Electronic Supplementary Material (ESI) for Journal of Materials Chemistry C.

Supporting Information

Purely Organic Near-Infrared Afterglow Systems based on a Triplet Excimer Donor

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Experimental Procedures

1. General materials

Materials: Cy7 fluorescent dye, K_2CO_3 and polyvinyl alcohol (PVA-1788, alcoholysis ratio of 87.0-89.0% mol/mol, Mw = 85000-124000) were all purchased from Aladdin Chemical (Shanghai, China). Naphthalene-2-hylotropic acid (NAB), phenanthren-9-ylboronic acid (PHB), pyren-1-ylboronic acid (PYB) and 4,4,5,5-tetramethyl-2-(pyren-1-yl)-1,3,2-dioxaborolane (PYBE) were purchased from Bide Pharmatech Ltd. (China). All these materials are analytical grade and used as received. The pig skin was purchased from a market close to the university.

Characterizations and instruments: The samples were measured in air atmosphere except of otherwise stated. The Fourier transform infrared spectroscopy (FT-IR) were recorded using a NICOLET 6700 spectrophotometer. The high performance liquid chromatography (HPLC) was measured on an Agilent HP1200 chromatography. The ultraviolet-visible (UV-Vis) absorption spectra were measured on a Hitachi UV-3600 or U-3900 spectrometer. The photoluminescence profiles were collected from a Horiba JY FL-3or a SHIMADZU FR-5301pc spectrophotometer. The delayed spectra were measured on an Ocean Optics QE65 Pro system which was triggered with an LED light source (365 nm, Rhino LED, Guangzhou Xi Pu Patellectomies Technology Co., Ltd.) or a Horiba JY FL-3 spectrophotometer (excited by 333 nm) or a Perkin Elmer Fluorescence Spectrometer FL 8500 (excited by 330 nm). The daylight photos, phosphorescence photos and NIR light photos are captured by the iPhone 13 camera, Redmi K50 camera and NIR camera respectively, using a 365 nm flashlight (100 mW/cm^2) as the light source. The luminescent lifetime decay curves were obtained on a Horiba JY FL-3 spectrophotometer. The lifetime decay curves of Cy7/PYB@PVA films at 808 nm were obtained from their delayed spectra. The PL quantum yields (QY) were performed on a Horiba JY FL-3 spectrophotometer equipped with a calibrated integrating sphere (HAMAMATSU C11347). And the phosphorescence QY was calculated based on the steady-state spectra and the total photoluminescence QY.

The quantum chemistry calculation was performed on the Gaussian 09[1] software at the B3LYP/6-31G+(d, p) level of theory using the density functional theory (DFT) method. The exciton energy levels were calculated from the corresponding ground state geometry using the combination of TD-B3LYP/6-31G+(d, p).

2. Preparation of doped film

A carbonate buffer was prepared with 1 g K_2CO_3 in 10 mL water, in order to maintain the alkaline of the aqueous solution. Alkaline PYB solution with PYB content of 10^{-3} mol/L was then obtained by adding 200 μL carbonate buffer into ethanol. The aqueous solution of PVA (1.0 wt%) was prepared in hot deionized water. The mixed solutions were then prepared by drop wise addition of aqueous PVA (1.0 mL) into a PYB solution with different volumes under continuous shaking. The PYB@PVA mixture was slowly poured into a culture dish, and then the dish was treated with ultrasound to drive out the bubbles. Finally, thin and transparent films were obtained after drying the mixtures at 65 °C for 5 hours. Other doped films are made in a similar process.

3. Figures and tables

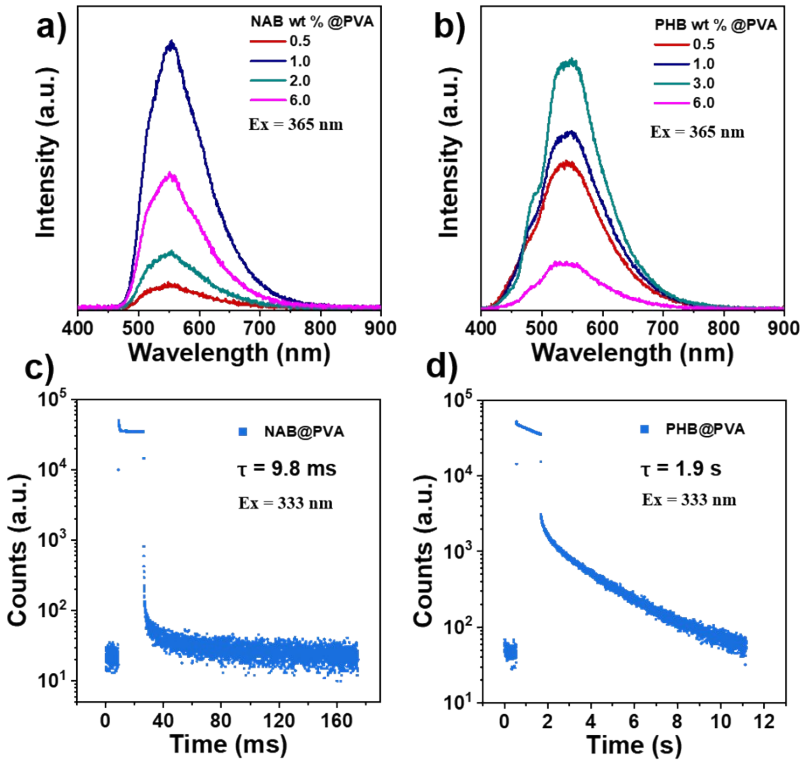


Figure S1. (a) The phosphorescence spectra (delayed 8 ms) of (a) NAB@PVA and (b) PHB@PVA films with different contents of NAB and PHB. The time-resolved intensity decay of (c) NAB_1@PVA containing 1.0 wt% NAB (measured at 551 nm) and (d) PHB_3@PVA containing 3.0 wt% PHB (measured at 547 nm).

