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Pure white-light and color-tuning of Eu^{3+}-Gd^{3+}-containing metallopolymer

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Experimental section

High performance liquid chromatography (HPLC)-grade tetrahydrofuran (THF) or acetonitrile (MeCN) was purchased from Fisher Scientific and purified over solvent columns. Other solvents were used as received from Sigma Aldrich and stored over 3 Å activated molecule sieves. Azobis(isobutyronitrile) (AIBN) was purified by recrystallization twice from absolute MeOH prior to use. Tetrakis(triphenylphosphine)palladium ($Pd(PPh_3)_4$),[1] the series of tris-*β*-diketonate dihydrate Ln^{3+}-complex precursors $[Ln(DBM)_3(H_2O)_2]$[2] (DBM = dibenzoylmethane; Ln = La, Eu, Tb or Gd) were synthesized according well-established procedures from the literatures, respectively. Other chemicals including N-vinyl-carbazole (NVK) were commercial products of reagent grade and were used without further purification. All manipulations of air and water sensitive compounds were carried out under dry N_2 using the standard Schlenk line techniques. Elemental analyses were performed on a Perkin-Elmer 240C elemental analyzer. Infrared spectra were recorded on a Nicolet Nagna-IR 550 spectrophotometer in the region 4000-400 cm^{-1} using KBr pellets. ^1H NMR or ^{13}C NMR spectra were recorded on a JEOL EX 400 or JEOL EX 100 spectrometer with $SiMe_4$ as internal standard in $CDCl_3$ and/or DMSO-δ_6 at room temperature, respectively. ESI-MS was performed on a Finnigan LCQ^{DECA} XP HPLC-MS$_n$ mass spectrometer with a mass to charge (*m/z*) range of 4000 using a standard electrospray ion source and $CHCl_3$ or MeCN as solvent. Electronic absorption spectra for a sample in solution at the UV/Visible region and diffuse reflection (DR) spectra for a solid sample in film form at room temperature were recorded with a Shimadzu UV-3159 UV-Vis-NIR spectrophotometer. Emission and excitation spectra were collected at room temperature or 77 K by a combined fluorescence lifetime and steady-state spectrometer (FLS-980, Edinburgh) with a 450 W Xe lamp. Excited-state decay times were obtained at room temperature or 77 K by the same spectrometer but with a μF900 Xe lamp. The luminescent absolute overall quantum yield (Φ_{em} or $\Phi_{Ln}{}^{L}$) for a sample in solution or solid film at room temperature was determined at by the same spectrometer using a 450 W Xe lamp and an integrating sphere (150 nm, $BaSO_4$ coating), and calculated by the following equation $\Phi_{overall} = A_H/(R_{ST} - R_H)$ developed by Mello et al[3], in which A_H is the area under the emission spectrum of the sample, and R_{ST} and R_H are the diffuse reflectance of the reflecting standard and the sample, respectively. The estimated error for the quantum yield is ±10%. The Commission International de I'Eclairage (CIE) coordinate, the color rendering index (CRI) and the corrected color temperature (CCT) of each sample were calculated on the basis of the international CIE standards. Gel permeation chromatography (GPC) analyses of the polymers were performed using a Waters 1525 binary pump coupled to a Waters 2414 refractive index detector with HPLC THF as the eluant on American Polymer Standard 10 μm particle size, linear mixed bed packing columns. The GPC was calibrated using polystyrene standards. X-ray photoelectron spectroscopy (XPS) was carried out on a PHI 5700 XPS system equipped with a dual Mg X-ray source and monochromatic Al X-ray source complete with depth profile and angle-resolved capabilities. Powder X-ray diffraction (PXRD) patterns were recorded on a D/Max-IIIA diffractometer with graphite-monochromatized Cu Kα radiation (λ = 1.5418 Å). Thermogravimetric (TG) analyses were carried out on a NETZSCH TG 209 instrument under flowing nitrogen by heating the samples from 25 to 600 °C.

Synthesis of 5-bromo-2,2'-bipyridine (5-Br-2,2'-bpy)

