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Water-Soluble Polyfluorenes as an Electron Injecting Layer in PLEDs for Extremely High Quantum Efficiency**

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For more than a decade, polymer light-emitting diodes (PLEDs)^[1] have attracted continuous attention because of their low costs, easy processability, and the possibility of easily fabricating flexible, large-area displays. In PLEDs, electrons and holes are injected from the anode and cathode to the emitting polymers where they then recombine, resulting in light emission. To achieve high efficiency, it is necessary that both electrons and holes are efficiently injected.^[2] Therefore, substantial improvements in the device efficiency have been obtained upon modification of the anode,^[3] by the introduction of low-work function cathodes,^[4] and by using hole and electron injecting/transporting layers.^[5–7] Efficient injection of electrons from the cathode to emissive conjugated polymers plays an important role in improving the device efficiency and stability. As the majority of carriers of most existing conjugated polymers are holes, electron injection is more difficult than hole injection.^[5a,8] It is well-known that electron injection is dependent on the energy barrier height between the emitting layer and the cathode. Thus, low-work-function metals such as Ca and Ba are used to enhance electron injection into organic materials; however, these metals are not stable in air, and they may sometimes react with and diffuse into organic materials, leading to the deterioration of the device.

To complement the weakness of low-work-function metals, a stable and high-work-function metal cathode such as Al along with an alkali or alkaline earth fluoride (LiF, CaF, MgF) as an electron injecting layer (EIL) was inserted using the thermal evaporation method. As a result, the efficiency of PLEDs was enhanced owing to a lowering of the effective barrier height by the space charge field.^[9] Water-soluble polymers are considered to be potential candidates for the EIL in PLEDs because they do not cause damage to the underlying

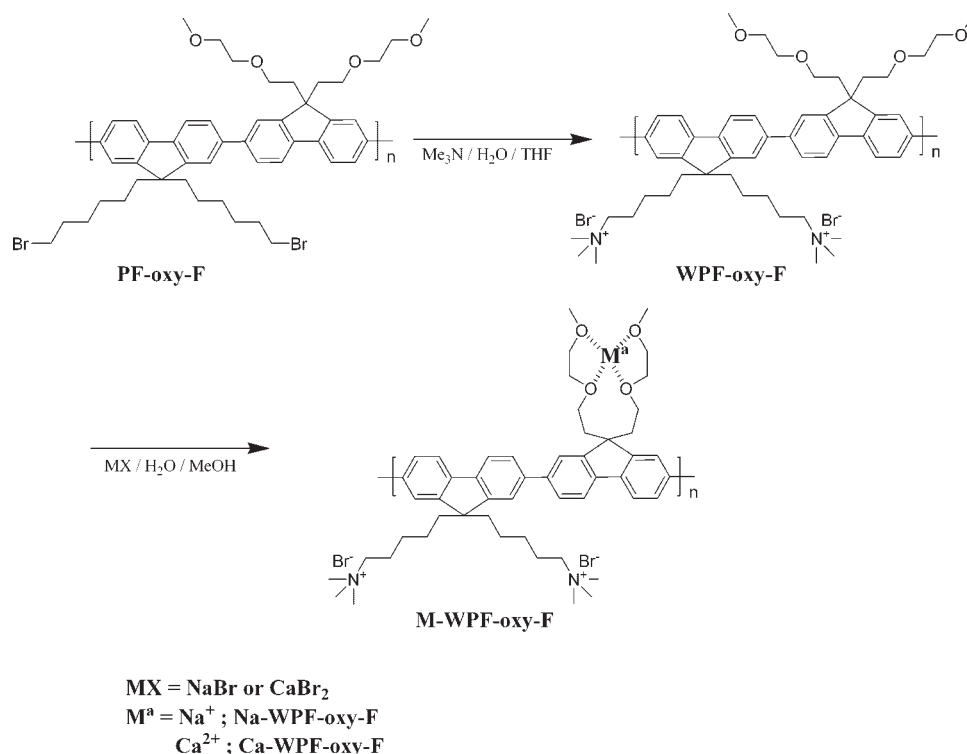
organic soluble polymer film and possess either ionic or polar groups that can effectively induce interface dipoles^[10] or space charge fields.^[6] Recently, water-soluble conjugated polymers with positive ammonium ions were used as an EIL.^[5] Owing to the enhancement in the interfacial dipole between the emitting layer and the metal cathode, it has been reported that this bilayer cathode could improve electron injection.^[5] Furthermore, water-soluble poly(ethylene glycol) (PEG)^[6,7] blending with ionic salt between the emitting layer and the Al cathode improved device efficiency by increasing the built-in potential. However, phase separation occurred between the ionic salt and PEG, and the use of PEG alone as an EIL was not possible because of its insulating properties.^[6]