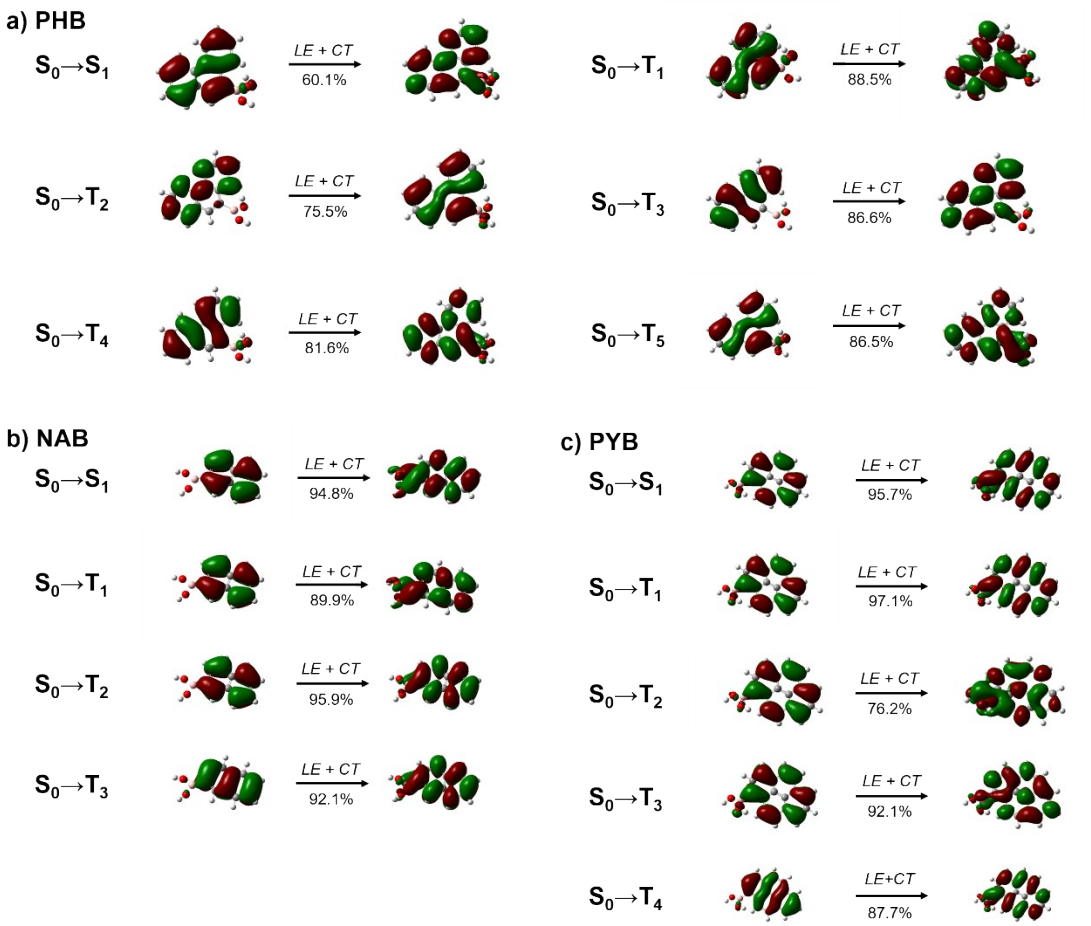


Figure S2. The natural transition orbitals of dyes: (a) PHB, (b) NAB and (c) PYB. LE, locally-excited; CT, charge transfer.

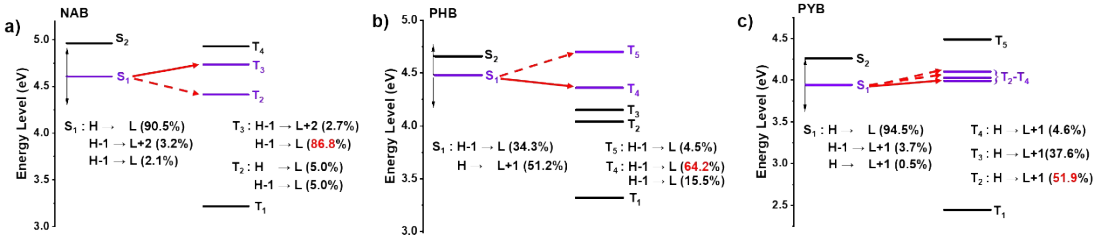


Figure S3. The arrangement of energy levels of (a) NAB, (b) PHB, and (c) PYB including their possible transition channels for ISC transition: the (n, π) → (π, π*) type transition channels of HOMO-1 → LUMO+1 (H-1 → L+1) and their contribution values are inset. The solid arrow is for major ISC channels, while the dashed arrow is for minor ISC channels, respectively.

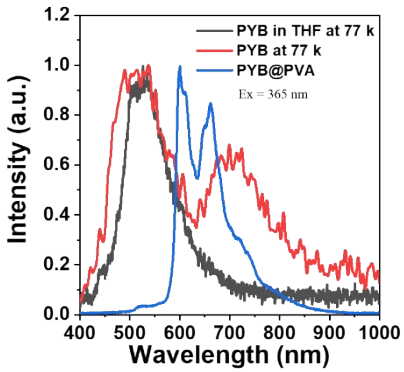


Figure S4. The phosphorescence spectra (delayed 8 ms) of PYB in THF solution at 77 k and PYB solid at 77 k and PYB@PVA film at room temperature.

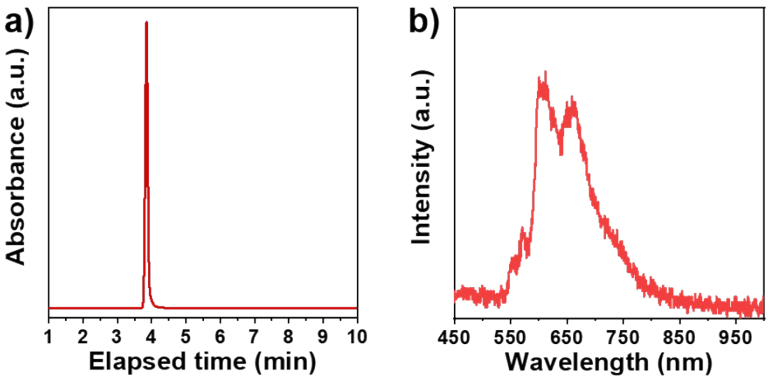


Figure S5. (a) The HPLC spectrum of PYB (Purity of PYB: 99.4%). (b) The phosphorescence spectrum (delayed 8 ms) of 4,4,5,5-tetramethyl-2-(pyren-1-yl)-1,3,2-dioxaborolane (PYBE)@PVA with 1 wt.% content.

