, 20.0 mmol) and **AIBN** (33 mg, 0.2 mmol) were dissolved in super dry **DMF** (30 mL). The solution was heated at 65°C for 12 h under argon atmosphere. Afterward, the mixture was added into methanol (200 mL) to precipitate polymeric materials, and the crude product was collected and subjected to Soxhlet extraction with acetone, petroleum ether and dichloromethane for 12 hours, respectively. Finally, the purified white powder (yield:89%) was dried at 45°C in a vacuum oven to get **PAM**.

Preparation of poly(acrylamide-co-N-vinylcarbazole) (PAMCz): The copolymers were prepared in the same way of **PAM** using **VCz** and acrylamide in different feed ratios to afford **PAMCz0.3**, **PAMCz0.5**, **PAMCz0.7**, **PAMCz1.0**, **PAMCz1.3**, **PAMCz2.0** and **PAMCz4.0**, respectively. Taken **PAMCz** with 1 mol% **VCz** as an example, **VCz** (39 mg, 0.2 mmol), acrylamide (1.42 g, 20.0 mmol) and **AIBN** (33 mg, 0.2 mmol) were mixed, dissolved in anhydrous **DMF** (30 mL), heated, precipitated, collected and dried to obtain the copolymer in white powder in a yield of 90%.

Synthesis of 7-(diethylamino)-3-vinyl-2H-chromen-2-one (**VCM**)

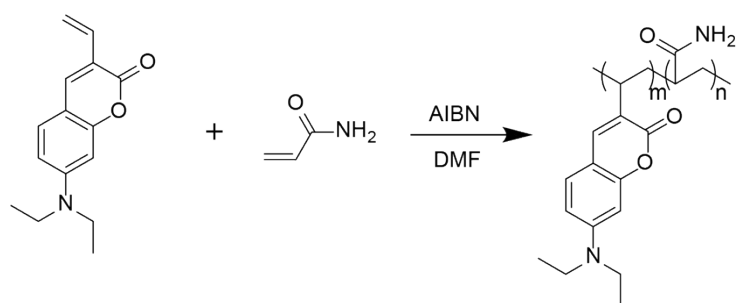


Scheme S1. Synthesis of **VCM**.

Under an atmosphere of argon, 3-butenoic acid (0.96 mL, 11.0 mmol) and **DCC** (2.27 g, 11.0 mmol) were dissolved in anhydrous dichloromethane and reacted at room temperature for 1 hour. Then, 4-diethylaminosalicylaldehyde (1.69 g, 8.7 mmol) and **DMAP** (1.22 g, 1.0 mmol) were added in sequence and reacted for 2 hours. The precipitate was removed by filtration and cesium carbonate (2.85 g, 8.7 mmol.) was added to the filtrate for the reaction at room temperature for 24 hours. Then, the reaction was quenched by water and the reaction mixture was washed by water and extracted by CH₂Cl₂ for three times. The organic phases were collected and dried with anhydrous sodium sulfate. The solvent was removed under reduced pressure and column chromatography (petroleum ether: ethyl acetate = 5:1) was used to obtain the purified product in orange-yellow solid with a yield of

53%. ^1H NMR (400 MHz, CDCl_3) δ 7.60 (s, 1H), 7.30-7.27 (m, 1H), 6.70 (dd, $J = 17.6, 11.3$ Hz, 1H), 6.60 (dd, $J = 8.8, 2.4$ Hz, 1H), 6.51 (d, $J = 2.5$ Hz, 1H), 6.04 (d, $J = 17.6$ Hz, 1H), 5.32 (d, $J = 11.3$ Hz, 1H), 3.44 (q, $J = 7.1$ Hz, 4H), 1.23 (t, $J = 7.1$ Hz, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 161.39 (s), 155.83 (s), 150.49 (s), 138.53 (s), 131.17 (s), 128.87 (s), 117.93 (s), 115.82 (s), 108.97 (s), 108.74 (s), 97.14 (s), 44.84 (s), 12.49 (s).

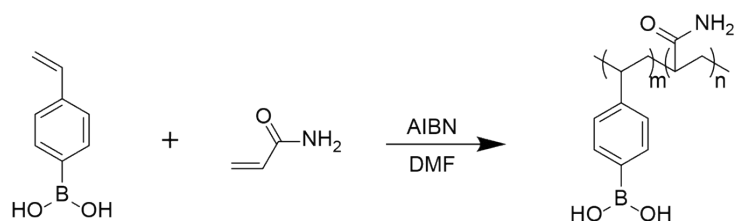
Synthesis of poly(acrylamide-co-7-(diethylamino)-3-vinyl-2H-chromen-2-one) (PAMCM)



Scheme S2. Synthesis of PAMCM.

PAMCM was obtained by free-radical copolymerization of VCM and acrylamide similar to PAMCz. Typically, VCM (49 mg, 0.2 mmol), acrylamide (1.78 g, 20.0 mmol) and AIBN (33 mg, 0.2 mmol) were used to afford the copolymer in powder with a yield of 94%.

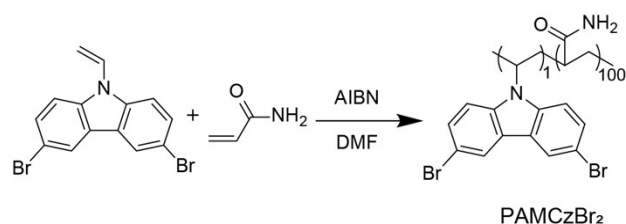
Synthesis of poly(acrylamide-co-4-Vinylphenylboronic acid) (PAMPhB)



Scheme S3. Synthesis of PAMPhB.

PAMPhB was similarly prepared using VPhB (37 mg, 0.25 mmol), acrylamide (1.78 g, 25 mmol) and AIBN (42 mg, 0.25 mmol) in a yield of 93%.

Synthesis of poly(acrylamide-co-3,6-dibromo-9-vinylcarbazole) (PAMCzBr₂)



Scheme S4. Synthesis of PAMCzBr₂.

PAMCzBr₂ (1 mol%) was similarly prepared using **VCzBr₂** (88 mg, 0.25 mmol), acrylamide (1.78 g, 25 mmol) and **AIBN** (42 mg, 0.25 mmol) in a yield of 86%.

Turn the powder form of the polymer to flake form

A facile water dissolution followed by slow evaporation process was developed to turn the powders of the polymers to flakes. As exemplified in the preparation of **PAMCz** in flake, 1.0 g **PAMCz** powder (**pPAMCz**) was dissolved in 1 mL deionized water; then, the aqueous solution was positioned in an oven at 70°C for 12 h. When the water was slowly evaporated, the transparent, smooth and tight flake of **PAMCz** (**wPAMCz**) was resulted conveniently.

The flakes of **PAM**, **9PrCz/PAM**, **PAMPhB**, and **PAMCM** were fabricated by the same method described above.

2 Measurements and methods

Nuclear magnetic resonance (NMR): NMR spectra were measured by a Bruker AV III HD 400 MHz spectrometer. D₂O and CDCl₃ were used as the solvents for the polymers and **VCM**, respectively. The actual contents of **VCz**, **VCM**, **VPhB** and **VCzBr₂** unit in the copolymers were figured out by the molar ratio of phenyl protons to methylene protons.

Scanning electron microscope (SEM): SEM images were collected after gold foil was sprayed on the polymers by a Hitachi scanning electron microscope (SU8010).

