Enhanced Performance in Organic Light-Emitting Diodes with Copolymers Containing Both Tris(8-hydroxyquinoline) Aluminum and 8-Hydroxyquinoline Lithium Groups

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ABSTRACT: We synthesized novel copolymers containing both tris(8-hydroxyquinoline) aluminum (Alq_3) and 8-hydroxyquinoline lithium (Liq) groups as emitting layers for use in conventional two-layer organic light-emitting diodes. The network structure and thermal stability of these materials is described. The optical and electroluminescent properties of the copolymers were also studied. The performance optimiza-tion of the devices with the copolymers through the variation of the ratio of Alq_3 to Liq is described. A mechanism responsible for the improved electron injection is put forward. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 102: 4404–4410, 2006

Key words: crosslinking; light-emitting diodes (LED); luminescence

INTRODUCTION

Organic light-emitting diodes (OLEDs) have become very attractive because of their potential applications in large area, flat-panel displays.[1,2] Since the demonstration of the first double-layer electroluminescent (EL) device with tris(8-hydroxyquinoline) aluminum (Alq_3) in 1987,[1] a great deal of effort has been made to improve the charge injection, which results in improved EL efficiency and the long-term stability of OLEDs. Low-work-function metals and their compounds, such as Li or LiF,[3] have been widely used to improve electron injection. Unfortunately, low-work-function metals are readily oxidized and limit the lifetime of the devices. It is, therefore, desirable to use more environmentally stable electrodes such as Al, but the high work function of the Al cathode results in a lower luminescent efficiency and a higher operating voltage (V) in OLEDs. Recently, a number of groups have reported that electron injection can be significantly improved by the doping of Alq_3 with a Li complex.[4] Al cathodes with LiF[5] or a thin layer of LiF between the organic layer and the Al cathode[6–8] have also been reported. Lithium quinolate complexes have also been used as interface materials to assist electron injection.[9,10] They can be deposited in much thicker layers in OLEDs than LiF, which has a thickness restriction (< 2 nm) because of its high electrical insulating properties.[11] In our laboratory, we were interested in a 8-hydroxyquinoline lithium (Liq) containing polymer, and in this article, we report our results of the thermal polymerization of copolymers containing both Alq_3 and Liq groups and the resulting OLEDs with the copolymers as the emitting layers.

EXPERIMENTAL

Materials

8-Hydroxyquinoline and *p*-methoxyphenol were purchased from Aldrich Chemical Co. (Milwaukee, WI) and were used without further purification. Triethylaluminum solution in hexane was purchased from Tokyo Kasei Kogyo Co., Ltd. (Fukaya City, Japan) 2-Hydroxyethyl methacrylate was supplied from Mitsubishi Corp. (New York, NY) and was used without further purification. LiOH and sodium acetate were dehydrated at 100°C *in vacuo* for 24 h and were then stored in a desiccator. All of the solvents and other chemicals were used after purification according to conventional methods.

General method

^{1}H-NMR spectra were recorded on a DRX 400-MHz superconducting magnet NMR spectrometer (Brukor, Ettlingen, Germany). The Fourier transform infrared (FTIR) spectra were carried out with an RFX-65A FTIR spectrometer (Anatel Co., Loveland, CO). The molecular weights were determined by a Waters 515-410 GPC gel permeation chromatograph (Waters, Milford, MA) with a UV detector and with THF as the eluent and polystyrene as the standard. Thermal analyses were performed on a TA differential scanning calorimeter (model 2910) (TA Instruments Inc., New Castle, DE) and a Netzsch thermogravimetric analyzer (TG 209 C Iris) (Netzsch, Selb/Bavarian, Germany) at a heating rate of 10 K/min under a nitrogen purge of 50 mL/min. Purification of the intermediates and products was mainly accomplished by column chromatography with silica gel 60 (200–400 mesh). All glassware was thoroughly cleaned by ultrasonic rinsing with acetone followed by distilled water and isopropyl alcohol and then dried in an oven at 100°C for 60 min. The purity of the final products was normally confirmed by elemental analysis.

Alq$_3$ monomer synthesis

The synthesis of the Alq$_3$ monomer and Liq monomer was carried out according to the literature with slight modifications.[12–14] The details are reported in the Appendix.