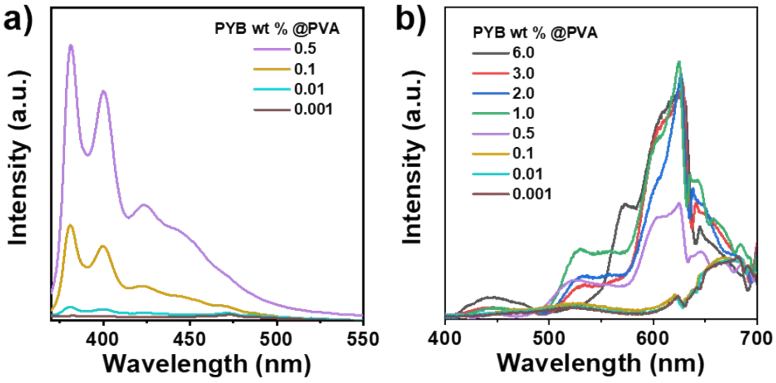


Figure S6. The PL properties of PYB@PVA films with different PYB contents: (a) Steady-state photoluminescence spectra. (b) Delayed luminescence spectra measured when delayed 25 ms, excited at 330 nm.

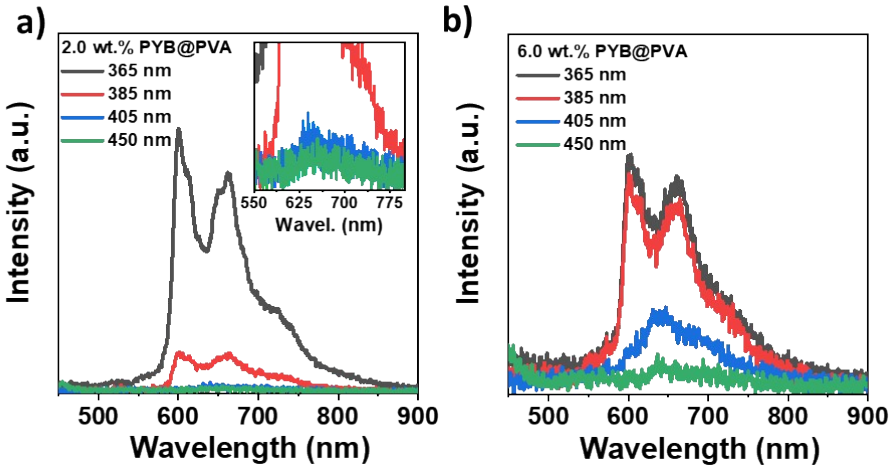


Figure S7. The delayed PL spectra (delayed 8 ms) of (a) PYB_2@PVA and (b) PYB_6@PVA films under different excitation wavelengths.

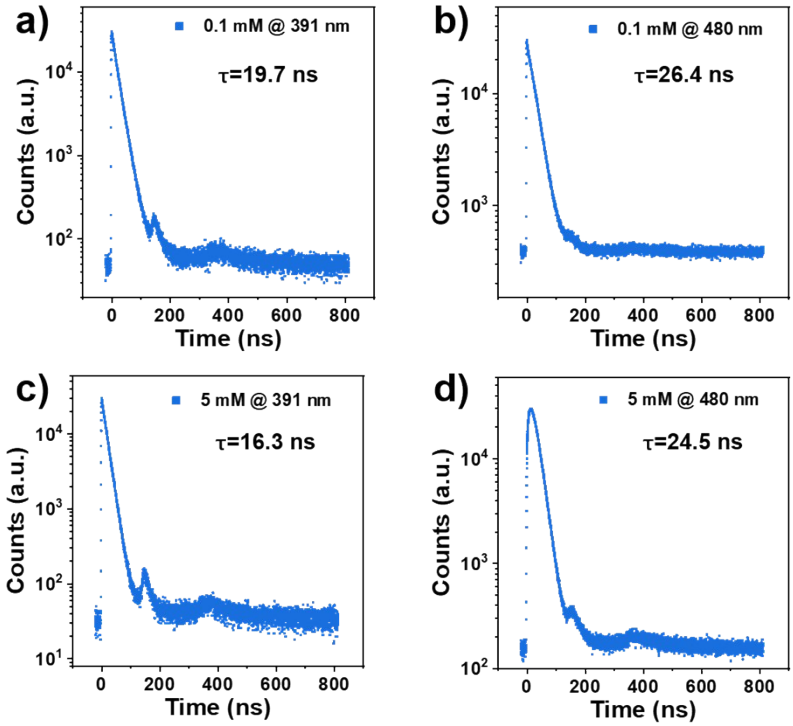


Figure S8. Time-resolved intensity decay of PYB in EtOH under different conditions (a) measured at 391 nm in 0.1mM, (b) measured at 480 nm in 0.1 mM, (c) measured at 391 nm in 5 mM and (d) measured at 480 nm in 5 mM (Ex = 352 nm).

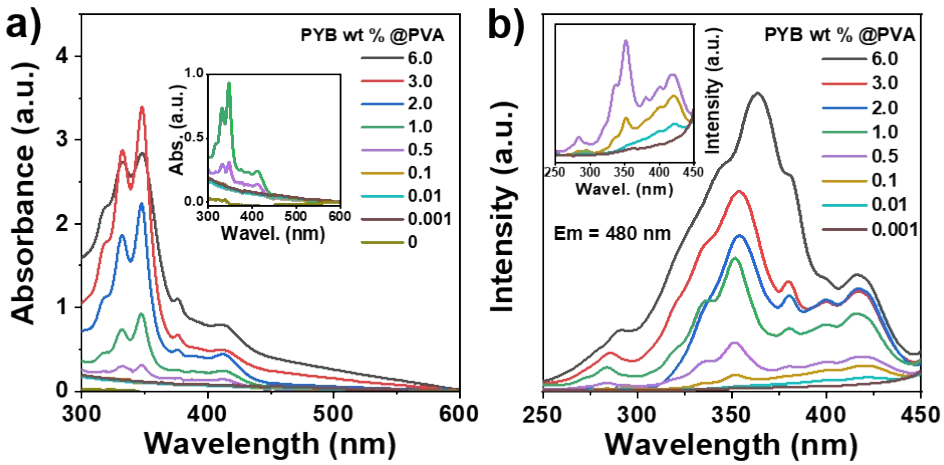


Figure S9. (a) UV absorption spectra and (b) excitation spectra (Em = 480 nm) of PYB@PVA films with different PYB contents. Partial enlarged spectra were inset, when PYB content was low. Wavel., wavelength; Abs., Absorbance.