Optical microscope photography : Optical microscope images were taken under brightfield by U-LH100L-3 type upright fluorescence microscope from olympus corporation.

Photograph and video: The photographs were taken by Nikon D9. The videos were recorded by iphone 12 and were edited and annotated by the video software of “jianying”.

Gel Permeation Chromatography (GPC): The molecular weights of all polymers were measured by 341 Viscotek TDA305 max, employing 0.1 mol/L NaNO₃ solution as the mobile phase at a flow rate of 0.7 mL min⁻¹.

Fourier transform infrared spectroscopy (FTIR): FTIR spectra were recorded using a Thermo Scientific Nicolet iS50 FTIR in a transmission mode at room temperature. The wavenumber scale was from 4000 cm⁻¹ to 1000 cm⁻¹.

Powder X-ray diffraction (XRD): XRD patterns were measured using a Bruker D8 Advance diffractometer (Cu K α : $\lambda = 1.5418 \text{ \AA}$) under ambient conditions.

Thermal properties: Differential scanning calorimetry (DSC) analyses were performed on a Netzsch DSC214 Polyma instrument under a heating rate of 10°C/min and a nitrogen flow rate of 20 cm³/min. Thermogravimetric analysis (TGA) was conducted on a DTG-60 Shimadzu thermal analyst system under a heating rate of 10°C/min and a nitrogen flow rate of 50 cm³/min.

Density measurement: A certain mass (*m*) of sample was weighted and put into a 10 mL volumetric flask. The total mass of the sample and volumetric flask was also weighted (*m*₁). A certain volume (~8 mL) of n-pentane liquid was introduced into the volumetric flask. After sonicating the mixture for 10 s, excess n-pentane was introduced into the volumetric flask to its exact volume of 10 mL. The total mass of the mixture was measured (*m*₂). Since the density of n-pentane is 0.626 g/cm³, from its weight (*m*₂-*m*₁), the volume of the n-pentane is (*m*₂-*m*₁)/0.626 mL. Thus, the volume of the sample (*V*_{sample}) is 10-(*m*₂-*m*₁)/0.626, and the density of the sample can be calculated by the density

formula (m/V_{sample}). To improve the accuracy of the measurement, the density was averaged for 5 times and the average of them (ρ_{Avg}) was adopted based these independent measurements ($\rho_{\text{Avg}}=(\rho_1+\rho_2+\rho_3+\rho_4+\rho_5)/5$).

3 Photophysical investigations

Photophysical characterizations: UV-vis adsorption spectra were measured by a UV-vis spectrometer (UV-3600, Shimadzu). Excitation-phosphorescence mapping spectra were measured by a HITACHI F-4700 fluorescence spectrophotometer. Steady-state photoluminescent (PL) spectra, phosphorescent spectra, fluorescent and phosphorescent lifetimes and transient PL decay image were measured on an Edinburgh FLS 980 fluorescence spectrophotometer equipped with a xenon arc lamp (Xe900), a microsecond flash-lamp (μF900) and a picosecond pulsed light emitting diodes. The absolute total photoluminescent quantum yields were also measured on FLS 980 equipment with the help of an integral sphere, which covers both fluorescent and organic ultralong room-temperature phosphorescent (OURTP) emission components.

Photophysical parameters were deduced by the following equations ¹⁻⁵.

$$\tau_F = \frac{1}{k_r^F + k_{nr}^F + k_{ISC}} \quad (\text{Equation S1)}$$

$$\tau_{ph} = \frac{1}{k_r^{ph} + k_{nr}^{ph}} \quad (\text{Equation S2)}$$

$$k_r^F = \frac{\Phi_F}{\tau_F} \quad (\text{Equation S3})$$

$$k_{nr}^F = \frac{1 - \Phi_F - \Phi_{ISC}}{\tau_F} \quad (\text{Equation S4})$$

$$k_{nr}^F \text{min} = \frac{1 - \Phi_F - \Phi_{ISC}^{max}}{\tau_F} \quad (\text{Equation S5})$$

$$k_{nr}^F \text{max} = \frac{1 - \Phi_F - \Phi_{ISC}^{min}}{\tau_F} \quad (\text{Equation S6})$$

$$\frac{\Phi_{ph}}{\tau_F} \leq k_{ISC} \leq \frac{1 - \Phi_F}{\tau_F} \quad (\text{Equation S7})$$

$$k_{ISC}^{min} = \frac{\Phi_{ph}}{\tau_F} \quad (\text{Equation S8})$$

$$k_{ISC}^{max} = \frac{1 - \Phi_F}{\tau_F} \quad (\text{Equation S9})$$

$$k_r^{ph} = \frac{\Phi_{ph}}{\Phi_{ISC} \times \tau_{ph}} \quad (\text{Equation S10})$$

$$k_r^{ph, min} = \frac{\Phi_{ph}}{\Phi_{ISC}^{max} \times \tau_{ph}} \quad (\text{Equation S11})$$

$$k_r^{ph, max} = \frac{\Phi_{ph}}{\Phi_{ISC}^{min} \times \tau_{ph}} \quad (\text{Equation S12})$$

$$k_{nr}^{ph} = \frac{1}{\tau_{ph}} - k_r^{ph} \quad (\text{Equation S13})$$

$$k_{nr}^{ph, min} = \frac{1}{\tau_{ph}} - k_r^{ph, max} \quad (\text{Equation S14})$$

$$k_{nr}^{ph, max} = \frac{1}{\tau_{ph}} - k_r^{ph, min} \quad (\text{Equation S15})$$

$$\Phi_{ISC} = \frac{k_{ISC}}{k_r^F + k_{nr}^F + k_{ISC}} = k_{ISC} \times \tau_F \quad (\text{Equation S16})$$

$$\Phi_{ISC}^{min} = k_{ISC}^{min} \times \tau_F \quad (\text{Equation S17})$$

$$\Phi_{ISC}^{max} = k_{ISC}^{max} \times \tau_F \quad (\text{Equation S18})$$

τ_F : fluorescence (F) lifetime; τ_{ph} : phosphorescence (ph) lifetime; Φ_{ISC} : quantum yield of intersystem crossing (ISC); k_r^F : rate constant of radiative decay from S_1 to the ground states (S_0) for fluorescence (F); k_{nr}^F : rate constant of non-radiative decay from S_1 to S_0 ; k_{ISC} : rate constant of ISC; k_r^{ph} : rate constant of radiative decay from T_1 to S_0 for phosphorescence (ph); k_{nr}^{ph} : rate constant of non-radiative decay from T_1 to S_0 ; min: the minimum value; max: the maximum value.

4 Applications

Water-jet printing of the lifetime-encrypted patterns: To realize the reprintable water-jet printing of the lifetime-encrypted patterns, three steps were carried out.

- (1) Fabrication of rewritable encryption paper: High-concentration **PAMCz1.0** (3.0 g/mL) aqueous solution was screen printed on a A4 paper and heated in an oven at 70°C for 2 h to remove the solvent of water.
- (2) Water-jet printing process: The desired pattern in high resolution was printed by a commercially available printer (HP DeskJet 1111) using pure water as ink. The part that is not printed will show persistent afterglow; the printed pattern appears in dark because the afterglow was quenched due to the presence of the printed water.
- (3) Erasing process: The pattern without afterglow emission printed on the rewritable paper was erased facilely by drying at 70°C for 30 min. Thus treated paper can be reused directly for next water-jet printing cycle of another pattern.