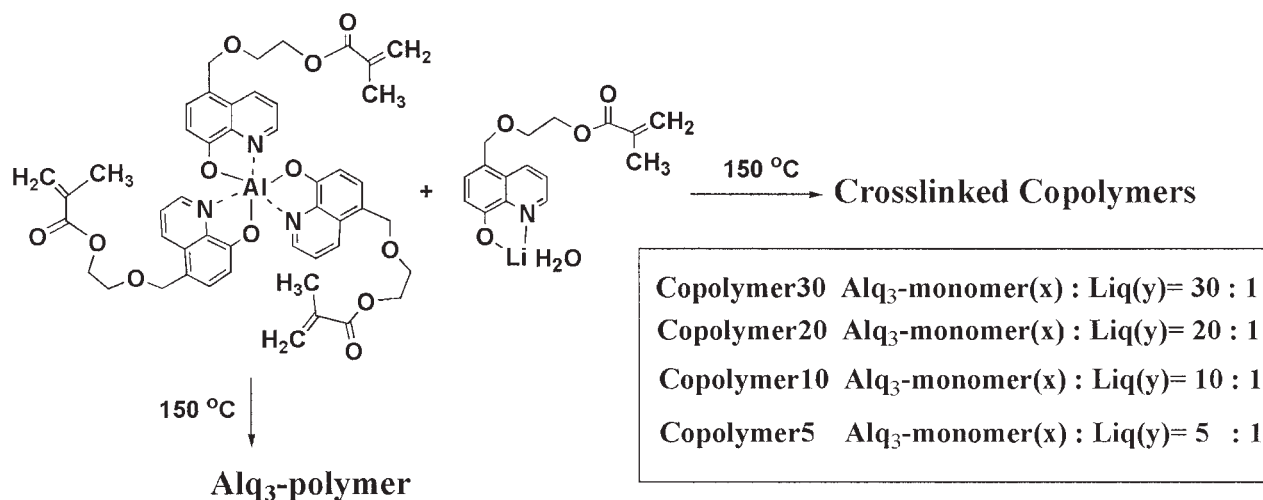Synthesis of the Alq$_3$ polymer and the copolymers containing both Alq$_3$ and Liq groups (Scheme 1)

Films of the Alq$_3$ monomer were prepared by spin-coating from a 2–8 wt % solution in toluene onto a glass substrate and were subsequently baked at 40°C for 30 min. The polymerization was carried out on a Cole Parmer Digital 04644 hot plate (Cole Parmer, Vernon Hills, IL) at 150°C for 15 min. No intiator was used. Sample processing was carried out in a dry-nitrogen-filled glovebox to avoid oxygen and water contamination. No change in the films was observed with an optical microscope over several months at room temperature. FTIR and differential scanning calorimetry (DSC) spectra and solubility tests were used to prove that the polymerization occurred and to also indicate the degree of polymerization. The polymers were washed in chloroform for 30 s, and then, the unpolymerized and partially polymerized regions of the film were washed away and analyzed by gel permeation chromatography. UV–visible absorption spectra of the films before and after washing were recorded on a HP 8453 instrument (Hewlett-Packard, Palo Alto, CA).

The processing and characterization of the copolymers was same as given for the synthesis of the Alq$_3$ polymer. The Alq$_3$ monomer and Liq monomer taken in the desired weight ratio were dissolved in 1,1,2,2-tetrachloroethane. The films were prepared by spin-coating from a 2–8 wt % solution onto glass substrates and polymerized at 150°C for 15 min.

Light-emitting diode (LED) device preparation

Photoluminescence (PL) measurements of the film were conducted in a Fluorolog-3 fluorescence spectrometer (HORIBA Jobin Yvon, Edison, NJ) at 390 nm. The PL quantum yields were tested as described by Greenham et al.[15] in an integrating sphere (IS-80, Lab-Sphere Co., North Sutton, New Hampshire) under an excitation of 405 nm. The monomers were dissolved in the solvent and filtered through a 0.45-μm filter. Indium tin oxide (ITO) coated glass substrates were cleaned with acetone, detergent, distilled water, and

Scheme 1 Synthesis of the copolymers containing both Alq$_3$ and Liq groups and the Alq$_3$ polymer.