This Communication reports the synthesis of novel poly[(9,9-bis((6'-(*N,N,N*-trimethylammonium)hexyl)-2,7-fluorene)-*alt*-(9,9-bis(2-(2-methoxyethoxy)ethyl)-fluorene))] dibromide, bound with mobile alkali or alkaline earth metal ions without their counter ions (**M-WPF-oxy-F**) as shown in Scheme 1, and the use of these water-soluble polymers as an EIL in the PLEDs. We previously reported a water-soluble polyfluorene derivative with ion-transporting side groups and mobile bromide ions.^[11] When this water-soluble polymer was inserted between the emitting layer and the high-work-function Ag cathode in PLEDs, the device efficiency was dramatically increased.^[11] This was because of the migration of mobile bromide ions that led to a large space charge field. As a result, the injection barrier could be reduced between the emitting layer and the Ag cathode, resulting in high electroluminescence efficiency. However, the migration of bromide ions was restricted because bromide anions formed strong ion pairs with ammonium counter ions.^[12] Facilitative migration could be expected when mobile metal ions without counter ions were bound to ethylene oxide side groups with ion-transporting properties.^[12] This enhanced ion mobility could improve the electroluminescence efficiency of PLEDs, even with a high-work-function metal cathode. When **M-WPF-oxy-F** binding Na⁺ or Ca²⁺ ions were inserted between the emitting layer and the Al cathode as an EIL, the device efficiency was significantly enhanced. These results show that by using **Na-WPF-oxy-F** or **Ca-WPF-oxy-F** as an EIL, a very high luminous efficiency of green fluorescent PLEDs with an Al cathode was obtained: 11.0 and 15.4 cd A^{−1}, respectively. In particular, when **Ca-WPF-oxy-F** was used, an extremely high external quantum efficiency of 4.8% was obtained, which is approaching the theoretical maximum external EL quantum efficiency of about 5%,^[13] despite the use of the stable high-work-function Al cathode.

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Scheme 1. Synthesis of M-WPF-oxy-F.

As shown in Scheme 1, the water-soluble polymer (**WPF-oxy-F**)^[11] was ionized by using a trimethylamine solution. Neutral polymers **PF-oxy-F** were dissolved in tetrahydrofuran (THF). A trimethylamine solution was added into the solution and was stirred at room temperature (rt) for 24 h. The solubility of the ionic polymer in THF was low, and the polymer was precipitated during this period. Water was added to the solution to dissolve the precipitate. The solution was evaporated, and the residue was collected and dried under vacuum overnight and then redissolved in methanol. The solution was slowly poured into stirred ether, yielding the ionic polymer. **WPF-oxy-F** was dissolved in methanol and water. Binding of the Na^+ or Ca^{2+} ions with the ethylene oxide groups in **WPF-oxy-F** was performed by adding a NaBr or

CaBr_2 solution into a **WPF-oxy-F** solution with vigorous stirring. Subsequently, non-binding and remnant metal ions were removed using a dialysis membrane with a 2000 molecular weight cut-off (MWCO) rating, and **M-WPF-oxy-F** was solidified by freeze-drying.

Figure 1 shows the binding energy between metal ions and oxygen atoms in ethylene oxide units. Binding between Na^+ or Ca^{2+} ions and the ethylene oxide units was confirmed by the binding energy of Na^+ or Ca^{2+} with the oxygen atom^[12a,14] using X-ray photoelectron spectroscopy. Owing to the presence of the ethylene oxide units that can stabilize and transport metal ions,^[12] Na^+ and Ca^{2+} ions were successfully bound to the polymer. In a previous study, we showed that **WPF-oxy-F** can serve as a good EIL even without metal ions owing to the large dipole or space charge formation.^[11]