Multi-color water-jet printing of anti-counterfeiting patterns: Similarly, the desired pattern was printed by a commercially available printer (HP DeskJet 1111) but using water soluble dye solution as ink. After drying at 70°C for 30 min to remove water, yellow and orange afterglow patterns appears due to triplet-to-singlet energy transfer.

5 Figures

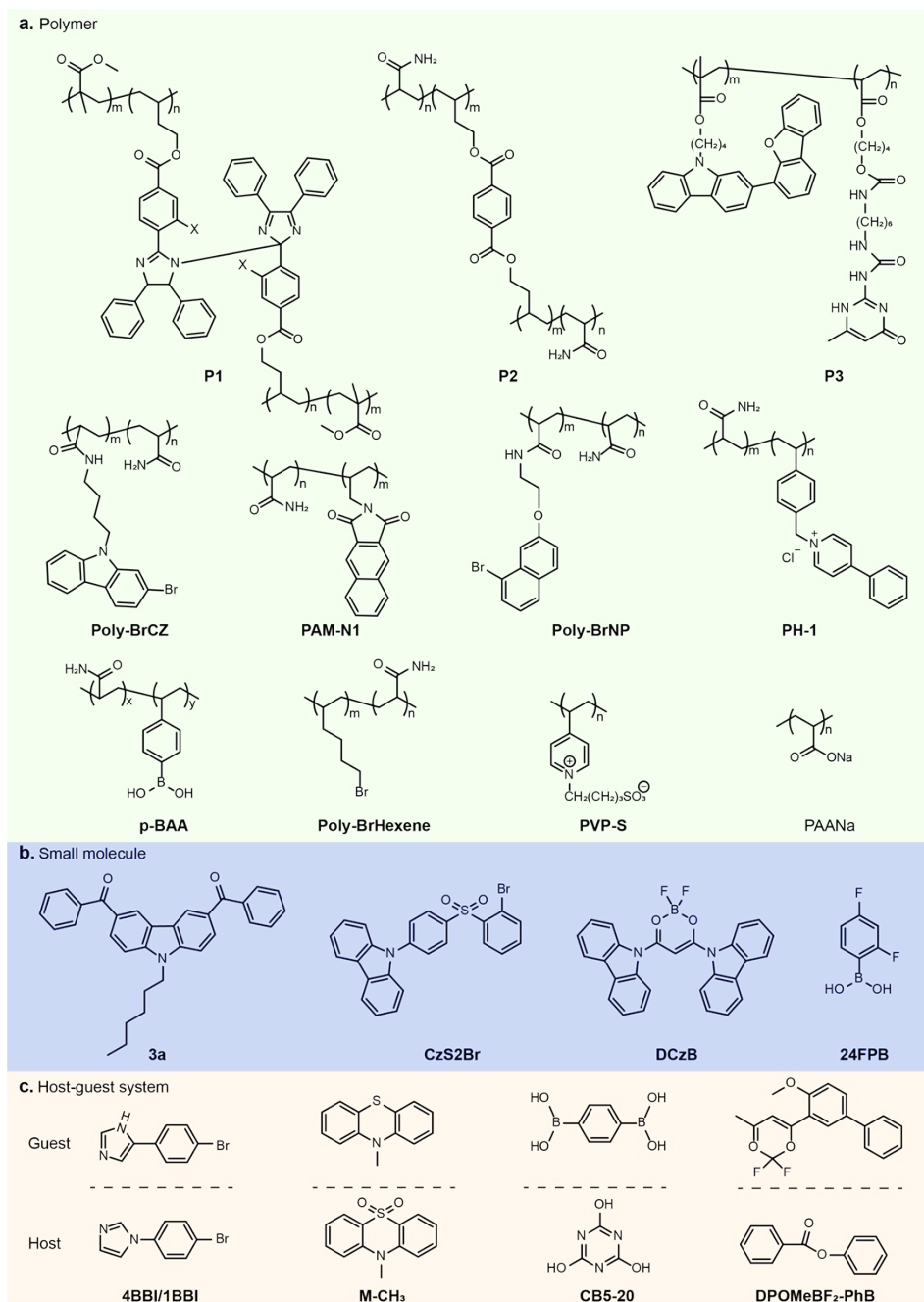


Fig. S1. Molecular structures of typical OURTP polymers (a), small molecules (b) and host-guest systems (c).^{2, 6-23}

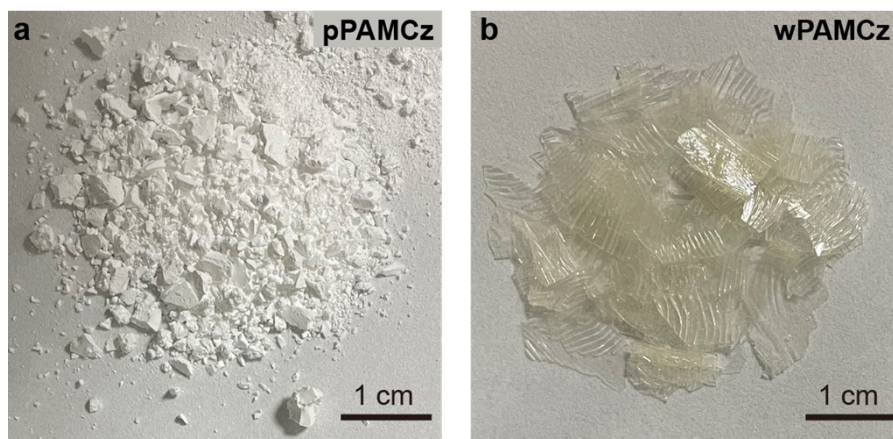


Fig. S2. Photographs of pPAMCz in powder (a) and wPAMCz in flake (b) forms under daylight taken by iPhone 12.

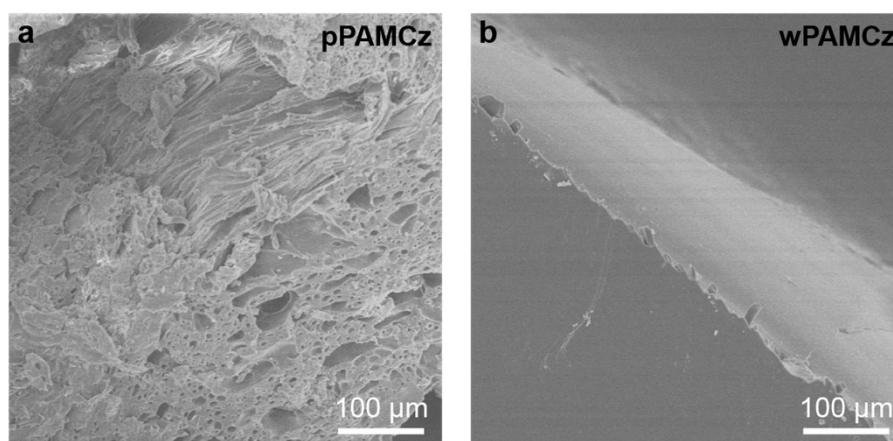


Fig. S3. SEM images of the cross sections of pPAMCz (a) and wPAMCz (b).