The synthesis of 5-Br-2,2'-bpy was carried out according to an improved Stille coupling reaction procedure from the literature.[4] To a degassed round-bottom flask equipped with a reflux condenser and a magnetic stirrer under an argon atmosphere, 2.84 g (12 mmol) of 2,5-dibromopyridine and 0.28 g (0.24 mmol) of $Pd(PPh_3)_4$ (0) were added, respectively. The system was degassed again, then a solution of 2-(tributylstannyl)pyridine (4.43 g, 12 mmol) in absolute xylene (30 mL) was added, and the resultant mixture was reacted at 120 °C for 12 h under an argon atmosphere. After cooling to room temperature, 40 mL of aqueous NaOH (2 M) was added. The reaction mixture was extracted with dry toluene (3 × 40 mL), and the organic layer was dried over anhydrous Na_2SO_4. After evaporation to dryness, the residue was purified by silica column chromatography using a solvent of mixture of n-hexane and ethyl acetate (5/1, v/v) as the eluent to give a white microcrystalline product. Yield: 1.89 g (67%). Elemental analysis (%): Calcd for $C_{10}H_7N_2Br$: C, 51.09; H, 3.00. Found: C, 51.02; H, 3.07. FT-IR (KBr, cm^{-1}): 3090 (w), 3044 (w), 3002 (w), 2924 (w), 2854 (w), 1986 (w), 1933 (w), 1864 (w), 1612 (w), 1564 (s), 1549 (s), 1451 (vs), 1431 (s), 1364 (s), 1313 (w), 1236 (w), 1144 (w), 1121 (w), 1088 (s), 1062 (m), 1038 (w), 1001 (s), 960 (w), 933 (m), 899 (w), 841 (m), 788 (s), 770 (m), 737 (s), 631 (m), 548 (w), 471 (m), 432 (w), 423 (w). ^{1}H NMR (400 MHz, DMSO-δ_6): δ (ppm) 8.81 (d, 1H, -Py), 8.70 (d, 1H, -Py), 8.35 (d, 2H, -Py), 8.20 (t, 1H, -Py), 7.97 (m, 1H, -Py), 7.49 (m, 1H, -Py). ^{13}C NMR (100 MHz, DMSO-δ_6): δ (ppm) 155.15, 154.61, 150.18, 149.25, 139.49, 137.01, 124.07, 122.33, 121.15, 120.97.

Synthesis of 2-(pyridin-2-yl)-5-(4-vinylphenyl)pyridine (4-vp-2,2'-bpy)

To a degassed three-necked round-bottom flask equipped with a reflux condenser, an addition funnel, and a magnetic stirrer under an argon atmosphere, 1.40 g (6 mmol) of solid 5-Br-2,2'-bpy from the previous reaction, 0.88 g (6 mmol) of 4-vinylphenylboronic acid, and 0.69 g (0.6 mmol) of $Pd(PPh_3)_4$ were added, respectively. The system was degassed again and the mixture of 50 mL of distilled THF and 6 mL of Na_2CO_3 (2 M) degassed aqueous solution was added. The resulting mixture was further refluxed for 48 h under an argon atmosphere. After cooling to room temperature, the reaction mixture was extracted using toluene (3 × 30 mL) and dried over Na_2SO_4. The solvent of the organic layer was removed under vacuum, and the resulting yellow solid was poured into n-hexane (50 mL). After being stirred at room temperature for 2 h, the mixture was filtered, and the filtrate was evaporated. The residue was further purified by silica column chromatography using a solvent of mixture of absolute CH_3OH and CH_2Cl_2 (v/v, 1/20) as the eluent to give a white powder product. Yield 1.33 g (86%). Elemental analysis (%): Calcd for $C_{18}H_{14}N_2$: C, 83.69; H, 5.46. Found: C, 83.58; H, 5.54. FT-IR (KBr, cm^{-1}): 3460 (w), 3150 (w), 1960 (w), 1910 (w), 1810 (w), 1630 (s), 1590 (s), 1510 (m), 1460 (s), 1440 (vs), 1410 (m), 1370 (m), 1270 (w), 1240 (w), 1170 (s), 1120 (s), 1090 (w), 1070 (w), 1040 (w), 1030 (w), 989 (s), 930 (w), 903 (m), 837 (s), 796 (s), 752 (s), 723 (s), 694 (s), 648 (w), 633 (w), 617 (w), 588 (w), 540 (s), 517 (w), 447 (w). ^{1}H NMR (400 MHz, DMSO-δ_6-$CDCl_3$ (v/v = 10:1)): δ (ppm) 9.06 (s, 1H, -Py), 8.72 (d, 1H, -Py), 8.46 (m, 2H, -Py), 8.28 (d, 1H, -Py), 7.99 (m, 1H, -Py), 7.83 (d, 2H, -Ph), 7.64 (d, 2H, -Ph), 7.48 (t, 1H, -Py), 6.82 (m, 1H, -CH=C), 5.95 (d, 1H, -C=CH_2), 5.34 (d, 1H, -C=CH_2). ^{13}C NMR (100 MHz, DMSO-δ_6): δ (ppm) 155.86, 154.92, 149.27, 147.48, 137.55, 137.01, 136.86, 136.17, 136.04, 135.02, 127.18, 126.98, 123.75, 121.10, 121.04, 114.64.