Further enhancement can be expected when these mobile metal ions are additionally incorporated into the polymer structure. Therefore, the **M-WPF-oxy-F** series was used as an electron injecting layer in PLEDs, with AIPF11^[15] as a hole injecting layer (HIL) and a green-light-emitting polyfluorene derivative (LUMINATION 1100 series, Dow Chemical Company) as an emitting layer. Figure 2 shows the device structure and schematic energy diagrams of water-soluble polyfluorene derivatives as an EIL. The structure of the devices was indium tin oxide (ITO)/AIPF11 (30 nm)/Dow Green (70 nm)/water-soluble polyfluorene (3 nm)/Al (150 nm). All devices were fabricated from the same batch. The device efficiency was determined by measuring the light output in only the forward direction.^[16]

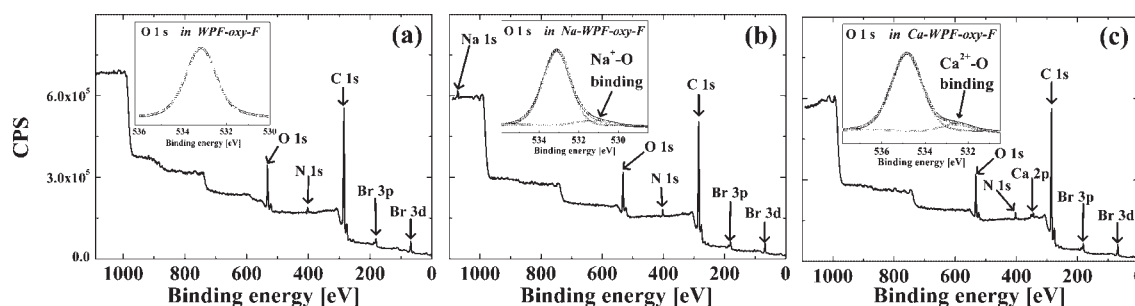


Figure 1. XPS spectra. a) WPF-oxy-F, XPS of the O1s (inset); b) Na-WPF-oxy-F, XPS of the Na^+ -O binding (inset); c) Ca-WPF-oxy-F, XPS of the Ca^{2+} -O binding (inset).

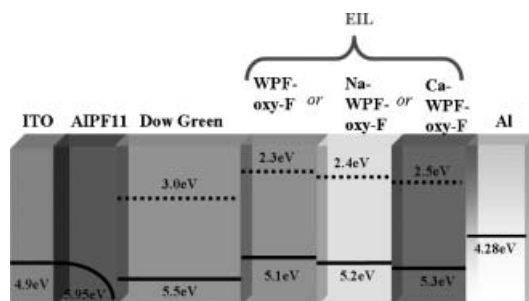


Figure 2. Schematic energy level of a device with configuration indium tin oxide (ITO)/AlPF11 (30 nm)/Dow Green (70 nm)/EIL (3 nm)/Al (150 nm).

Figure 3 shows the current density–luminance–voltage (I – V – L) characteristics of each device. The operating voltage at 100 cd m^{-2} of the device without EIL was 11.5 V, and the maximum luminance was 267 cd m^{-2} at 13.5 V. An injection of electrons from the Al cathode was not efficient owing to the high injection barrier between the emitting layer and the Al cathode. However, when **WPF-oxy-F**, **Na-WPF-oxy-F** and **Ca-WPF-oxy-F** were used as electron-injection layers, the operating voltage at 100 cd m^{-2} was reduced to approximately 5.3 V, 4.2 V, and 4.2 V and the maximum luminance of each device was dramatically increased to 14000, 16000 and 20000 cd m^{-2} , respectively.