2-propanol and, subsequently, in an ultrasonic bath. After treatment with oxygen plasma (Plasma-Preen-862, Plasmatic Systems Inc., North Brunswick, NJ), 44 nm of poly(3,4-ethylenedioxythiophene) (PEDOT) (4083, EL-grade, Bayer, Krefeld, Germany) from the 1,1,2,2-tetrachloroethane solution was spin-coated onto the substrate followed by drying in a vacuum oven at 80°c for 8 h. The thin films of the EL monomers were coated onto PEDOT by spin-coating and were then polymerized by heating. The film thickness of the active layers was around 100 nm, as measured with an Alfa Step 500 surface profiler (Tencor). The LiF (EL-grade, Aldrich, St. Louis, MO, 2.5 nm) and Al layers were vacuum-evaporated on the top of an EL polymer layer under a vacuum of $1–10^{-4}$ Pa. Current (I)–V characteristics were recorded with a Keithley 236 source meter (Keithley Instruments Inc., Cleveland, OH). EL spectra were recorded by an Oriel Instaspec IV (Stratford, CI) charged coupling device (CCD) spectrograph. Luminance and external quantum efficiencies were determined by a calibrated photodiode. Device fabrication and measurement were done inside a dry-nitrogen-filled glovebox.

RESULTS AND DISCUSSION

Synthesis

Generally, lithium complexes are highly sensitive to moisture and oxygen due to their chemical reactivity, which results in difficulties in device fabrication. The Liq monomer was also a hygroscopic material. The preparation and polymerization were carried out in a glovebox filled with dry N_2 to avoid contamination by H_2O and O_2. In our experiments, we used LiF/Al as the bilayer cathode and a series of copolymers containing both Alq_3 and Liq groups as the emitting layer. The Liq monomer with one molecule of crystallization of water was directly obtained by the reaction of 5-(2-methacryloylethyloxymethyl)-8-quinolinol with LiOH (1 : 1) and was characterized by ^{1}H-NMR, FTIR spectroscopy, and elemental analysis.

The copolymers with the crosslinkable structures were prepared with different weight concentration ratios of the Alq_3 monomer and Liq monomer. The Alq_3 polymer was also polymerized under the same conditions as described previously. The Alq_3 polymer films were spectroscopically investigated with FTIR, as reported previously.[12] The copolymerization of the Alq_3 and Liq monomers was similar to the conversion of the Alq_3 polymer. For example, the IR absorption peaks corresponding to the methacrylate group for copolymer 20 from monomers located at 815, 932, 1168, 1298, 1321, 1635, and 1718 cm^{-1} are shown in Figure 1. The FTIR spectra confirmed that a high degree of polymerization had occurred. After polymerization, the
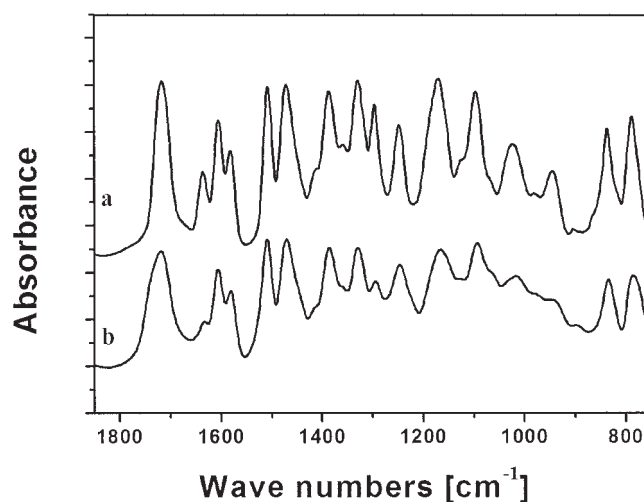
Figure 1 Spectroscopic verification of the thermal polymerization in a thin film (copolymer 30) at 150°C by FTIR (a) before and (b) after polymerization.

characteristic band located at 1635 cm^{-1}, which could be attributed to the stretching vibration of the C=C double bonds of the methacrylic moieties, had almost completely disappeared. With increasing concentration of Liq groups in the copolymers, the characteristic band of the Liq groups at 1375 cm^{-1} (Li—O) appeared, which confirmed that the Liq groups were covalently bound to the network.