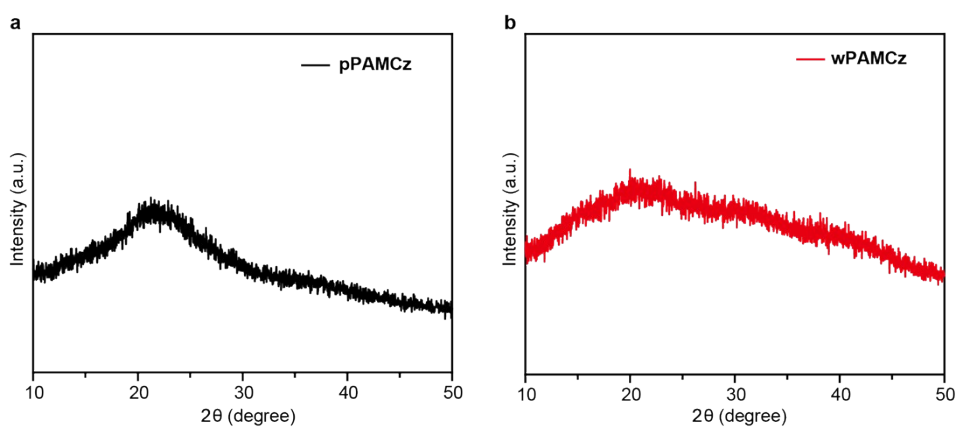


Fig. S4. XRD patterns of pPAMCz (a) and wPAMCz (b).

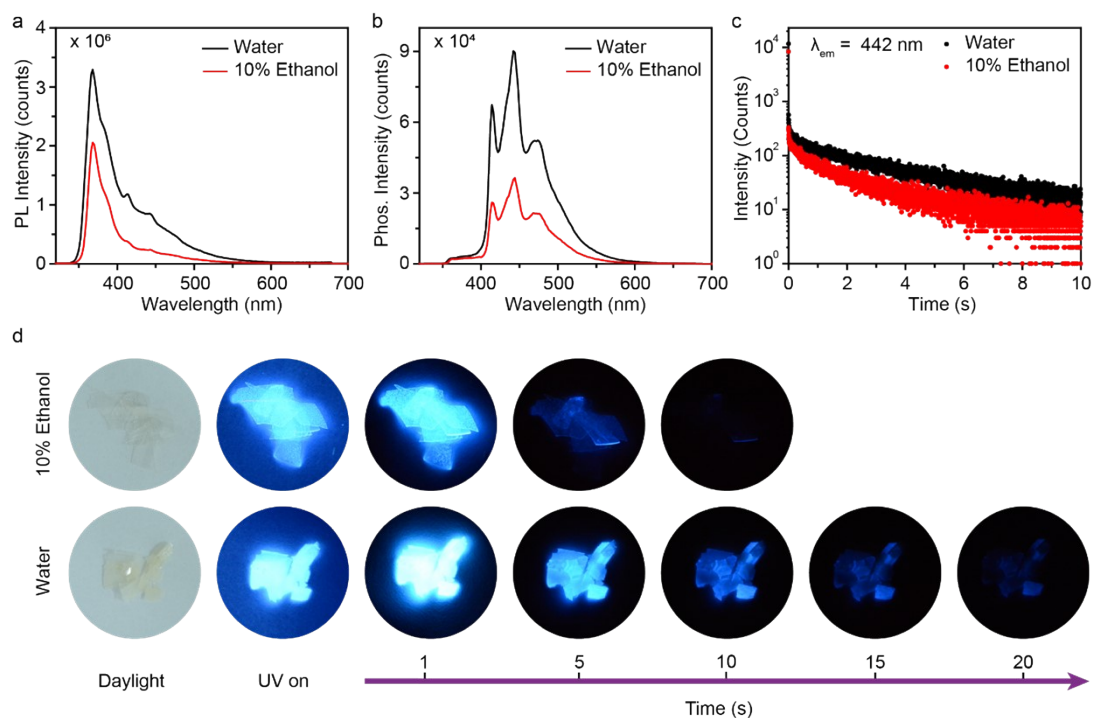


Fig. S5. Steady-state PL (a) and phosphorescence (d) spectra, 442 nm decay profiles (c) and photographs (d) of wPAMCz prepared by aggregation engineering using pure water (Water, black line) and 10 wt% ethanol aqueous solvent (10% Ethanol, red line) at 298 K. The photographs were taken under daylight, 365 nm UV on and UV off by Nikon D9.

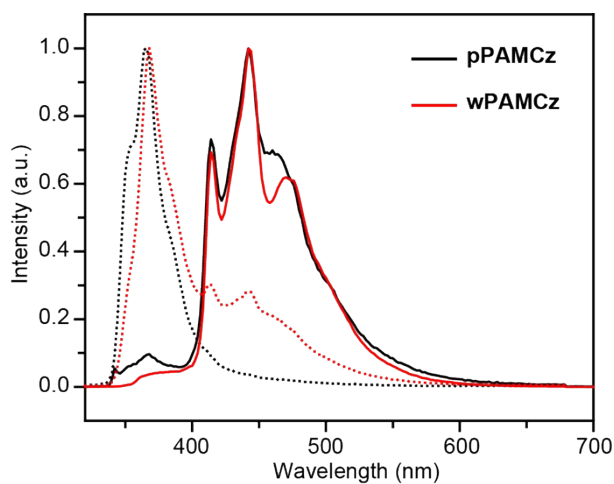


Fig. S6. Steady-state PL (dash line) and phosphorescence (solid line, delay 10 ms) spectra of pPAMCz and wPAMCz at 298 K.

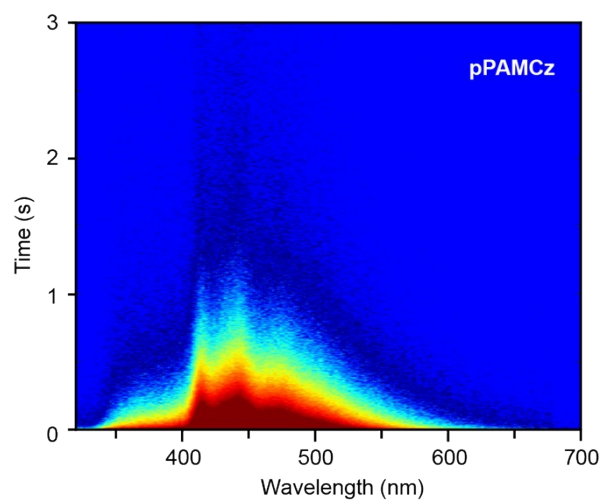


Fig. S7. Time resolved emission spectroscopy of **pPAMCz** at room temperature. The excitation wavelength is 300 nm.