Synthesis of complex monomers [Ln(DBM)$_3$(4-vp-2,2'-bpy)] (Ln = La (1), Eu (2), Tb (3) or Gd (4))

To a stirred suspension of [Ln(DBM)$_3$(H_2O)$_2$] (0.3 mmol; Ln = La, 0.253 g; Ln = Eu, 0.257 g; Ln = Tb, 0.259 g; Ln = Gd, 0.259 g) in absolute MeCN (10 mL), solid 4-vp-2,2'-bpy (0.078 g, 0.3 mmol) was added, and the reaction mixture was refluxed for 4 h under an argon atmosphere. After cooling to room temperature, each of resultant clear yellow solution was filtered and left to stand at room temperature for several days to give pale yellow crystalline products, respectively. For **1**: Yield: 0.230 g (72%). Elemental analysis (%): Calcd for $C_{63}H_{47}N_2O_6La$: C, 70.92; H, 4.44; N, 2.63. Found: C, 70.86; H, 4.53; N, 2.59. FT-IR (KBr, cm^{-1}): 3055 (w), 3026 (w), 1593 (m), 1552 (s), 1514 (s), 1477 (m), 1460 (s), 1437 (w), 1410 (vs), 1313 (m), 1284 (w), 1221 (w), 1179 (w), 1159 (w), 1065 (w), 1027 (w), 941 (w), 835 (w), 785 (w), 746 (w), 723 (s), 688 (m), 610 (m). ^{1}H NMR (400 MHz, DMSO-δ_6-$CDCl_3$ (v/v = 10:1)): δ (ppm) 9.07 (d, 1H, -Py), 8.73 (d, 1H, -Py), 8.42 (m, 2H, -Py), 8.29 (m, 1H, -Py), 8.03 (m, 12H, -Ph), 8.00 (m, 1H, -Py), 7.83 (d, 2H, -Ph), 7.64 (d, 2H, -Ph), 7.49 (t, 1H, -Py), 7.44 (t, 6H, -Ph), 7.34 (m, 12H, -Ph), 6.84 (m, 1H, -CH=C), 6.75 (s, 3H, -CH), 5.96 (d, 1H, -C=CH$_2$), 5.35 (d, 1H, -C=CH$_2$). ^{13}C NMR (100 MHz, DMSO-δ_6-CDCl$_3$ (v/v = 10:1)): δ(ppm) 182.84, 155.56, 154.73, 149.38, 147.39, 141.32, 137.46, 137.13, 136.62, 136.24, 135.72, 134.92, 130.36, 128.09, 127.46, 127.18, 127.11, 124.03, 120.92, 114.87, 93.98, 78.89.

ESI-MS (MeCN, *m*/*z*): 827.26 (100%), [M-H]$^+$. For **2**: Yield: 0.253 g (78%). Elemental analysis (%): Calcd for $C_{63}H_{47}N_2O_6Eu$: C, 70.06; H, 4.39; N, 2.59. Found: C, 70.02; H, 4.55; N, 2.52. FT-IR (KBr, cm^{-1}): 3058 (w), 3025 (w), 1597 (m), 1548 (s), 1514 (s), 1478 (m), 1458 (s), 1437 (w), 1411 (vs), 1310 (m), 1284 (w), 1220 (w), 1179 (w), 1157 (w), 1064 (w), 1025 (w), 944 (w), 835 (w), 784 (w), 746 (w), 721 (s), 688 (m), 609 (m). ESI-MS (MeCN, *m*/*z*): 840.82 (100%), [M-H]$^+$. For **3**: Yield: 0.248 g (76%). Elemental analysis (%): Calcd for $C_{63}H_{47}N_2O_6Tb$: C, 69.61; H, 4.36; N, 2.58. Found: C, 69.53; H, 4.47; N, 2.52. FT-IR (KBr, cm^{-1}): 3059 (w), 3026 (w), 1594 (m), 1550 (s), 1515 (s), 1477 (m), 1458 (s), 1437 (w), 1412 (vs), 1310 (m), 1284 (w), 1220 (w), 1178 (w), 1157 (w), 1067 (w), 1024 (w), 941 (w), 836 (w), 784 (w), 744 (w), 720 (s), 688 (m), 608 (m). ESI-MS (MeCN, *m*/*z*): 847.78 (100%), [M-H]$^+$. For **4**: Yield: 0.257 g (79%). Elemental analysis (%): Calcd for $C_{63}H_{47}N_2O_6Gd$: C, 69.72; H, 4.36; N, 2.58. Found: C, 69.66; H, 4.47; N, 2.53. FT-IR (KBr, cm^{-1}): 3059 (w), 3023 (w), 1594 (m), 1550 (s), 1518 (s), 1476 (m), 1458 (s), 1437 (w), 1415 (vs), 1310 (m), 1284 (w), 1223 (w), 1177 (w), 1158 (w), 1067 (w), 1026 (w), 943 (w), 836 (w), 785 (w), 746 (w), 720 (s), 670 (m), 608 (m). ESI-MS (MeCN, *m*/*z*): 846.10 (100%), [M-H]$^+$.