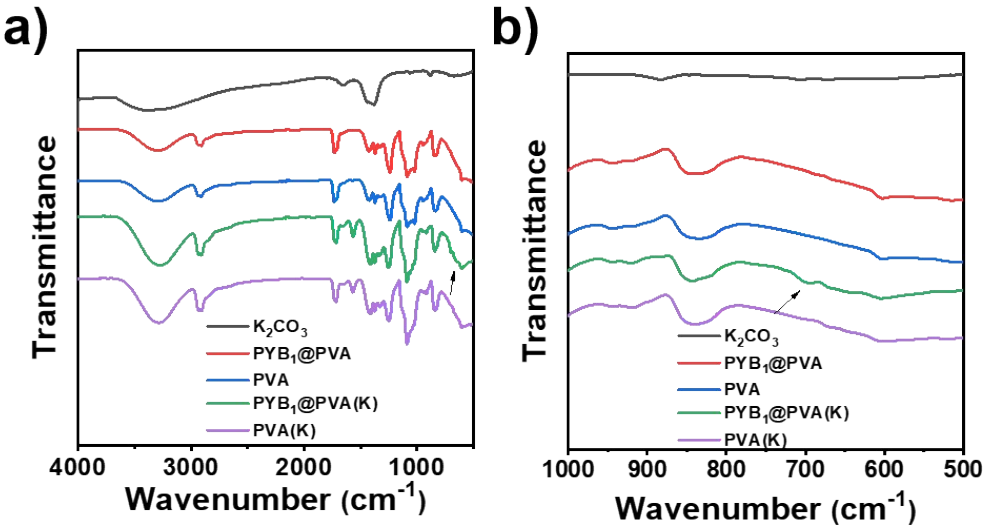


Figure S10. FTIR spectra (a) from 4000 cm^{-1} to 500 cm^{-1} and (b) from 1000 cm^{-1} to 500 cm^{-1} of K_2CO_3, PYB@PVA film, PVA film, PYB@PVA (K) film containing K_2CO_3 and PVA (K) containing K_2CO_3.

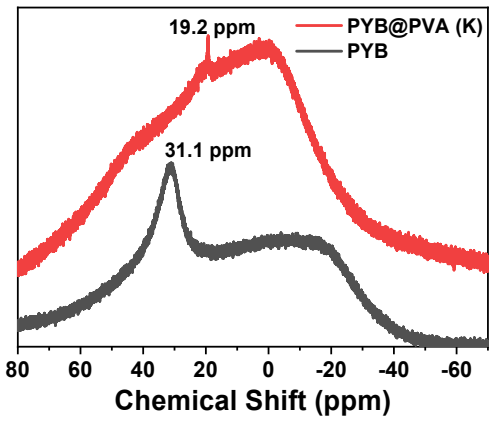


Figure S11. ^{11}B-NMR spectra of PYB@PVA (K) containing K_2CO_3 and PYB in DMSO-d_6.

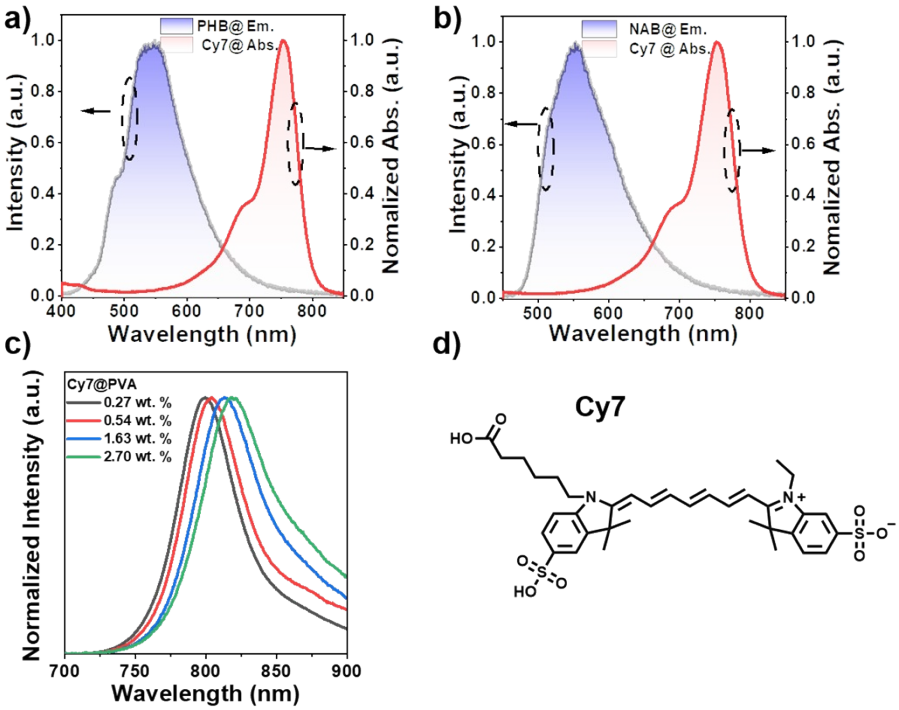


Figure S12. (a) Delayed luminescence spectrum of PHB_3@PVA (blue line, delayed 8 ms after turning off the UV light of 302 nm) and absorbance spectrum of Cy7@PVA (red line). (b) Delayed luminescence spectrum of NAB_1@PVA (blue line, delayed 8 ms after turning off the UV light of 302 nm) and absorbance spectrum of Cy7@PVA (red line). (c) The steady-state PL spectra of Cy7@PVA with different Cy7 contents (*E*x = 365 nm). (d) The chemical structure of Cy7.