The values of the heat of reaction under isothermal conditions were about 70 J/g, as taken from scans of the series copolymers polymerized at 150°C. Because the dynamically cured samples did not show any noticeable residual heat on the second heating, we assumed the reaction had reached completion.

The Alq_3 polymer film and the copolymers films on glass substrates were washed with CHCl$_3$ to demonstrate the insolubility of these polymer networks and the polymeric degree. The UV absorption spectra of the Alq_3 polymer and the copolymer network were obtained directly after polymerization and after the films were rinsed with CHCl$_3$ for 30 s. The polymer films showed only a very slight decrease in absorbance (the copolymers were about 16%, and the Alq_3 polymer film was about 11%). The part that washed off was characterized by gel permeation chromatography and proved to be unreactive monomers and oligomers. The linear Alq_3 polymer, Alq_3 monomer, and Liq monomer reported before[16] were readily removed from the substrate by rinsing once with CHCl$_3$, even with poorer solvents. All of the copolymers and the Alq_3 polymer were converted to insoluble polymer films after thermal polymerization, and most of the monomers were covalently incorporated into the network. No significant differences were observed between the crosslinked structures in the series of copolymers in this test.

TABLE I
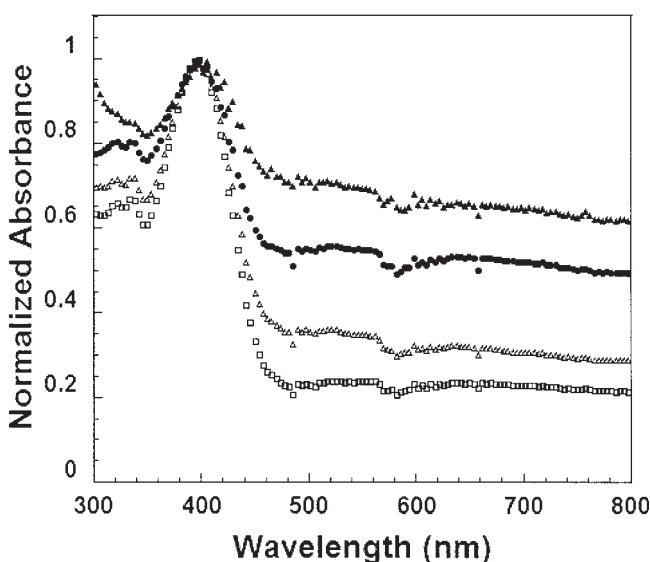**Absorption, Fluorescence, and Thermal Data of the Liq Polymer, Alq$_3$ Polymer, and Copolymers**

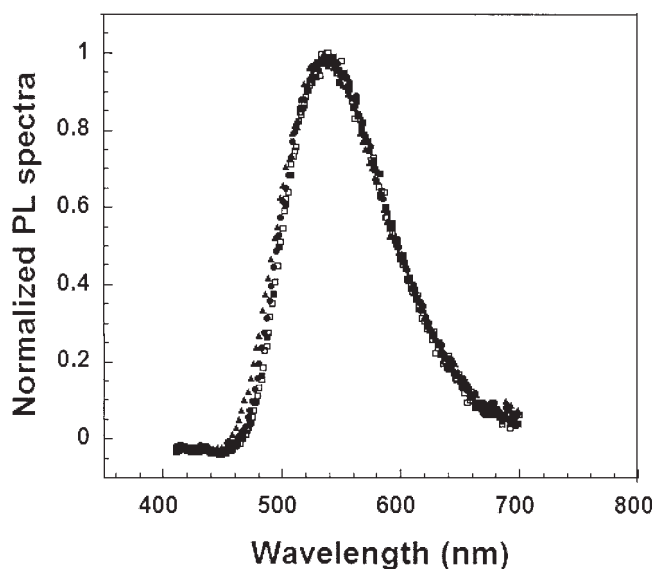| Polymer	Absorbance (nm)	PL (nm)	PL efficiency	T_g at 8% weight loss (°C)
Liq copolymer	366	490	11.82	176
Alq$_3$ polymer	398	547	20.42	200
Copolymer 30	399	539	19.37	193
Copolymer 20	399	540	18.15	182
Copolymer 10	399	536	17.25	179
Copolymer 5	399	536	12.24	174

Optical and thermal properties

The absorption, fluorescence, and thermal properties of the linear Liq copolymer,[14] Alq$_3$ polymer, and copolymers are summarized in Table I.