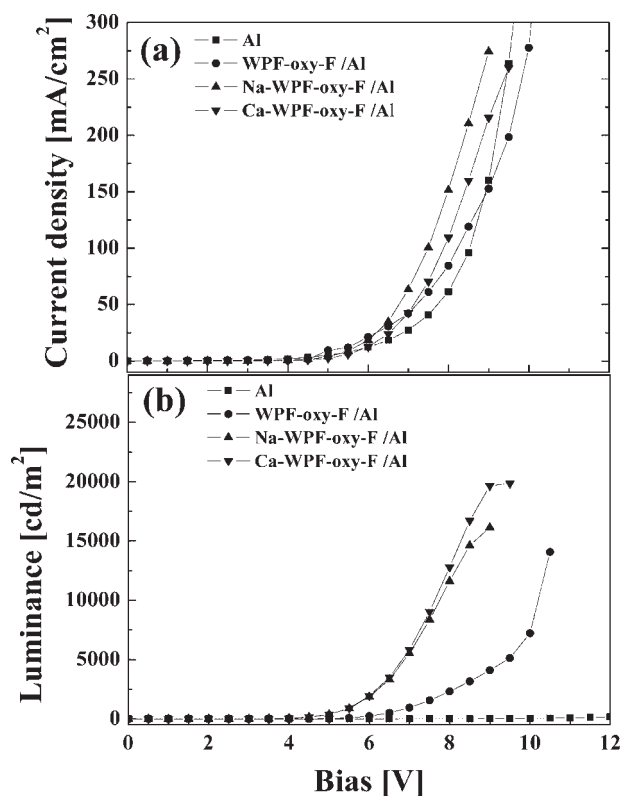


Figure 3. I – V – L characteristics of each device.

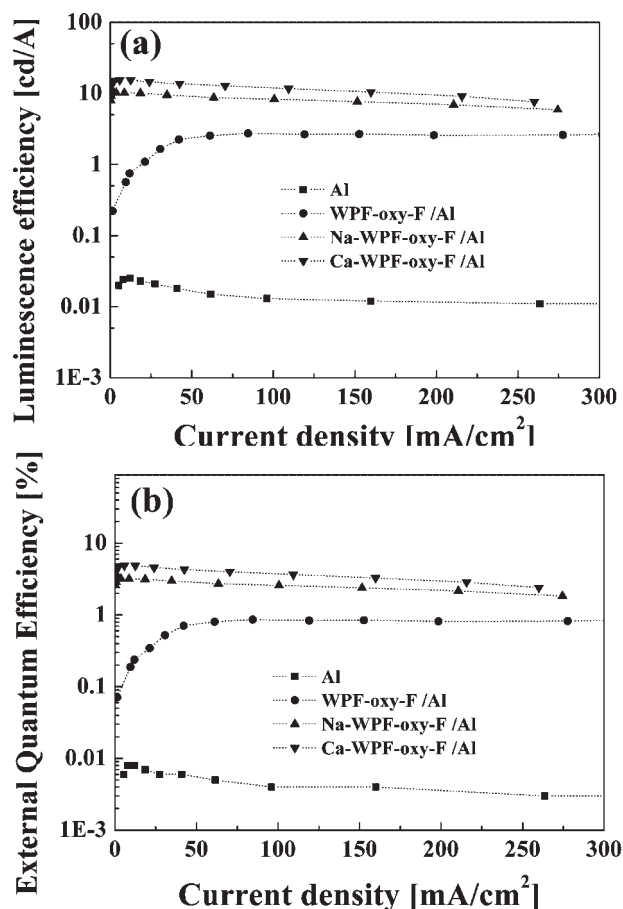


Figure 4. a) Luminescence efficiency of each device. b) External quantum efficiency of each device.

Figure 4 compares the efficiency of each device. The maximum luminescence efficiency of the device with only an Al cathode was 0.02 cd A^{-1} and the external quantum efficiency (η_{ext}) was 0.008%. Conversely, when **WPF-oxy-F**, **Na-WPF-oxy-F**, and **Ca-WPF-oxy-F** were used as EILs, the maximum luminescence efficiency was 3.0, 11.0, and 15.4 cd A^{-1} and η_{ext} was 0.9, 3.4, and 4.8%, respectively. This enhancement was produced because two ethylene oxide side groups in **WPF-oxy-F** at the 9-position led to an interfacial dipole interaction between the Al cathode and the lone-pair electrons on the oxygen atoms. Therefore, the effective charge injection barrier could be reduced between the emitting layer and the Al cathode.^[6,7] As can be seen from Figure 4a and b, the efficiency of the device with **Na-WPF-oxy-F** or **Ca-WPF-oxy-F** containing metal ions was much higher than that of a device with only a **WPF-oxy-F** layer. In the case of **Na-WPF-oxy-F** and **Ca-WPF-oxy-F**, a much larger enhancement was observed compared to the **WPF-oxy-F** device. The η_{ext} of PLEDs for **Na-WPF-oxy-F** was 3.4%, and that of **Ca-WPF-oxy-F** was 4.8%, which is close to the theoretical maximum η_{ext} of about 5%.^[13] To our knowledge, this extremely high efficiency has