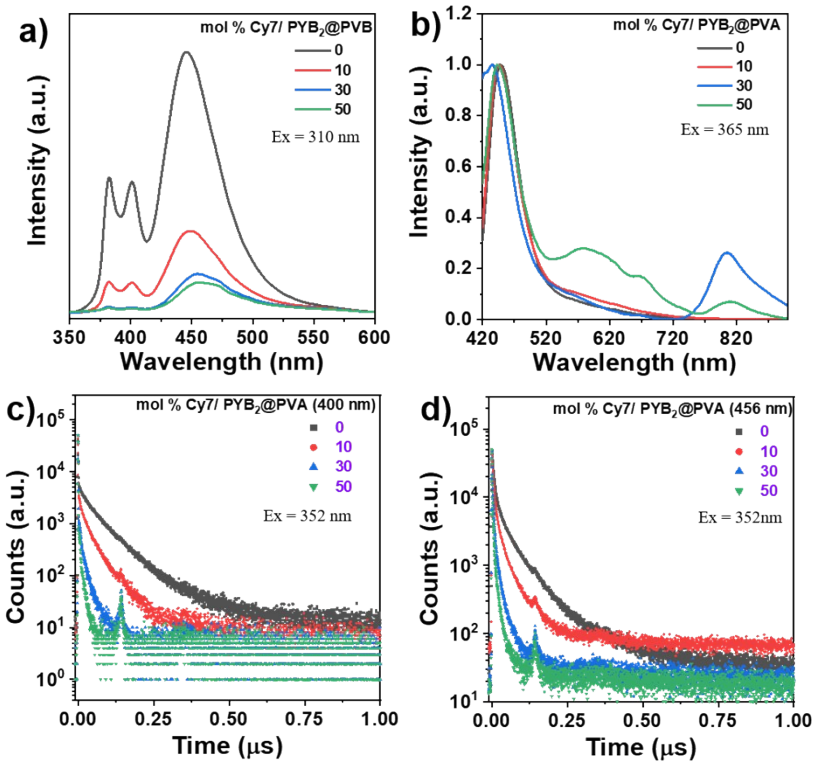


Figure S13. The steady-state spectra of Cy7/PYB_2@PVA with different contents of Cy7 (a) measured at 310 nm and (b) measured at 365 nm. Time-resolved intensity decay of Cy7/PYB_2@PVA with different contents of Cy7 (c) measured at 400 nm and (d) measured at 456 nm. The content of Cy7 is mol% to PYB amount and the content of PYB doped in PVA film is 2.0 wt% to PVA.

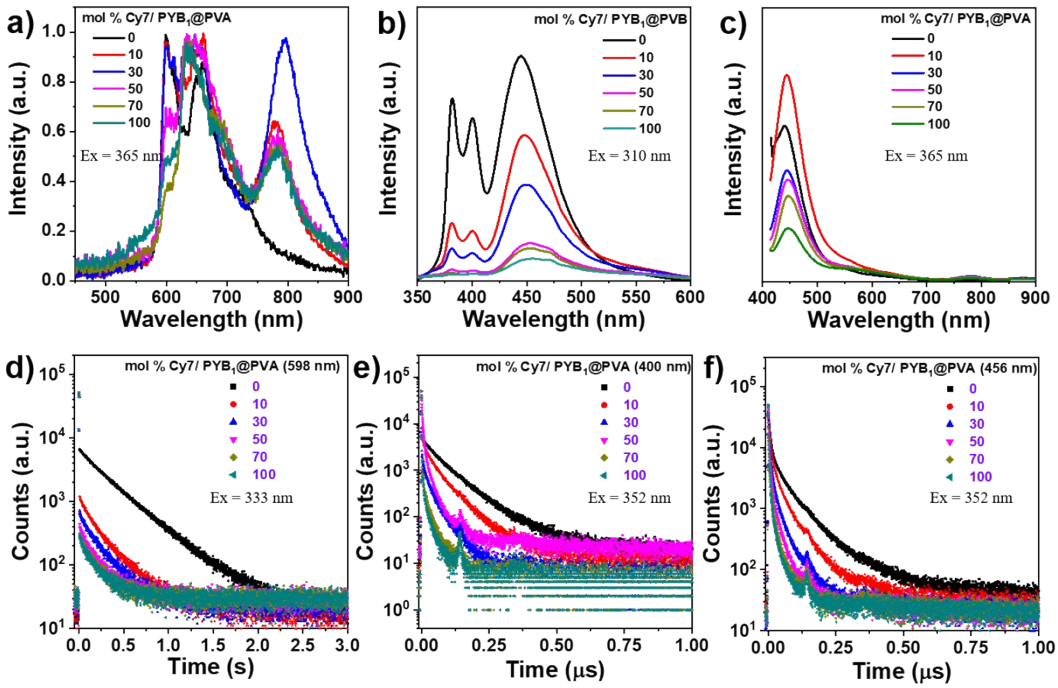


Figure S14. (a) The delayed spectra (delayed 8 ms) of Cy7/PYB_1@ PVA doped films with different Cy7 contents. The steady-state spectra of Cy7/PYB_1@ PVA doped films with different Cy7 contents (b) measured at 310 nm and (c) measured at 365 nm. (d) The phosphorescence lifetime curves of Cy7/PYB_1@ PVA doped films with different Cy7 contents at 598 nm. The fluorescence lifetime curves of Cy7/PYB_1@ PVA doped films with different Cy7 contents (e) measured at 400 nm and (f) measured at 456 nm. The content of Cy7 is mol.% to PYB amount and the content of PYB doped in PVA film is 1.0 wt% to PVA.

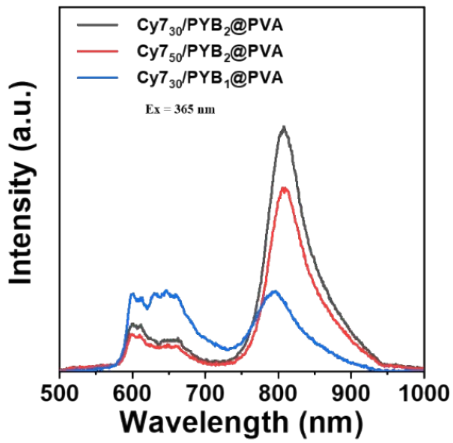


Figure S15. The delayed PL spectra (delayed 8 ms) of Cy7/PYB_1@ PVA and Cy7/PYB_2@ PVA doped films for comparison.