The thermal properties of the copolymers were studied by DSC and thermogravimetric analysis (TGA). Compared to the linear Liq copolymer synthesized previously[14] [glass-transition temperature (T_g) = 124.64°C], the copolymers had no observable T_g between 25 and 250°C and showed an 8% weight loss from 174 to 198°C (Table I). The thermal stability of the copolymers was enhanced slightly with increasing Alq$_3$ content. The Alq$_3$ polymer showed an 8% weight loss at 200°C. It was more thermally stable because of the mere existence of the Alq$_3$ cross linking structure in the polymers.

As shown in Figure 2, the spectra of the copolymers were quite similar in the spectral range between 350 and 400 nm. The copolymers showed maximum absorptions at about 360 and 398 nm, which were the characteristic absorption bands of Liq and Alq$_3$, respectively; this indicated that the copolymers con-


Figure 3 PL spectra of the copolymers on glass: copolymers (□) 30, (■) 20, (●) 10, and (△) 5.

tained both Alq$_3$ and Liq groups. Compared to the Alq$_3$ polymer, the PL emissions of the copolymers blueshifted slightly with increasing Liq content (Fig. 3). It has been observed that the luminescence of 8-hydroxyquinoline metal chelates can be either blueshifted or redshifted by the addition of substituents or by the introduction of optically inactive spacer molecules into the crystalline network of 8-hydroxyquinoline metal chelates.[17] The PL emission peaks at about 540 nm indicated that the fluorescence centers of the copolymers still came from Alq$_3$. We concluded that the fluorescence energy of Liq was transferred to the Alq$_3$ groups. In our previous experiments, the PL efficiency of the linear Liq copolymer was about 11.82%, which was lower than that of the Alq$_3$ polymer. In the copolymers, when the Liq concentration increased, the PL efficiency of the copolymers also decreased because of the less efficient emitter molecules.

Figure 2 UV spectra of the films of the copolymer on glass: copolymers (□) 30, (■) 20, (●) 10, and (△) 5.
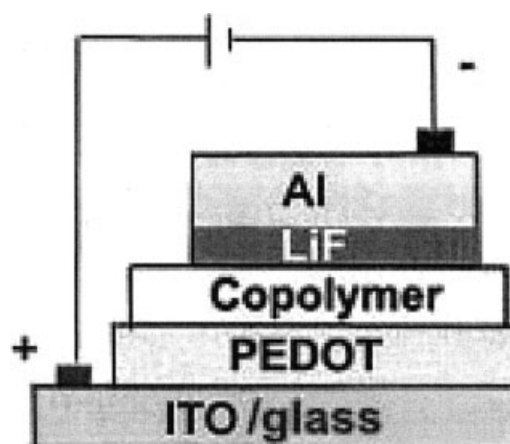
Figure 4 Configuration of the organic EL devices made from the copolymers.

OLEDs with copolymers as emitters

Figure 4 shows the configuration of the organic EL devices made from the copolymers. The bilayer cathode consisted of Al and LiF. The LiF layer was about 2.5 nm and was used as interface material to lower the driving V. PEDOT, used as hole transport layer, was insoluble in the organic solvent, and the monomers could be deposited onto it. The thickness of the emitting layer from the copolymers was 100 nm.