X-ray crystallography

Single crystals of complex **3**·1.75H$_2$O·MeCN ([Ln(DBM)$_3$(4-vp-2,2'-bpy)]·1.75H$_2$O·MeCN) of suitable dimensions were mounted onto thin glass fibers. All the intensity data were collected on a Bruker SMART CCD diffractometer (Mo-Kα radiation and $\lambda = 0.71073$ Å) in Φ and ω scan modes. Structures were solved by Direct methods followed by difference Fourier syntheses, and then refined by full-matrix least-squares techniques against F^2 using SHELXTL.[5] All other non-hydrogen atoms were refined with anisotropic thermal parameters. Absorption corrections were applied using SADABS.[6] All hydrogen atoms were placed in calculated positions and refined isotropically using a riding model. Crystallographic data and selected atomic distances and bond angles are presented in Tables 1-2s, respectively. CCDC reference number 1439094 for **3**$\cdot 1.75H_2O \cdot$MeCN.

Synthesis of PVK-supported metallopolymer Poly(NVK-*co*-1) (200:1)

To a stirred mixture of NVK (1.391 g, 7.2 mmol) and the complex monomer **1** with a stipulated feed molar ratio of 200:1 in dry 1,2-dichlorobenzene (15 mL), AIBN initiator (17.8 mg, 1.5 mol% of NVK) was added, and the resultant homogeneous solution was purged with N_2 for 10 min and sealed under a reduced N_2 atmosphere. The homogeneous solution was heated to 80 °C with continuous stirring for 48 h. After cooling to room temperature, the obtained viscous mixtures was diluted with dry 1,2-dichlorobenzene (10 mL) and precipitated with absolute diethyl ether (100 mL) three times. The resultant solid product was collected by filtration and dried at 45 °C under vacuum to constant weight. For **Poly(NVK-*co*-1)** (200:1): Yield: 90%. FT-IR (KBr, cm^{-1}): 3050 (w), 2941 (w), 1723 (w), 1624 (w), 1598 (w), 1547 (w), 1483 (w), 1452 (m), 1330 (m), 1219 (s), 1157 (w), 1124 (w), 1027 (w), 928 (w), 773 (vs), 746 (s), 722 (s), 682 (w), 651 (w), 634 (w). ^1H NMR (400 MHz, DMSO-δ_6-$CDCl_3$ (v/v = 10:1)): δ

(ppm) 9.08 (b, 1H, -Py), 8.74 (b, 1H, -Py), 8.42 (b, 2H, -Py), 8.25-3.95 (b, 1440H + 40H), 3.95-2.15 (b, 180H + 1H), 2.15-0.50 (b, 360H + 2H). ^{13}C NMR (100 MHz, DMSO-δ_6-$CDCl_3$ (v/v = 10:1)): δ (ppm) 182.81, 155.03, 154.34, 149.59, 147.09, 141.19, 140.09 (b), 137.67 (b), 136.58, 136.33, 135.80, 134.76, 130.11, 129.41, 129.10, 128.83, 128.43, 127.77, 125.49 (b), 123.72 (b), 121.98 (b), 120.44 (b), 119.03 (b), 110.7 (b), 108.26 (b), 93.96, 78.83, 49.82 (b), 38.77, 35.84 (b), 25.69. GPC result (HPLC THF): M_n = 31042 g/mol and PDI = M_w/M_n = 1.15.

Synthesis of PVK-supported metallopolymers Poly(2-*co*-NVK-*co*-4) (200:1; [2/4] = 1:5 or 1:7)

The PVK-supported while Eu^{3+}-Gd^{3+}-containing metallopolymers **Poly(2-*co*-NVK-*co*-4)** with a stipulated feed molar ratio 200:1 ([**2**/**4**] = 1:5 or 1:7) were prepared in the same way as **Poly(NVK-*co*-1)** (200:1) except that two complex monomers **2** and **4** with different Ln^{3+}-mixed molar ratios ([**2**/**4**] = 1:5 or 1:7) were used instead of complex monomer **1**. For **Poly(2-*co*-NVK-*co*-4)**: Yield: 89% ([**2**/**4** = 1:5); 91% ([**2**/**4** = 1:7]). FT-IR (KBr, cm^{-1}): 3052 (w), 2940 (w), 1723 (w), 1627 (w), 1599 (w), 1543 (w), 1483 (w), 1451 (m), 1330 (m), 1220 (s), 1158 (w), 1124 (w), 1029 (w), 929 (w), 773 (vs), 745 (s), 724 (s), 682 (w), 650 (w), 631 (w). GPC results (HPLC THF): M_n = 30898 g/mol and PDI = M_w/M_n = 1.12 for **Poly(2-*co*-NVK-*co*-4)** (200:1; [**2**/**4**] = 1:5); M_n = 31476 g/mol and PDI = M_w/M_n = 1.21 for **Poly(2-*co*-NVK-*co*-4)** (200:1; [**2**/**4**] = 1:7). XPS results: Found atomic ratio (1:5.03) of Eu:Gd for **Poly(2-*co*-NVK-*co*-4)** (200:1; [**2**/**4**] = 1:5); Found atomic ratio (1:6.85) of Eu:Gd for **Poly(2-*co*-NVK-*co*-4)** (200:1; [**2**/**4**] = 1:7).