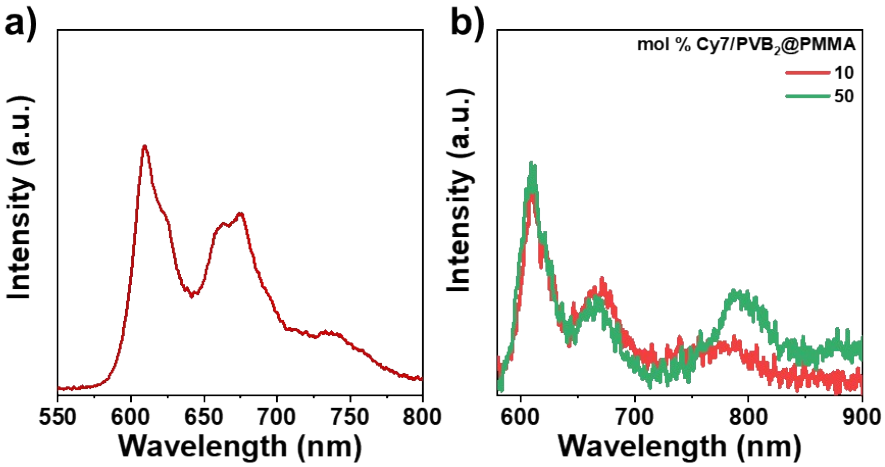


Figure S16. The delayed PL spectra (delayed 8 ms) of (a) PYB_2@PMMA and (b) Cy7/PYB_2@PMMA with different Cy7 contents. (*E*x = 365 nm).

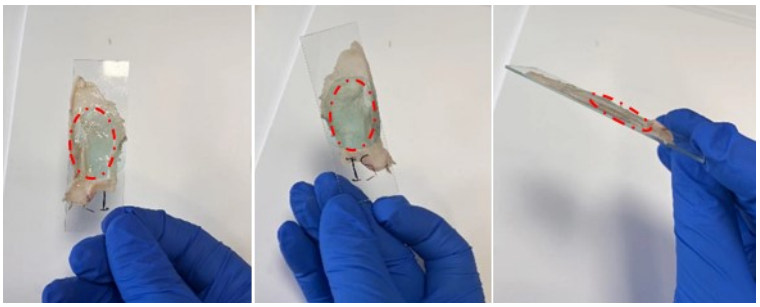
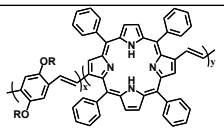
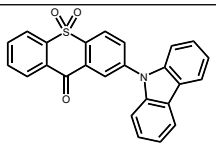
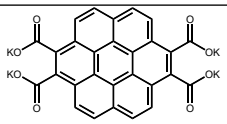
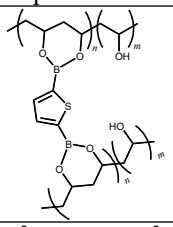
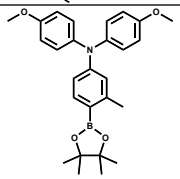
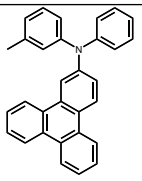
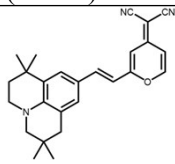
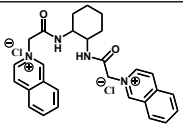
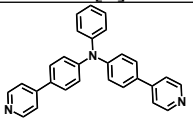
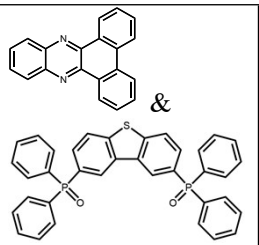
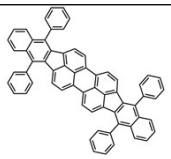


Figure S17. Photos of a $Cy7_{30}$/PYB_2@PVA film that was put on a glass, covered with a piece of pork (left: captured from top to bottom, middle: captured from bottom to top, right: captured from the side).

Table S1. Delayed luminescence properties of organic red or NIR materials through triplet-singlet energy transfer strategy

No.	Donor	Acceptor	Luminescence of donor [a]	Luminescence of acceptor [b]	Ref.
1		traphenylporphyrin (TPP)	λ_{em} = 580 nm Lifetime was not given.	λ_{em} = ~ 720 nm Lifetime was not given.	Adv. Healthcare Mater. 2018, 7, 1800329
2		Nile Red (NR)	τ = 0.24 s λ_{em} = 511, 559, and 605 nm	τ = 0.1 s λ_{em} = 640 nm	ACS Appl. Mater. Interfaces 2020, 12, 5073
3	 doped into PVA	Sulpharhodamine G (SRG)	τ = 2.46 s λ_{em} = 572 nm	λ_{em} = 610 nm Lifetime was not given.	Angew. Chem. Int. Ed. 2020, 59, 9393
4		Rhodamine (RB)	τ = 256 ms λ_{em} = 475 nm	λ_{em} = 590 nm Lifetime was not given.	Bull. Chem. Soc. Jpn. 2021, 94, 1204
5		silicon 2,3-naphthalocyanine bis(trihexylsilyloxide) (NCBS)	τ = 25 ms λ_{em} = 530 nm	τ = 9.1 us, λ_{em} = ~780 nm	Adv. Mater. 2020, 32, 2006752
6	 doped into estradiol		τ = 0.60 s λ_{em} = 485 nm	τ = 0.42 s λ_{em} = ~ 640 nm	Chem. Mater. 2022, 34, 1627
7	polyacrylamide & polyhexamethylene biguanidine hydrochloride mixed sample	Bromophenol blue (BPB)	τ = 291 ms (513 nm) with broad emission band from 400 to 700 nm	τ = 0.48 ms λ_{em} = 800 nm	Adv. Optical Mater. 2023, 11, 2202482
8	 mixture with cucurbit[8]uril	Intermediate acceptor: NiB; Final acceptor: IR780	τ = 0.365 ms λ_{em} = 555 nm	τ =79.8 μs λ_{em} = 825 nm (IR780)	Adv. Sci. 2022, 9, 2201523
9	 doped in PVA	Rhodamine (RB)	τ = 798.4 ms λ_{em} = 506 nm	τ = 201.6 ms λ_{em} = ~590 nm	Adv. Mater. 2023, 35, 2301874

10			τ = 0.18 s λ_{em} = 560 nm	τ = 0.34 s λ_{em} = ~ 600 nm	Adv. Funct. Mater. 2023, 33, 2211604

[a] luminescence properties including lifetime and maximum emission wavelength of host before energy transfer.

[b] luminescence properties including lifetime and maximum emission wavelength of acceptor through energy transfer.

Table S2. Phosphorescence lifetime of PYB/PVA films with different contents of PYB at 598 nm.