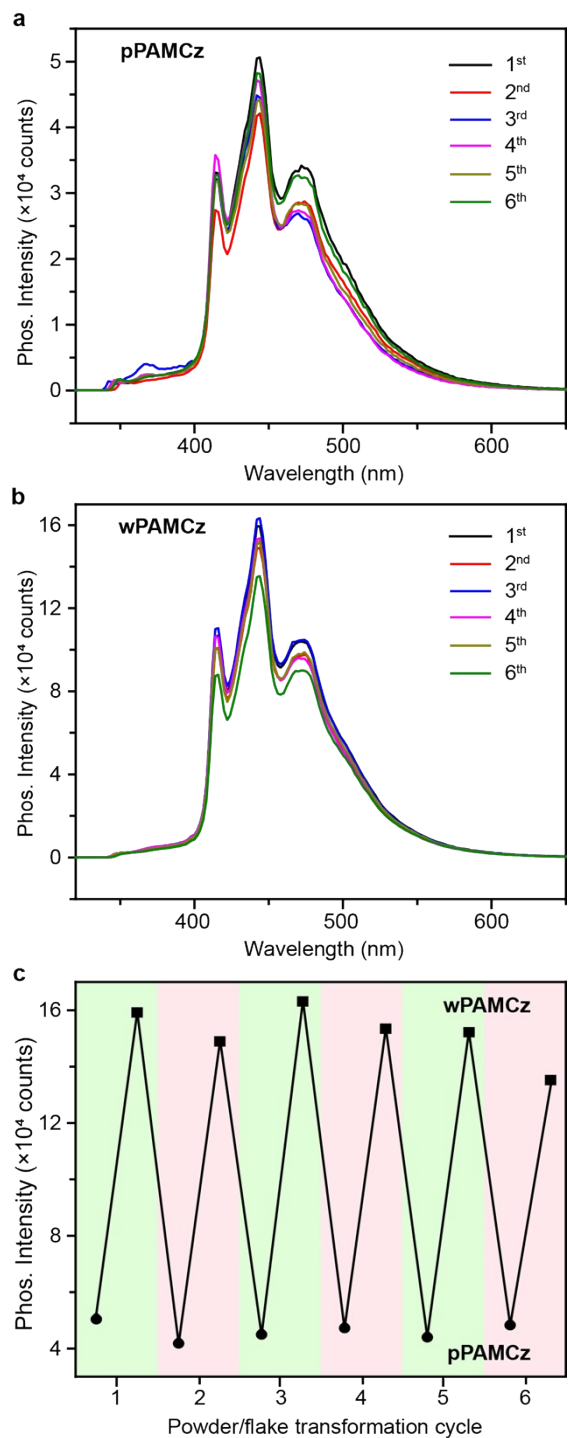


Fig. S8. High transformation repeatability of **PAMCz** from powder to flake: OURTP spectra of **PAMCz** in powder (a) and flake (b) forms for different transformation cycles with slightly varied OURTP intensities (c).

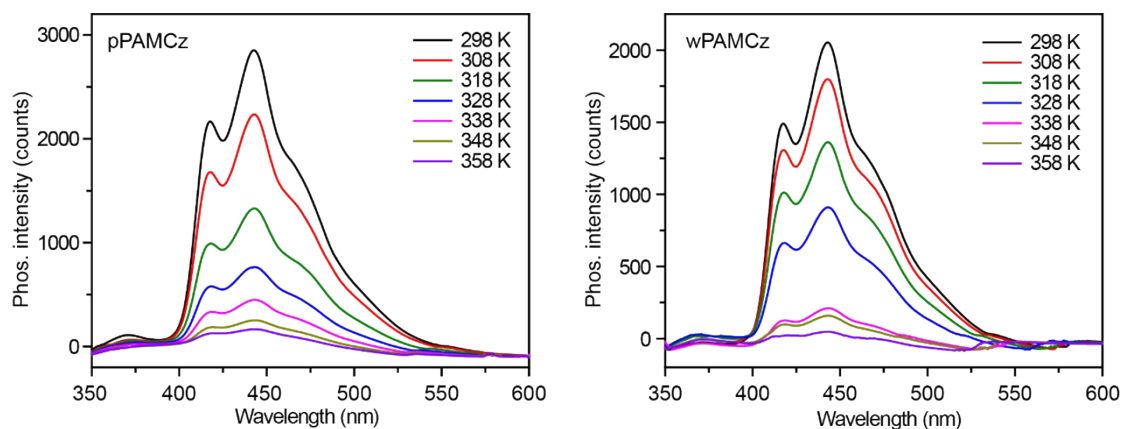


Fig. S9. Phosphorescence spectra of pPAMCz and wPAMCz at temperatures from 298 to 358 K.

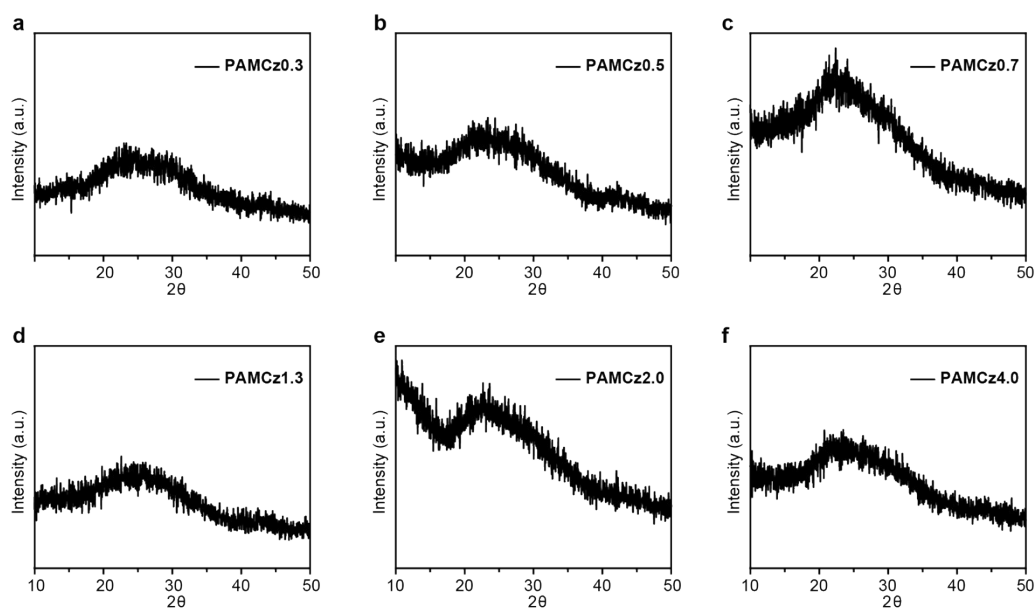


Fig. S10. XRD patterns of PAMCz0.3 (a), PAMCz0.5 (b), PAMCz0.7 (c), PAMCz1.3 (d), PAMCz2.0 (e) and PAMCz4.0 (f) in flakes.

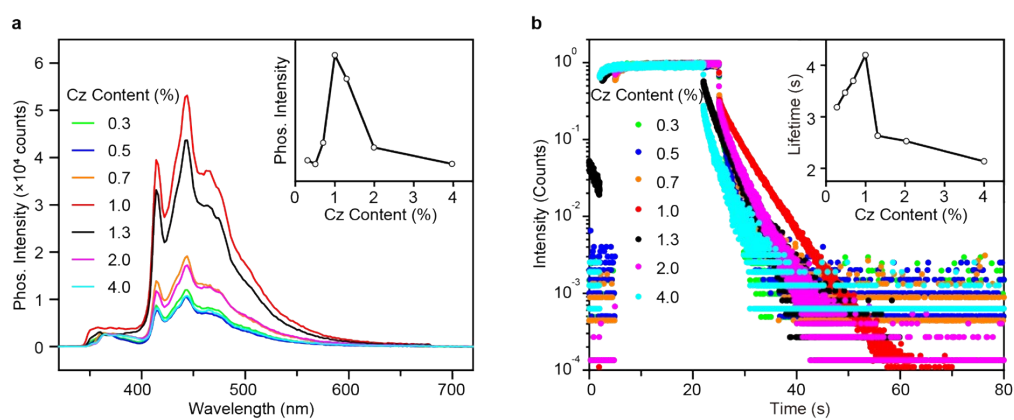


Fig. S11. Phosphorescence spectra (a) and OURTP decay curves (b) of PAMCz copolymers with different carbazole (Cz) contents in feed under the same test conditions.