Preparation of the films from PVK-supported Ln^{3+}-containing metallopolymers

Each of the transparent films was easily prepared by first dissolving the PVK-supported Ln^{3+}-containing metallopolymer sample in tolune (10 mL) at a concentration of 5 mg/mL, then spin-coating at 3000 rpm on a clean quartz slide, and finally drying in air. The almost similar film thickness of 95 nm due to the same concentration was measured by ellipsometry by collecting data every 5° from 65° to 75° and was fitted using a Cauchy film on gold model.

References

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Table S1 Crystal data and structure refinement for complex **3**·$1.75H_2O$·MeCN

Compound	**3**·$1.75H_2O$·MeCN
Empirical formula	$C_{65}H_{53.5}N_3O_{7.75}Tb$
Formula weight	1159.53
Crystal size/mm	0.27 × 0.25 × 0.22
T/K	293(2)
λ/Å	0.71073
Crystal system	Monoclinic
Space group	$P2/c$
a/Å	26.8876(3)
b/Å	16.8644(2)
c/Å	25.0129(3)
α/°	90
β/°	95.2480(10)
γ/°	90
V/Å^3	11294.4(2)
Z	8
ρ/g·cm^{-3}	1.364
μ/mm^{-1}	1.310
F(000)	4732
Data/restraints/parameters	18774/79/1352
Quality-of-fit indicator	1.015
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0781$
	$wR_2 = 0.2335$
R indices (all data)	$R_1 = 0.1030$
	$wR_2 = 0.2577$

Table S2 Selected bond lengths (Å) and bond angles (°) for complex **3**·1.75H_2O·MeCN

3·1.75H_2O·MeCN			
Tb(1)-N(1)	2.605(7)	Tb(2)-N(3)	2.570(7)
Tb(1)-N(2)	2.601(7)	Tb(2)-N(4)	2.591(6)
Tb(1)-O(1)	2.324(6)	Tb(2)-O(7)	2.336(6)
Tb(1)-O(2)	2.311(6)	Tb(2)-O(8)	2.327(6)
Tb(1)-O(3)	2.330(5)	Tb(2)-O(9)	2.333(6)
Tb(1)-O(4)	2.310(6)	Tb(2)-O(10)	2.308(6)
Tb(1)-O(5)	2.304(6)	Tb(2)-O(11)	2.338(6)
Tb(1)-O(6)	2.349(5)	Tb(2)-O(12)	2.315(6)
C(62)-C(63)	1.301(16)	C(125)-C126)	1.318(17)
N(1)-Tb(1)-N(2)	62.2(2)	N(3)-Tb(2)-N(4)	62.5(2)
O(1)-Tb(1)-O(2)	71.7(2)	O(7)-Tb(2)-O(8)	72.3(2)
O(3)-Tb(1)-O(4)	71.42(19)	O(9)-Tb(2)-O(10)	71.5(2)
O(5)-Tb(1)-O(6)	70.8(2)	O(11)-Tb(2)-O(12)	72.7(2)

Figure S1 ^{1}H NMR spectra of **1**, **PVK** and **Poly(NVK-*co*-1)** (200:1) in DMSO-δ_6-CDCl$_3$ (v/v = 10:1) at RT.

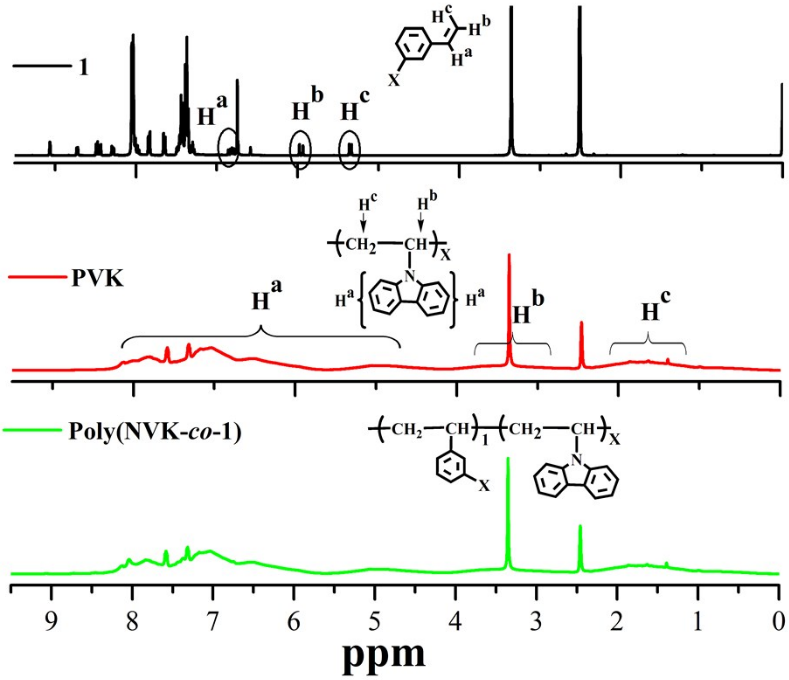

Figure S2 UV-visible absorption spectra of DBM, 4-vp-2,2'-bpy and complexes **2-4** in MeCN solution at 1 × 10^{-5} M at RT.