We compared the EL spectra of the copolymers and the Alq_3 polymer. As shown in Figure 5 and Table II, the EL spectrum of the Alq_3 polymer had an emission maximum at 583 nm. The EL emission maxima of the copolymer devices were affected by the Liq concentration and were blueshifted from 557 to 538 nm, even at low Liq concentrations. The characteristic EL emission maximum of Liq (ca. 495 nm[18]) disappeared, even at a high Liq contents. We assumed that both holes and electrons trapped in the Alq_3 groups were waiting for the opposite charge to recombine, and this eventually led to light emission.
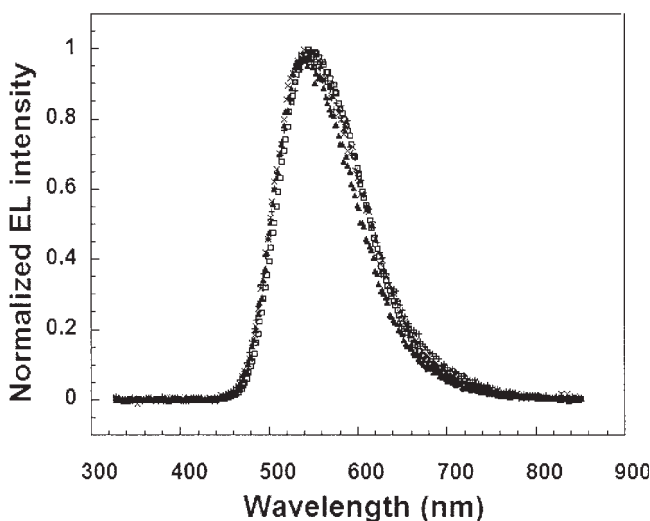
Figure 6 shows the I–V and electroluminance–V characteristics of the devices prepared from the copolymers. The results show that the device performances were clearly changed by the introduction of Liq content. Device performances at maximum external quantum efficiency for the Alq_3 polymer and the copolymers are summarized in Table II. A comparison between the devices from the copolymers and the Alq_3 polymer revealed that the devices with the copolymers had lower threshold Vs for a given luminance and a faster increase in luminance with increasing driving V. The threshold Vs of the devices were also affected with the introduction of even a small amount of Liq in the copolymers. In the series of copolymers, the observed

threshold V changed from 8.4 to 3.84 V. The higher the Liq content was, the lower the threshold V was, whereas the threshold V of the Alq_3 polymer was about 14 V. At a higher concentration of Liq in the copolymers, the threshold V did not decrease so obviously.

Researchers have brought forward different mechanisms responsible for improved electron injection. In the case of LiF, they have usually suggested that the band bending in the organic layer lowers the electron injection barrier height or that the presence of a dipole at the LiF/metal interface shifts the work function of the metal cathode to lower energies.[6,18] However, more and more researchers have now agreed that it is free lithium metal released at the LiF/metal interface and doped into Alq_3 that improves electron injection. It has also been suggested that the Alq_3 anion states produced by the interaction of Alq_3 and liberated Li are responsible for the EL performance improvement.[3(b),19–21] The concept of LiF doping has also been put forward in the case of Liq. Liu's results supported the assumption that free lithium released from lithium quinolate is responsible for improved device performance and that this release is more effective than that observed in

TABLE II
Device Performances at Maximum External Quantum Efficiency for the Alq_3 Polymer and Copolymers

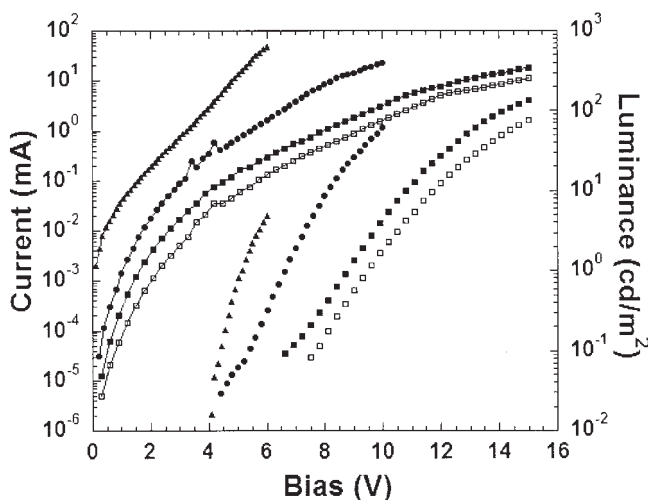|	Device performance				
Active layer	λ_{ELmax} (nm)	V (V)	I (mA)	Luminance (cd m^{-2})	$(QE)_{EL}$ (%)
Alq_3 polymer	583	31.50	13.80	104	0.101
Copolymer 30	549	15.00	11.23	77	0.179
Copolymer 20	538	15.00	17.62	134	0.198
Copolymer 10	538	10.00	22.64	61	0.070
Copolymer 5	538	6.00	47.85	5	0.003

Figure 5 EL spectra of the copolymers of device ITO/PEDOT/copolymers/Liq/Al: copolymers (□) 30, (▲) 20, (×) 10, and (+) 5.