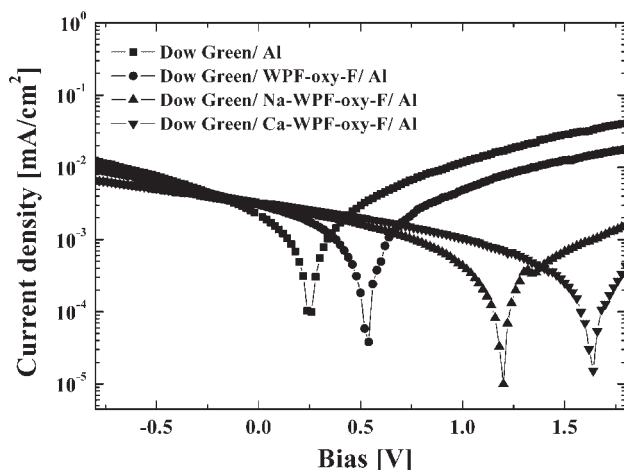


Figure 5. Photovoltaic characteristics of the device with configuration ITO/AIPF 11/Dow Green/EIL/Al.

never before been reported in PLEDs with Al cathodes, rather than from an unstable low-work-function cathode. These water-soluble polyfluorenes have identical polymer main chains except for their metal ions. Therefore, metal ions binding to ethylene oxide groups in **WPF-oxy-F** seem to strongly affect the performance of PLEDs.

To investigate and compare the effects of these EILs on the electron injection barrier, photovoltaic behavior was observed by photovoltaic measurements of the open-circuit voltage (V_{oc}) across the device.^[5,17] Figure 5 shows the photovoltaic behaviors of each device. As the anode of each device was identical, the effective work function of the cathode influenced the V_{oc} . In the case of the Dow Green/Al device, the V_{oc} was 0.24 V. However, when **WPF-oxy-F**, **Na-WPF-oxy-F**, and **Ca-WPF-oxy-F** were used as EIL, the V_{oc} was found to be 0.53 V, 1.20 V, and 1.64 V, respectively. The V_{oc} of the devices with **Na-WPF-oxy-F** or **Ca-WPF-oxy-F** as an EIL was higher than **WPF-oxy-F** without metal ions because of the enhanced interfacial dipole formation or space charge field and the consequent lower barriers.^[5,18] Therefore, V_{oc} studies confirmed that the efficiency of PLEDs has been improved by balancing the hole with the electron via enhanced electron injection from the cathode to the emissive layer.

In the case of **Na-WPF-oxy-F** or **Ca-WPF-oxy-F**, which are binding metal ions by ethylene oxide side groups, the device efficiency was significantly enhanced. It has been reported that because ethylene oxide units are able to transport mobile ions,^[12] polymers containing ethylene oxide units, such as PEG, can be used as solid-state ionic transporting materials in light-emitting electrochemical cells (LECs).^[19,20] LECs have low turn-on voltages and high efficiencies, even though LECs typically have a slow response time because of the low ionic mobility of the solid-state electrolyte. One mechanism of the enhanced device efficiency in the case of **Na-WPF-oxy-F** or **Ca-WPF-oxy-F** can be attributed to mobile metal ions binding

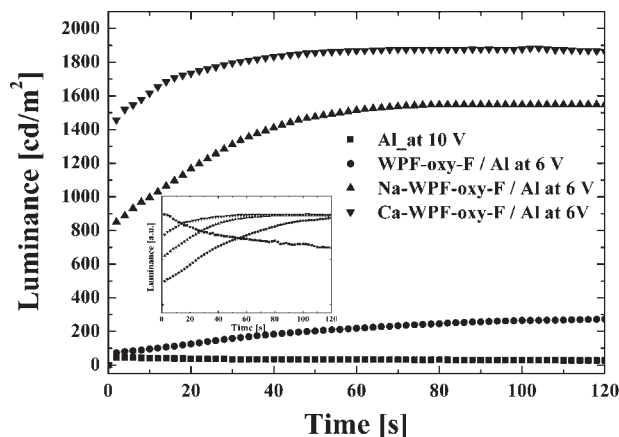


Figure 6. Luminance as a function of time of each device with EIL biased at 6 V, except for Al, which is biased at 10 V. Inset: normalized luminance vs. time. (Device configuration: ITO/AIPF 11/Dow Green/EIL/Al.)