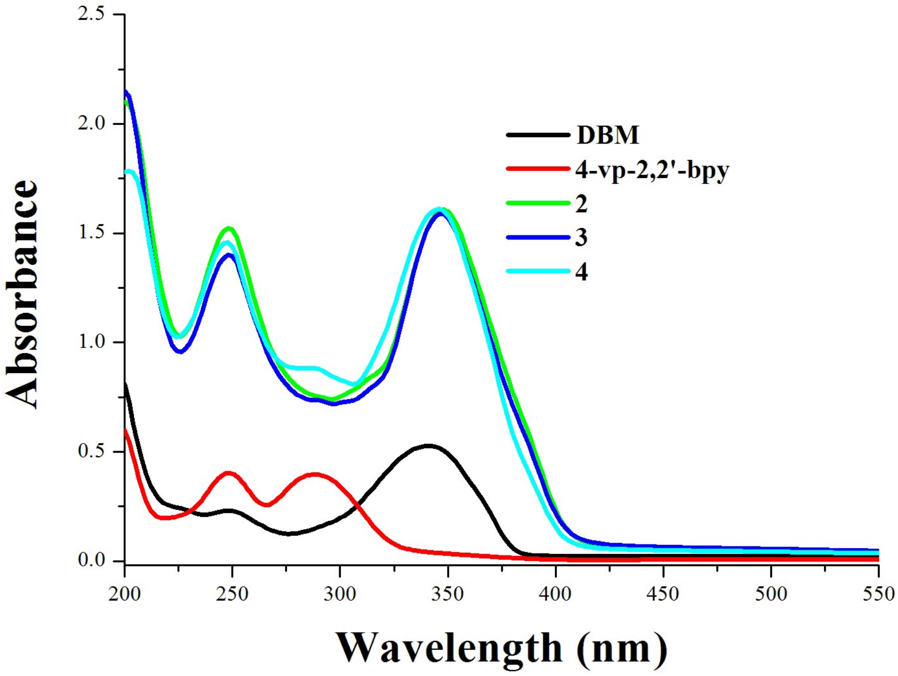

Figure S3 Schematic energy level diagram and energy transfer process of Eu^{3+} or Tb^{3+} for complexes **2-3** in solution.

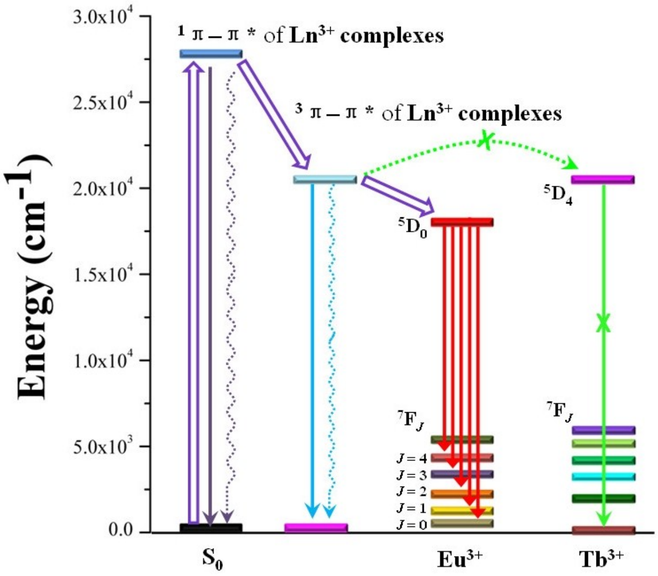

Figure S4 PXRD patterns of PVK and its metallopolymers **Poly(2-*co*-NVK-*co*-4)** (200:1; [**2**/**4** = 1:5 or 1:7]) in solid film.