Figure 6 I–V and electroluminance–V curve of the LED devices from the copolymers: copolymers (□) 30, (■) 20, (●) 10, and (▲) 5. The luminance is shown by the corresponding symbols without lines.

devices with LiF.[11] In our copolymers, the Liq content was just like a dopant in the Alq₃ polymer. The difference was that the Liq groups were covalently incorporated into the network structure of the copolymers. The mechanism was similar to the free-lithium-metal-releasing theory.

As shown in Table II, the EL efficiency of the devices was improved only in the copolymers at the proper ratio of Alq₃ to Liq. Improvement in the device performance was possible with a small number of Liq groups. If the concentration of the Liq groups was too high, it exhibited a negative effect because of limited solubility of the Liq monomer and the poor luminescence properties of the Liq groups. This was in agreement with the device characteristics, as shown in Figure 5. Our results for the copolymers show that the best device performance corresponded to copolymer 20 with a Alq₃ : Liq ratio of about 20 : 1. The threshold V was 6.3 V. The maximum luminescence of this device was about 134 cd/m^2, and the EL efficiency was about 0.2%, which was twice as efficient as that of the Alq₃ polymer. With increasing Liq groups in the copolymers, the devices became less efficient because of a decrease in the concentration of Alq₃ groups.

CONCLUSIONS

We synthesized crosslinkable copolymers containing both Alq₃ and Liq groups by thermal polymerization. The network structure and the degree of polymerization of the copolymers were investigated with FTIR spectroscopy and by solvent washing. The result indicated that after thermal polymerization, most monomers were covalently incorporated into the network. The copolymers became more thermally stable with increasing Alq₃ content, as shown by DSC and TGA. The optical properties indicated that Alq₃ and Liq groups both existed in the copolymers and that the luminescence came from Alq₃. The copolymers were used as the emitting layer in double-layer OLEDs consisting of PEDOT as the hole-transport layer and LiF/AI as the double cathode, which resulted in a lower turn on Vs. The performance of the devices was enhanced with copolymers at a Alq₃ : Liq ratio of about 20 : 1. The device was twice as efficient as the device with the Alq₃ polymer. We suggest that the mechanism responsible for the improved electron injection was similar to the free-lithium-metal-releasing theory. This assumption will be the subject of further study.

APPENDIX: Alq₃ MONOMER SYNTHESIS

A mixture of 8-quinolinol, hydrochloric acid, and formaldehyde was added to a 250-mL three-necked flask with a magnetic stirrer, and treated with hydrogen chloride gas for 12 h. The yellow solid was collected on a filter and washed with acetone several times. After drying at 40°C *in vacuo*, 8.41g of the compound **1** was obtained (94% yield, mp 280°C).

The reaction mixture of sodium acetate, *p*-methoxyphenol and 2-hydroxyethylmethacrylate (HEMA) was stirred at 50°C for 0.5 h, then 5 g (0.0217 mole) 5-chloromethyl-8-quinolinol hydrochloride was added to the mixture. The suspension was heated at 80–90°C for 2 h. After cooling, the complex was poured into cool water and dissolved. The solution was neutralized with dilute ammonia. The precipitate was washed with a large amount of water, then collected by filtration and dried to give a gray solid (80.6% yield). After recrystallizations from petroleum ether, a white flocculent solid was obtained (compound **2**, mp 85.5–87°C).

To a 150-mL three-necked flask equipped with a nitrogen inlet and a magnetic stirrer, triethylaluminum in hexane solution (10 mL, 0.01 mole) was added via syringe, and then compound **2** (0.03mole) in anhydrous THF was added dropwise into the solution. After stirring at room temperature for 24 h, the mixture of compounds **2** and **3** and some insoluble aluminum complex was filtered through a G4 funnel to remove any precipitate. The solution was purified by column chromatography (silica gel, toluene : CHCl₃ 1 : 1) and concentrated, then added dropwise to petroleum ether. The precipitate was filtered off and dried at 40°C *in vacuo*. A yellow solid was obtained (compound **3**, 97% yield).