PYB content/wt%	0.5	1.0	2.0	6.0
τ_{PYB-1}/s[a]	0.09	1.04	1.03	0.95
(%)	(19.91)	(12.64)	(12.40)	(16.72)
τ_{PYB-2}/s[b]	0.34	0.35	0.35	0.33
(%)	(80.09)	(87.36)	(87.60)	(83.28)
τ_{PYB}/s[c]	0.29	0.44	0.43	0.43

[a, b] Lifetime of different components. [c] Average phosphorescence lifetime.

Table S3. Phosphorescence lifetime of PYB at 598 nm of Cy7/PYB_2@PVA films with different contents of Cy7 dye.

Cy7 content/mol%	0	10	30	50
τ_{PYB-1}/s[a]	1.03	0.52	0.079	0.44
(%)	(12.40)	(31.79)	(21.40)	(34.91)
τ_{PYB-2}/s[b]	0.35	0.18	0.24	0.15
(%)	(87.60)	(68.21)	(79.60)	(65.09)
τ_{PYB}/s[c]	0.43	0.29	0.21	0.25
η_{ET} / %[d]		32.6	51.2	41.9

[a, b] Lifetime of different components. [c] Average phosphorescence lifetime. [d] FRET efficiency, η_{ET} = ($\tau_{PYB0} - \tau_{PYB}$)/τ_{PYB0}, where τ_{PYB0} is the original lifetime (0.43 s) of PYB_2@PVA film, and τ_{PYB} is the new lifetime of Cy7/PYB_2@PVA films with different contents of Cy7 dye. Regarding to $Cy7_{30}$/PYB_2@PVA, T-S FRET efficiency was calculated as: η_{ET} = ($\tau_{PYB0} - \tau_{PYB}$)/τ_{PYB0} = (0.43-0.21)/0.43 = 0.512 = 51.2%.

Table S4. Fluorescence lifetime of PYB at 400 nm and 456 nm of Cy7/PYB_2@PVA films with different contents of Cy7 dye.

Cy7 content	400 nm				456 nm			
/mol%	0	10	30	50	0	10	30	50
τ_{PYB-1}/ns[a]	33.11	18.39	6.95	4.37	39.6	19.35	8.13	3.68
(%)	(33.49)	(37.95)	(42.51)	(36.08)	(41.42)	(39.50)	(48.63)	(55.94)
τ_{PYB-2}/ns[a]	90.00	0.52	29.05	13.84	112.15	71.47	44.72	17.70
(%)	(58.33)	(22.25)	(36.88)	(25.51)	(24.41)	(21.12)	(14.70)	(23.82)
τ_{PYB-3}/ns[a]	0.52	63.5	0.028	0.027	6.29	4.05	1.51	0.72
(%)	(8.19)	(39.79)	(20.61)	(38.41)	(34.17)	(39.38)	(36.67)	(20.24)
τ_{PYB}/ns[b]	53.70	32.36	13.67	5.12	45.93	24.33	11.08	6.42
η_{ET} / %[c]		39.7	74.5	90.5		47.0	75.9	86.0

[a] Lifetime of different components. [b] Average fluorescence lifetime. [c] FRET efficiency, $\eta_{ET} = (\tau_{PYB0} - \tau_{PYB})/\tau_{PYB0}$, where τ_{PYB0} is the original lifetime (53.70 ns for 400 nm and 45.93 ns for 456 nm) of PYB_2@PVA film, and τ_{PYB} is the new lifetime of Cy7/PYB_2@PVA films with different contents of Cy7 dye. Regarding to $Cy7_{30}$/PYB_2@PVA, S-S FRET efficiency was calculated as: $\eta_{ET400} = (\tau_{PYB0} - \tau_{PYB})/\tau_{PYB0}$ = (53.70-13.67)/53.70 = 0.745 = 74.5%; $\eta_{ET456} = (\tau_{PYB0} - \tau_{PYB})/\tau_{PYB0}$ = (45.93-11.08)/45.93 = 0.759 = 75.9%.

Table S5. Phosphorescence lifetime of PYB at 598 nm of Cy7/PYB_1@PVA films with different contents of Cy7 dye.

Cy7 content/mol%	0	10	30	50	70	100
τ_{PYB-1}/s[a]	1.04	0.11	0.093	0.053	0.25	0.062
(%)	(12.64)	(32.30)	(20.46)	(13.13)	(66.42)	(18.57)
τ_{PYB-2}/s[b]	0.35	0.33	0.26	0.21	0.092	0.22
(%)	(87.36)	(67.70)	(79.54)	(86.87)	(33.58)	(81.43)
τ_{PYB}/s[c]	0.44	0.26	0.23	0.19	0.20	0.19
η_{ET} / %[d]		40.9	47.7	56.8	54.5	56.8

[a, b] Lifetime of different components. [c] Average phosphorescence lifetime. [d] FRET efficiency, $\eta_{ET} = (\tau_{PYB0} - \tau_{PYB})/\tau_{PYB0}$, where τ_{PYB0} is the original lifetime (0.43 s) of PYB_1@PVA film, and τ_{PYB} is the new lifetime of Cy7/PYB_1@PVA films with different contents of Cy7 dye. T-S FRET efficiency was calculated as: $\eta_{ET} = (\tau_{PYB0} - \tau_{PYB})/\tau_{PYB0}$ = (0.44-0.23)/0.44 = 0.47.7 = 47.7%.

Table S6. Fluorescence lifetime of PYB at 400 nm of Cy7/PYB_1@PVA films with different contents of Cy7 dye.

Cy7 content/mol%	0	10	30	50	70	100
τ_{PYB-1}/ns[a]	47.07	26.39	18.27	11.93	8.39	11.46
(%)	(34.32)	(34.67)	(33.47)	(39.63)	(45.91)	(17.01)
τ_{PYB-2}/ns[a]	109.54	82.77	0.52	59.62	39.89	45.83
(%)	(58.61)	(53.05)	(36.91)	(14.63)	(48.40)	(9.3)
τ_{PYB-3}/ns[a]	0.54	0.56	65.83	2.14	0.025	0.050
(%)	(7.07)	(2.28)	(29.62)	(45.73)	(5.69)	(73.69)
τ_{PYB}/ns[b]	80.39	53.07	25.80	14.43	23.16	6.25
η_{ET} / %[c]		34.0	67.9	82.1	71.2	92.2

[a] Lifetime of different components. [b] Average fluorescence lifetime. [c] FRET efficiency, $\eta_{ET} = (\tau_{PYB0} - \tau_{PYB})/\tau_{PYB0}$, where τ_{PYB0} is the original lifetime (80.39 ns) of PYB_1@PVA film, and τ_{PYB} is the new lifetime of Cy7/PYB_1@PVA films with different contents of Cy7 dye.