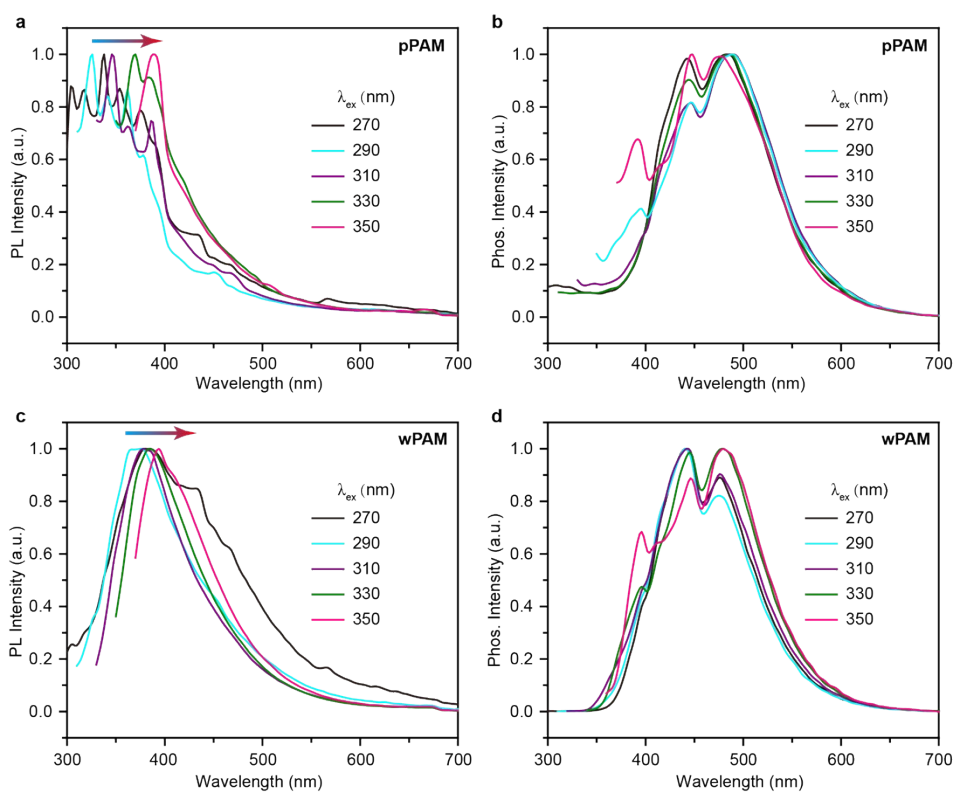


Fig. S12. Steady-state PL (a, c) and phosphorescence spectra (b, d) of **pPAM** (a, b) and **wPAM** (c, d) at different excitation wavelength (λ_{ex}) under ambient conditions.

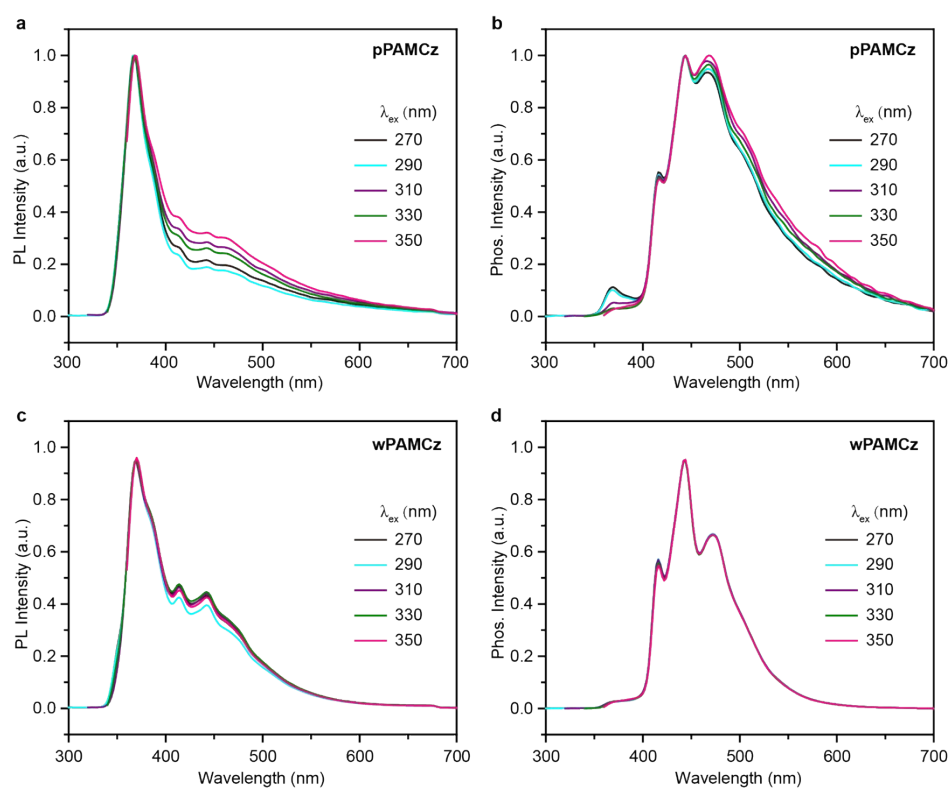


Fig. S13. Steady-state PL (a, c) and phosphorescence spectra (b, d) of **pPAMCz** (a, b) and **wPAMCz** (c, d) at different excitation wavelength (λ_{ex}) under ambient conditions.

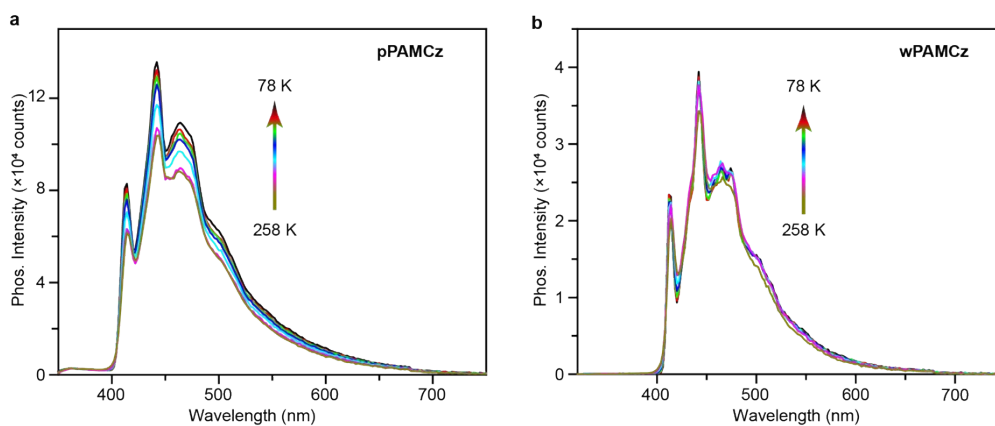


Fig. S14. Phosphorescence spectra of **pPAMCz** (a) and **wPAMCz** (b) under argon atmosphere at varied temperature from 258 to 77 K.