LiOH and compound 2 (molar ratio was 1 : 1) were added to anhydrous dichloromethane. A yellow-white color appeared immediately. The reaction was kept at room temperature for 2 days. The mixture was then poured into petroleum ether to get a white powder. After drying at 40°C *in vacuo* for 24 h, a white solid was obtained (compound **4**, 98% yield, mp 118.5–120°C).

5-Chloromethyl-8-quinolinol hydrochloride (compound 1)

^{1}H-NMR (D₂O): 9.04 (1H, dd, Ph—H), 8.81 (1H, t, Ph—H), 7.90 (1H, m, Ph—H), 7.48 (1H, d, Ph—H), 7.08 (1H, m, Ph—H), 4.85 (2H, s, —CH₂—); FTIR (KBr, cm^{-1}): 3300 (—OH), 1625, 1596 (C—C and pyridine ring), 1550, 1492 (aromatic), 1390 (C—N), 698 (C—Cl). Anal. Calcd for C₁₀H₈NOCl·HCl: C, 52.17; H, 3.91; O, 6.95; N,6.08. Found: C, 52.50; H, 4.02; O, 6.78; N, 5.95.

5-(2-Methacryloylethyloxymethyl)-8-quinolinol (compound 2)

^{1}H-NMR (CDCl₃): 8.78 (1H, m, Ph—H), 8.50 (1H, dd, Ph—H), 7.46 (1H, m, Ph—H), 7.40 (1H, d, Ph—H), 7.08 (1H, m, Ph—H), 6.03 (1H, s, =CH₂), 5 52 (1H, t, =CH₂), 4.87 (2H, S, —CH₂—Ph), 4.27, 3.69 (4H, t, —CH₂—O), 1.88 (3H, s, —CH₃). IR (KBr, cm^{-1}): 3300

(—OH), 2956 (—CH$_3$) 171 8 (C=O), 1633 (C=C), 161 3, 1581 (C—C and pyridine ring), 1506, 1475 (aromatic), 1384, 1319 (C—N). Anal. Calcd for C$_{16}$H$_{17}$NO$_4$: C, 66.90; H, 5.92; O, 22.30; N, 4.87. Found: C, 67.32; H, 6.00; O, 22.17; N, 4.75.

Alq$_3$ monomer: Tris(5-(2-methacryloylethyloxymethyl)-8-hydroxyquinoline) aluminum (compound 3)

^{1}H-NMR (CDCl$_3$): 8.84–6.45 (15H, m, H—Ph), 6.04(3H, d, =CH$_2$), 5.49(3H, t, =CH$_2$), 4.83(6H, t, —CH$_2$—Ph), 4.30(6H, t, —CH$_2$—O), 3.76 (6H, t, —CH$_2$—O), 1.87 (9H, CH$_3$). IR (KBr, cm^{-1}), 2952, 2991(—CH$_3$), 1716 (C=O), 1635 (C=C), 1604, 1581(C—C and pyridine ring), 1506, 1469 (aromatic), 1384, 1328 (C—N) Anal. Calcd for C$_{48}$H$_{48}$N$_3$O$_{12}$Al: C, 65.08; H, 5.46; N, 4.74. Found: C, 65.01; H, 5.41; N, 4.37.

Liq monomer: 5-(2-Methacryloylethyloxymethyl)-8-hydroxyquinoline lithium (compound 4)

^{1}H-NMR (DMSO): 8.47 (1H, d, Ph—H), 8.29 (1H, dd, Ph—H), 7.34 (1H, m, Ph—H), 7.18 (1H, d, Ph—H), 6.45 (1H, d, Ph—H), 5.96 (1H, s, =CH$_2$), 5.65 (1H, t, =CH$_2$), 4.68 (2H, s, —CH$_2$—Ph), 4.21, 3.64 (4H, t, —CH$_2$—O), 3.33 (2H, s, H—O), 1.83 (3H, s, CH$_3$). IR (KBr, cm^{-1}), 3450 (H—O), 2950, 2991 (—CH$_3$), 1712 (C=O), 1631 (C=C), 1594, 1571 (C—C and pyridine ring), 1504, 1469 (aromatic), 1375 (Li—O), 1322 (C—N). Anal. Calcd for C$_{16}$H$_{18}$NO$_5$Li: C, 61.75; H, 5.79 ; N, 4.50. Found: C, 62.40; H, 5.93; N, 4.45.

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