Table S7. Fluorescence lifetime of PYB at 456 nm of Cy7/PYB_1@PVA films with different contents of Cy7 dye.

Cy7 content/mol%	0	10	30	50	70	100
τ_{PYB-1}/ns[a]	29.41	27.03	16.81	11.93	6.69	0.90
(%)	(31.57)	(37.22)	(41.59)	(39.63)	(45.77)	(31.14)
τ_{PYB-2}/ns[a]	100.9	88.37	68.21	59.62	40.16	5.36
(%)	(46.84)	(24.95)	(21.94)	(14.63)	(20.28)	(47.73)
τ_{PYB-3}/ns[a]	4.53	4.63	3.15	2.15	1.22	3.51
(%)	(21.59)	(37.83)	(36.47)	(45.73)	(33.94)	(21.13)
τ_{PYB}/ns[b]	57.52	33.86	23.11	14.43	11.62	3.58
η_{ET} / %[c]		41.1	59.8	74.9	79.8	93.8%

[a] Lifetime of different components. [b] Average fluorescence lifetime. [c] FRET efficiency, $\eta_{ET} = (\tau_{PYB0} - \tau_{PYB})/\tau_{PYB0}$, where τ_{PYB0} is the original lifetime (55.52 ns) of PYB_1@PVA film, and τ_{PYB} is the new lifetime of Cy7/PYB_1@PVA films with different contents of Cy7 dye.

4. Luminescence quantum yield (QY) calculations

Before the calculation, it is important to describe the ratio of delayed luminescence in the total luminescence, as only the total emission calculated in general luminescence QY measurements by using a calibrated integrating sphere. There are some references discussed about this, based on the analysis of time-resolved intensity decay curves.[1-4] The general method is described as follow:

The delayed luminescence/afterglow QY ($\Phi_{delayed}$) were determined by using total QY (Φ_{total}) and the ratio between prompt (r_1) and delayed (r_2) components which were calculated from emission lifetime (τ_1, τ_2) and fitting parameter (A_1, A_2) as follows:

$$I(t) = A_1 e^{-\frac{t}{\tau_1}} + A_2 e^{-\frac{t}{\tau_2}} \quad (1)$$

$$r_1 = \frac{A_1\tau_1}{A_1\tau_1 + A_2\tau_2} \quad (2)$$

$$r_2 = \frac{A_2\tau_2}{A_1\tau_1 + A_2\tau_2} \quad (3)$$

The Φ_{prompt} and $\Phi_{delayed}$ were calculated using intensity ratio (r_1, r_2) and Φ_{total} as follows:

$$\Phi_{total} = \Phi_{prompt} + \Phi_{delayed} \quad (4)$$

$$\Phi_{prompt} = r_1\Phi_{total} \quad (5)$$

$$\Phi_{delayed} = r_2\Phi_{total} \quad (6)$$

There are huge difference on the lifetimes of prompt (fluorescence in nanoseconds, Table S4) and delayed emission (RTP in milliseconds, Table S2) components of PYB@PVA systems. So, it is difficult to obtain the exact fitting parameters (A_1, A_2) directly from the time-resolved intensity decay curves. Thus the beginning intensity of prompt and delayed components in the intensity decay curves (as shown in Figure S4c) were used as fitting parameters. And the lifetime of prompt (τ_1) and delayed (τ_2) emission were obtained from the intensity decay curves measured in different time range, respectively (Figure S8d and Table S4 for prompt component, while Figure 4c and Table S3 for the delayed one).

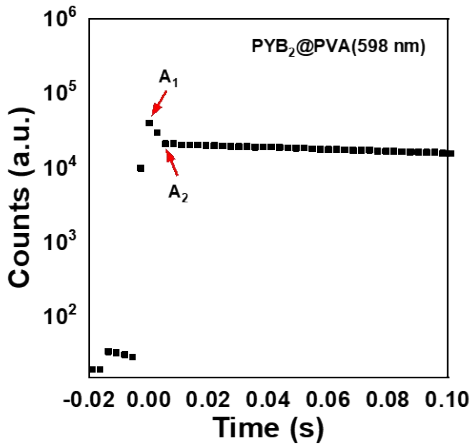


Figure S18. The identification of different parameters for efficiency calculation from the time-resolved intensity decay curves: (a) the fitting parameters (A_1 and A_2) from the decay curve in millisecond regime.

The corresponding parameters and results for the RTP efficiency calculation were list as follows:

Table S8. The fitted lifetimes, parameters and corresponding calculated RTP efficiencies for PYB_2@PVA and $Cy7_{30}$/PYB_2@PVA (measured at 598 nm).

Samples	A_1	τ_1 (ns)	A_2	τ_2 (s)	Φ_{total} / %	$\Phi_{delayed}$ / %
PYB_2@PVA	39219	45.93	20812	0.43	2.0	2.0
$Cy7_{30}$/PYB_2@PVA	40462	11.08	4824	0.21	0.43	0.43

The Φ_{total} were measured from 600 to 700 nm by using a calibrated integrating sphere excited at 365 nm. The $\Phi_{delayed}$ were the RTP efficiency of PYB.

In the system of $Cy7_{30}$/PYB_2@PVA, the RTP efficiency ($\Phi_{delayed}$) of Cy7 was calculated from the ratio of Cy7 peak (750-900 nm) to PYB excimer peak (600-700 nm) in the delayed spectra of the doped PVA film (Figure 4b). As the Cy7 peak' area is about 4.8 folds of the PYB peak's area, the $\Phi_{delayed}$ of Cy7 thus is calculated to be 2.1% (0.43% × 4.8). On the other hand, PYB peak possessed a longer RTP lifetime (0.21 s, Figure 4c and Table S2), which is about 1.31 folds of lifetime (0.16 s, Figure 4d) for Cy7 at the same PVA film. Therefore, the final $\Phi_{delayed}$ of Cy7 is calculated to be 1.6% (2.1% / 1.31).

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