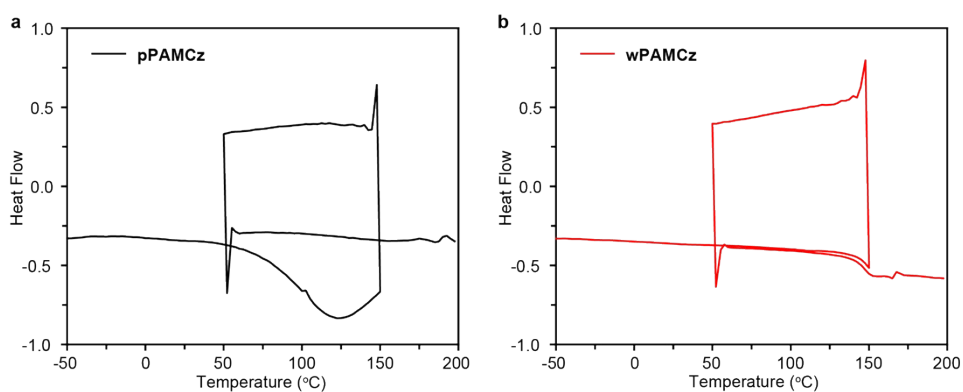


Fig. S15. DSC curves of (a) **pPAMCz** and (b) **wPAMCz**. The test process can be divided into three stages: firstly, heating from -50°C to 150°C ; then, cooling from 150°C to 50°C ; and, finally heating from 50°C to 200°C .

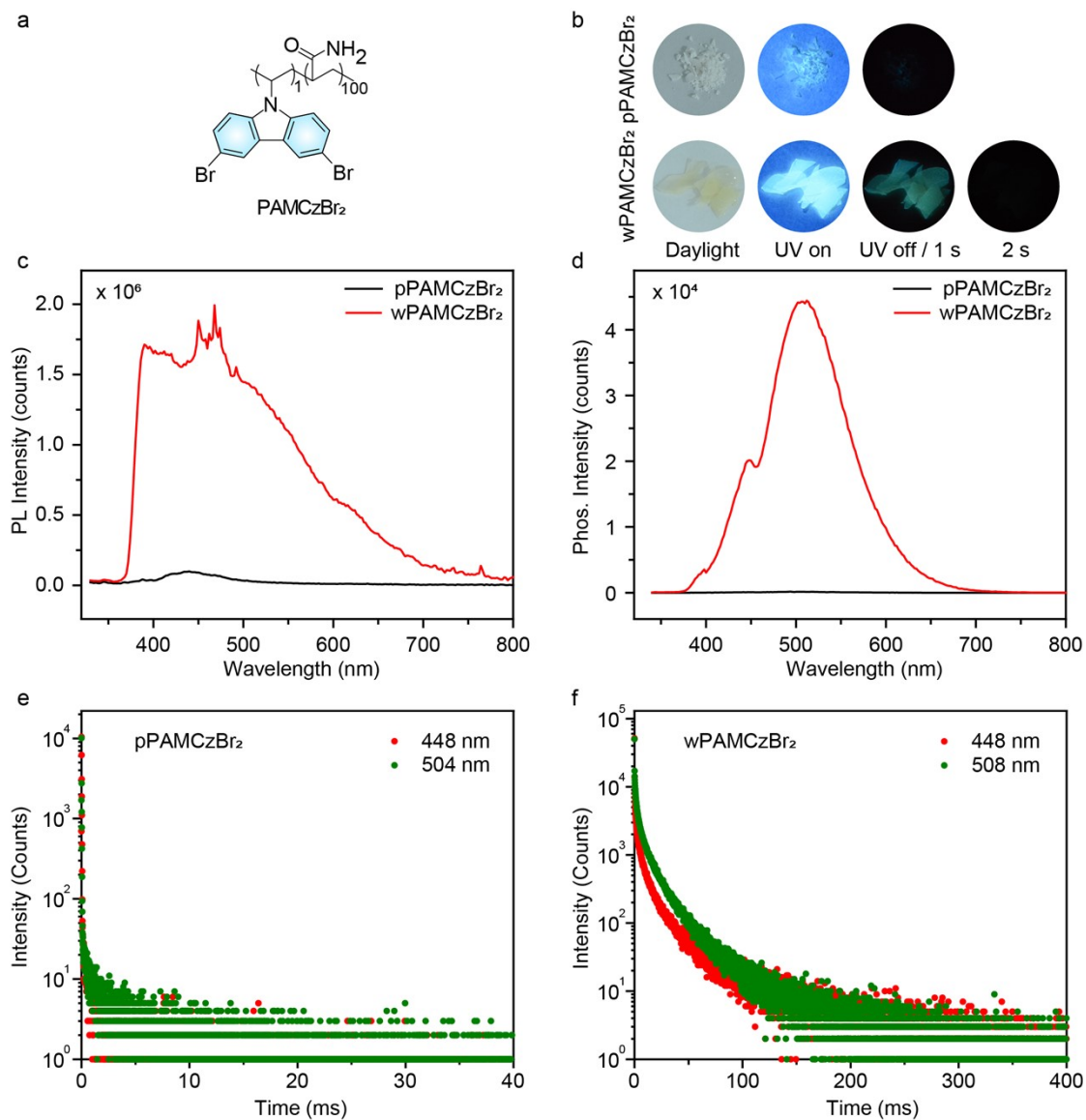


Fig. S16. (a) Structural formula of **PAMCzBr₂**. (b) Photographs of **pPAMCzBr₂** and **wPAMCzBr₂** under daylight, 365 nm UV on and UV off taken by Nikon D9. (c-d) Steady-state PL (c) and phosphorescence (d) spectra of **pPAMCzBr₂** (black) and **wPAMCzBr₂** (red) at 298 K. (e-f) Decay profiles of **pPAMCzBr₂** (e) and **wPAMCzBr₂** (f).

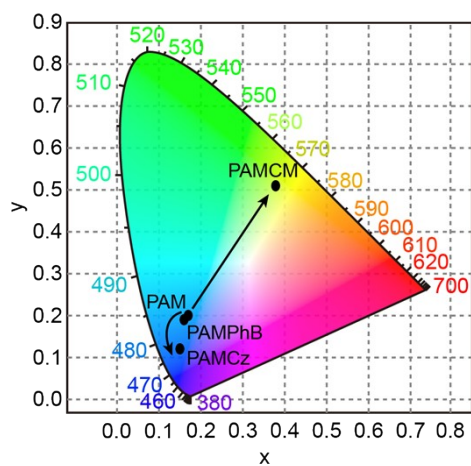


Fig. S17. CIE 1931 coordinates of afterglow emissions of **wPAM**, **wPAMCz**, **wPAMPhB** and **wPAMCM** at 298 K.

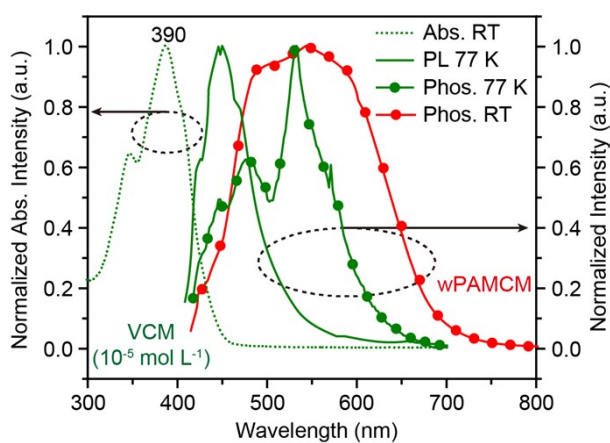


Fig. S18. UV-Visible absorption (green dashed line, 298 K), steady-state PL (green solid line, 77 K) and phosphorescent spectra (green dot dash line, 77 K) of **VCM** in toluene (10^{-5} mol L $^{-1}$) with the excitation of 390 nm UV light. Phosphorescent spectra (red dot dash line, 298 K) of **wPAMCM** with the excitation of 396 nm UV light.