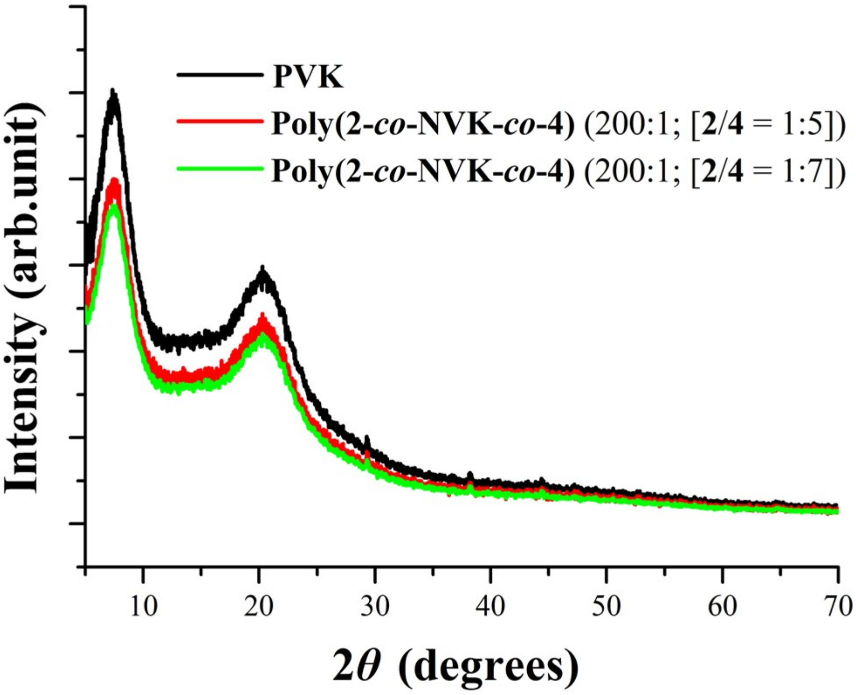

Figure S5 TG curves of PVK, complexes **2** and **4**, and metallopolymers **Poly(2-*co*-NVK-*co*-4)** (200:1; [**2**/**4** = 1:5 or 1:7]) in solid state.

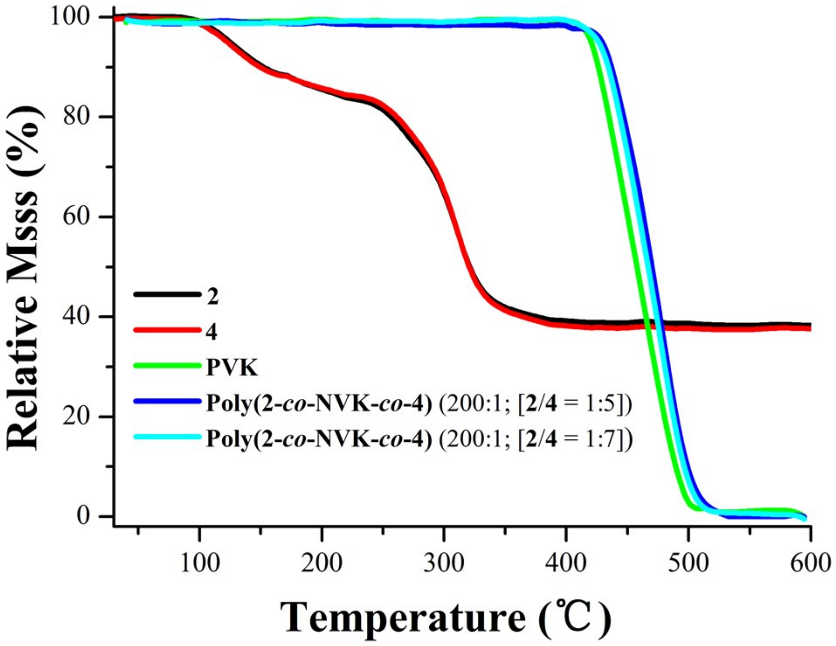

Figure S6 DR spectra of PVK and **Poly(2-*co*-NVK-*co*-4)** (200:1; [**2**/**4** = 1:5 or 1:7]) in solid film.

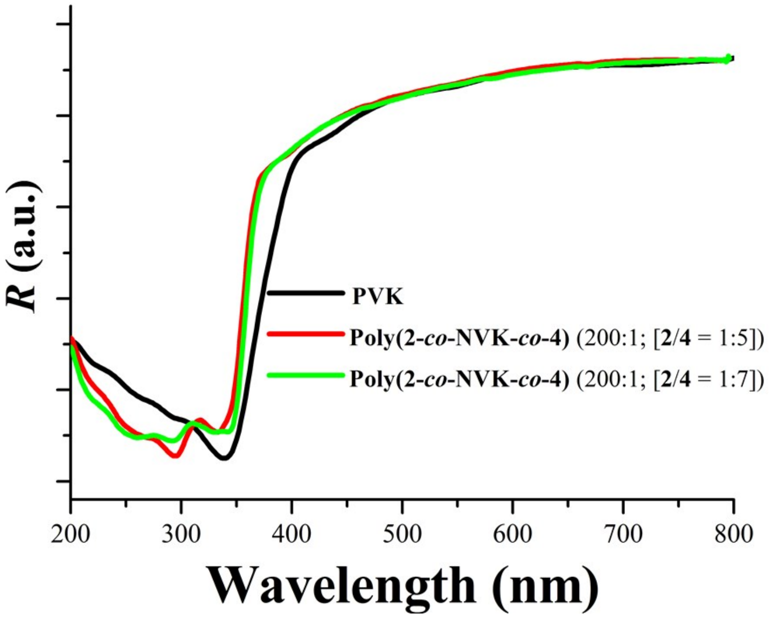

Figure S7 Excitation spectra of **Poly(2-*co*-NVK-*co*-4)** (200:1; [**2**/**4** = 1:5 or 1:7]) in solid film by λ_{em} = 613 nm.