to ethylene oxide side groups similar to the ion transporting mechanism of LEC. Thus, the luminance of each device was measured as a function of time. As can be seen from Figure 6, as soon as the bias was applied to each device, a gradual increase in the luminance of the **Na**-, **Ca**-, and **WPF-oxy-F** were observed, when compared to the device without EIL. These results implied that mobile metal ions and bromide ions migrate through the ethylene oxide groups. In the Figure 6 inset, the rate of the increase in luminance of **WPF-oxy-F** was slower than that of **Na-WPF-oxy-F** or **Ca-WPF-oxy-F** because **WPF-oxy-F** contained bromide ions that formed strong ion-pairs with ammonium ions.^[21] However, in the cases of **Na-WPF-oxy-F** or **Ca-WPF-oxy-F**, the rate of the increase in luminance was faster than **WPF-oxy-F**, since the migration of mobile metal ions without counter ions was facilitated. Migrated metal and bromide ions could accumulate in the vicinity of the electrode and induce a larger space charge field,^[9,22] more effectively reducing the barrier of electron injection^[18] than **WPF-oxy-F**. Therefore, the efficient migration of metal ions loosely bound to ethylene oxide units seems to play an important role in efficient electron injection for high-efficiency PLEDs with high-work-function Al cathodes.

In conclusion, novel water-soluble polyfluorene derivatives containing mobile metal ions bound to ethylene oxide groups have been developed for use as an efficient electron injecting layer for high electroluminescence efficiency in PLEDs with a high-work-function Al cathode. Facilitated migration of metal ions by ion transporting ethylene oxide groups dramatically improved the device efficiency through effectively reducing the electron injection barrier. Consequently, by using **Na-WPF-oxy-F** or **Ca-WPF-oxy-F** as an EIL, a high luminous efficiency of 11.0 and 15.4 cd A^{-1} was obtained in green fluorescence PLEDs with Al cathodes, respectively. In particular, when **Ca-WPF-oxy-F** was used, an extremely high

external quantum efficiency of 4.8% was obtained, which is approaching the theoretical limit.

Experimental

Poly[(9,9-bis(6-bromohexyl)-fluorene)-alt-(9,9-bis(2-(2-methoxyethoxy)ethyl)-fluorene)] (PF-oxy-F): 0.74 g of 2,7-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-9,9-(6-dibromohexyl)-fluorene, 0.52 g of 2,7-dibromo-9,9-bis(2-(2-methoxyethoxy)ethyl)-fluorene and 60 mL THF were placed in a two-neck flask under a nitrogen atmosphere. 20 mL of 2 M K₂CO₃ solution was injected, and 1 mol % of Pd(PPh₃)₄ was added to the solution. The solution was refluxed for 3 days. The reaction mixture was slowly cooled to room temperature and poured into a mixture of 250 mL of methanol and water (methanol/water = 4:1). The precipitate was collected, redissolved in chloroform and reprecipitated several times by adding methanol. The resulting polymer was a yellowish-white powder (yield: 70%). ¹H-NMR (300 MHz, CDCl₃, Me₄Si, δ): 7.85 (br, 6H, fluorene), 7.73 (br, 6H, fluorene), 3.35 (br, 4H, -CH₂Br), 3.29 (br, 6H, -OCH₃), 2.95 (br, 4H, -CH₂O-), 2.62 (br, 4H, -OCH₂-), 2.50 (br, 4H, -CH₂O-), 2.19 (br, 8H, -CH₂-), 1.71 (br, 4H, -CH₂-), 1.26 (br, 8H, -CH₂-), 0.81 (br, 4H, -CH₂-). Anal. calcd. for C₄₈H₅₈Br₂O₄: C 67.13, H 6.81; found: C 67.27, H 6.54.