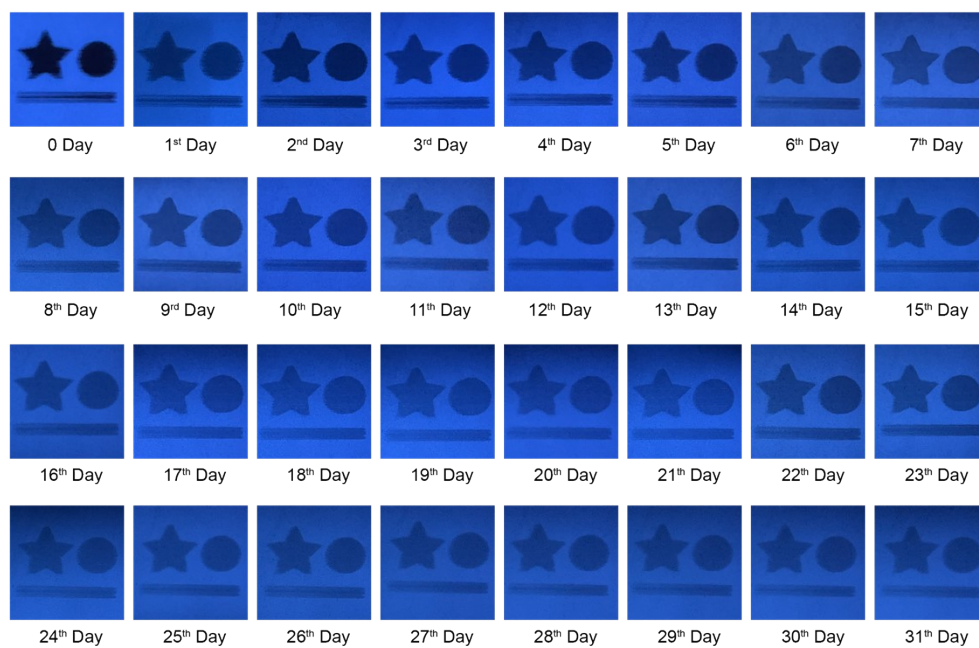


Fig. S19. Photographs of the freshly printed pattern (0 day) and in 31 days. The photographs were taken by iphone 12 with 1 s delay after the removal of 254 nm UV lamp.

6 Tables

Table S1. OURTP performance of amorphous polymers (green shaded), small molecular crystals (blue shaded) and host-guest systems (brown shaded). λ_{ph} , τ_{p} and Φ_{p} are OURTP peak, lifetime and quantum yield and the molecular structures were illustrated in Supplementary Figure 1.

Sample	λ_{ph} (nm)	τ_{p} (ms)	Φ_{p} (%)	Ref.
P1	545	20	8	22
poly-BrCZ	453	1	5	17
P2	427	537	15	11
poly-BrNp	520	6	8	8
<i>p</i> -BAA	477	351	-	6
P3	530	1700	8	10
PAM-N1	494	1535	12	18
PH-1	490	1463	10	16
PVP-S	524	578	6	13
poly-BrHexene	436	1	3	23
PAANa	480	712	6	19
3a	517	4171	11	12
CzS2Br	550	180	52	15
DCzB	495	381	45	20
24FPB	515	2500	2	9
M-CH ₃	500	17000	20	7
CB5-20	406	5080	38	2
1BBI-4BBI	482	8	74	21
DPOMeBF ₂ -PhB	462	301	69	14

Table S2. Characterizations of the acrylamide (AM)-based polymers. ^a

Polymer	Feed ratio of VCz:AM (mol:mol)	VCz in feed (mol %)	VCz in copolymer (mol %)	M_{n} (KDa)	M_{w} (KDa)	PDI
PAM	0:100	0	0	9.0	27.2	2.3
PAMCz0.3	1:300	0.33	0.36	13.2	34.2	2.6
PAMCz0.5	1:200	0.5	0.49	13.5	34.7	2.5
PAMCz0.7	1:150	0.67	0.65	13.0	35.8	2.7
PAMCz1.0	1:100	0.99	0.93	13.3	33.7	2.5
PAMCz1.3	1:75	1.32	1.37	11.3	51.1	4.5
PAMCz2.0	1:50	1.96	1.89	18.6	39.4	2.1

PAMCz4.						
0	1:25	3.85	3.64	14.7	43.4	2.9

a: The actual content of VCz unit was calculated from ^1H NMR spectrum. The number-averaged (M_n) and weight-averaged (M_w) molecular weights of the polymers with different polydispersity (PDI) were measured by GPC.

Table S3. Photoluminescence properties and photophysical kinetic parameters of **pPAM**, **wPAM**, **pPAMCz** and **wPAMCz** at room temperature.

Parameter	pPAM	wPAM	pPAMCz	wPAMCz
λ_F (nm)	370	378	368	368
λ_{Ph} (nm)	443	444	442	442
τ_F (ns)	7.98	4.70	12.41	13.20
PLQY	0.057	0.055	0.288	0.235
Φ_{Ph}	0.017	0.032	0.107	0.155
τ_{Ph} (s)	0.039	0.196	0.511	4.205
Φ_F	0.040	0.023	0.181	0.080
k_r^F (s^{-1})	5.01×10^6	4.89×10^6	1.46×10^7	6.06×10^6
$k_{nr}^F \text{ max}$ (s^{-1})	1.18×10^8	2.01×10^8	5.74×10^7	5.80×10^7
k_{ISC}^{max} (s^{-1})	1.20×10^8	2.08×10^8	6.60×10^7	6.97×10^7
k_{ISC}^{min} (s^{-1})	2.13×10^6	6.81×10^6	8.62×10^6	1.17×10^7
Φ_{ISC}^{max}	0.960	0.977	0.819	0.920
Φ_{ISC}^{min}	0.017	0.032	0.107	0.155
$k_r^{\text{Ph max}}$ (s^{-1})	25.641	5.102	1.957	0.238
$k_r^{\text{Ph min}}$ (s^{-1})	0.454	0.167	0.256	0.040
$k_{nr}^{\text{Ph max}}$ (s^{-1})	25.187	4.935	1.701	0.198

Table S4. Photoluminescence properties and photophysical kinetic parameters of **pPAMPhB**, **wPAMPhB**, **pPAMCM** and **wPAMCM** at room temperature.

Parameter	pPAMPhB	wPAMPhB	pPAMCM	wPAMCM
λ_F (nm)	374	410	498	498
λ_{Ph} (nm)	470	468	532	546
PLQY	0.035	0.039	0.072	0.040
Φ_{Ph}	0.031	0.034	0.021	0.042
τ_F (ns)	2.99	3.26	3.61	3.41
τ_{Ph} (s)	0.152	0.252	0.122	0.248

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