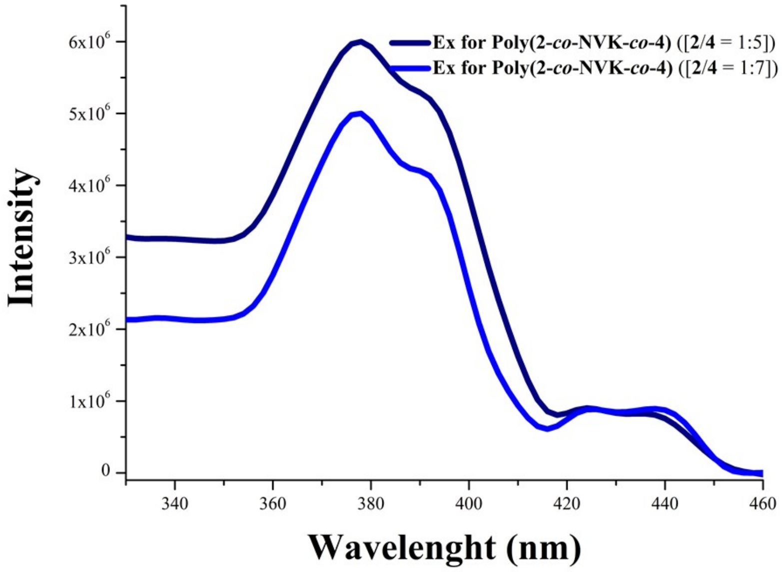

Figure S8 Emission spectra and corresponding CIE coordinates of **Poly(2-*co*-NVK-*co*-4)** (200:1; [**2**/**4** = 1:5]) in solid film upon excitation of λ_{ex} = 350, 355 or 360 nm.

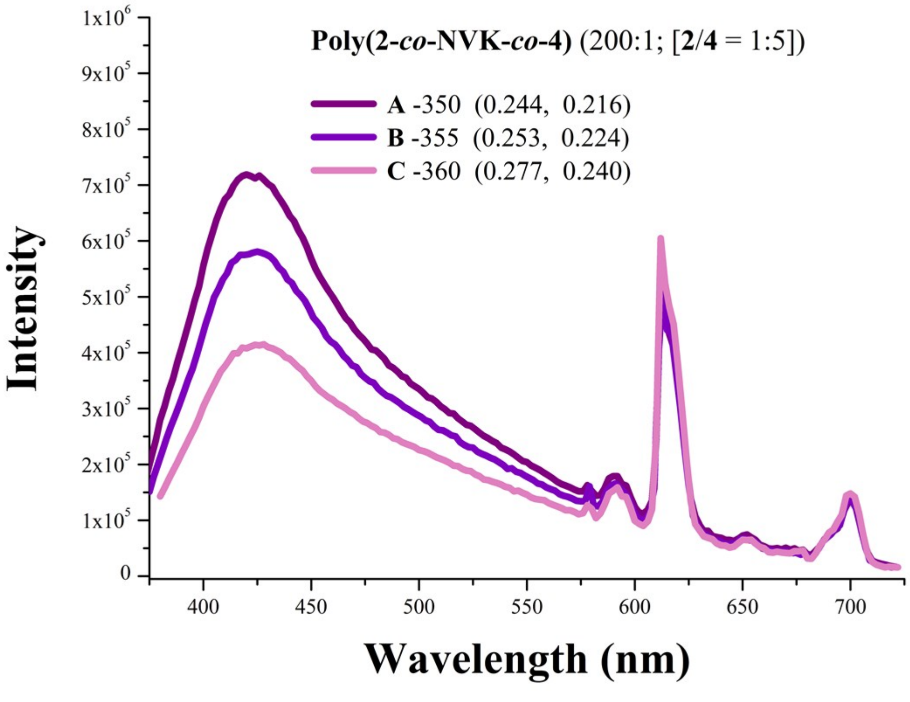

Figure S9 Emission spectra and corresponding CIE coordinates of **Poly(2-*co*-NVK-*co*-4)** (200:1; [**2**/**4** = 1:7]) in solid film upon excitation of λ_{ex} = 348 or 358 nm.