Poly[(9,9-bis((6'-(N,N,N-trimethylammonium)hexyl)-2,7-fluorene)-alt-(9,9-bis(2-(2-methoxyethoxy)ethyl)-fluorene)]Dibromide (WPF-oxy-F): 0.25 g of PF-oxy-F was dissolved in 100 mL of THF. 5 mL of trimethylamine solution was added to the solution, which was then stirred at rt for 24 h. The solubility of the ionic polymer was reduced in THF and precipitated during this period. 20 mL of water was added to the solution in order to dissolve the precipitate. The solution was evaporated and the residue was redissolved in methanol. The ionic polymer was precipitated from ether. The ionic polymer was a yellow powder (yield: 88%). ¹H NMR (300 MHz, CD₃OD, Me₄Si, δ): 7.91 (br, 6H, fluorene), 7.80 (br, 6H, fluorene), 3.26 (br, 4H, -CH₂N-), 3.23 (br, 6H, -OCH₃), 3.00 (br, 18H, -NCH₃), 2.90 (br, 4H, -CH₂O-), 2.61 (br, 4H, -OCH₂-), 2.49 (br, 4H, -CH₂O-), 2.29 (br, 8H, -CH₂-), 1.58 (br, 4H, -CH₂-), 1.19 (br, 8H, -CH₂-), 0.75 (br, 4H, -CH₂-). Anal. calcd. for C₅₄H₇₆Br₂N₂O₄: C 66.38, H 7.84, N 2.87; found: C 66.49, H 7.54, N 2.78.

General Procedure for Binding to Metal Ions in Poly[(9,9-bis((6'-(N,N,N-trimethylammonium)hexyl)-2,7-fluorene)-alt-(9,9-bis(2-(2-methoxyethoxy)ethyl)-fluorene)]Dibromide): 20 mg of poly[(9,9-bis((6'-(N,N,N-trimethylammonium)hexyl)-2,7-fluorene)-alt-(9,9-bis(2-(2-methoxyethoxy)ethyl)-fluorene)]dibromide (WPF-oxy-F) was dissolved in 5 mL of methanol and 5 mL water. 10 mL of 0.1 M NaBr or CaBr₂ in water was added to the solution with vigorous stirring for 4 h. Subsequently, non-binding and remnant metal ions were removed by using dialysis membrane with 2000 MWCO for 24 h. The solution was removed by freeze drying for 48 h.

X-Ray Photoelectron Spectroscopy: The X-ray spectroscopy (XPS) measurements were carried out using a MultiLab2000 (THERMO VG SCIENTIFIC) system equipped with a monochromatized Mg K α radiation at $h\nu = 1253.6$ eV and with a base pressure 1×10^{-9} Pa. The binding energies were calibrated to the C 1s peak at 285.0 eV.

Device fabrication and Characterization: These water-soluble polyfluorenes were used as an electron-injecting layer in PLEDs. The structure of the devices was ITO/AIPF11 (30 nm)/Dow Green (70 nm)/water-soluble polyfluorene (3 nm)/Al (150 nm). The glass substrate, precoated with ITO, was cleaned in an ultrasonic bath with acetone, followed by 2-propanol. A surface treatment was carried out by UV-ozone. The hole injecting layer (AIPF11) was spin-coated onto the ITO at a thickness of 30 nm and baked at 200 °C for 5 min on a hot plate under air. A green-light-emitting polyfluorene derivative (LUMATION 1100 series Green Polymer, Dow Chemical company (currently Sumitomo)) dissolved in toluene was spin-coated onto the HIL layers to obtain a thickness of 70 nm, and was subsequently baked

at 130 °C for 15 min under N₂. A 0.2 wt % solution of WPF-oxy-F, Na-WPF-oxy-F, or Ca-WPF-oxy-F in methanol was spin-coated onto the emitting layer as an electron-injecting layer and baked at 130 °C for 5 min under N₂. Al was deposited as the cathode through a shadow mask by thermal evaporation at a base pressure of 1×10^{-6} Torr (1 Torr = 1.333×10^2 Pa). Device efficiency was determined by measuring the light output only in the front direction under ambient conditions. The pixel size was 4.0 mm². The current-voltage-luminescence (*I-V-L*) characteristics of the devices were obtained with a Keithley 2400 source measure unit and a Photo Research PR650 spectrophotometer. Photocurrent-voltage measurements were performed using a Keithley 4200 measurement in N₂ under AM1.5 G illumination (100 mW cm⁻²) by a xenon light source (Müller).

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