One of the most challenging tasks encountered in developing highly efficient electro-optic (EO) devices is to find a material system that possesses all desirable properties such as large EO coefficients, good thermal and mechanical stability, and low optical loss. In order to meet this stringent requirement, we have developed a series of crosslinkable EO dendrimers using the standardized AJL8-type chromophore as the center core and the furyl- and anthryl-containing dendrons as the periphery. Upon adding a trismaleimide (TMI) dienophile, these dendrimers could be in-situ crosslinked via the Diels–Alder cycloaddition and efficiently poled under a high electric field. Through this dynamic process, the spatially voided and π-electron-rich surrounding of the chromophore core changes into a dense and more aliphatic network, with the dipolar chromophore embedded and aligned inside. The resultant materials exhibit large EO coefficients (63–99 pm V–1 at 1.31 µm), excellent temporal stability (the original r33 values remain unchanged at 100 °C for more than 500 h), and blue-shifted near-IR absorption. With these combined desirable properties, a poled EOD2/TMI film could be processed through multiple lithographic and etching steps to fabricate a racetrack-shaped micro-ring resonator. By coupling this ring resonator with a side-polished optical fiber, a novel broadband electric-field sensor with high sensitivity of 100 mV m–1 at 550 MHz was successfully demonstrated.

1. Introduction

The ability to control the surrounding environment of an electroactive or photoactive moiety is important for tuning its bulk properties.[1–4] On a macromolecular basis, highly branched, monodisperse dendrimers can furnish an unique interior nanoenvironment around their functional cores through the encapsulation of dendritic wedges. In our recent pursuit of finding efficient organic nonlinear optical (NLO) materials, we have demonstrated that nanoscale architectural control of NLO-active chromophores and polymers could be achieved by introducing dendritic moieties on the periphery of the NLO materials.[5,6] This was driven by the necessity of translating large molecular hyperpolarizabilities (µβ) of “push-pull”-type chromophores into high macroscopic electro-optic coefficients (r33) for device applications. The dendrons used in these studies are mostly the first generation Fréchet-type poly(aryl ether) or triazine dendrons. They could be crosslinked at a later stage by using a trismaleimide (TMI) dienophile, these dendrimers could be in-situ crosslinked via the Diels–Alder cycloaddition and efficiently poled under a high electric field. Through this dynamic process, the spatially voided and π-electron-rich surrounding of the chromophore core changes into a dense and more aliphatic network, with the dipolar chromophore embedded and aligned inside. The resultant materials exhibit large EO coefficients (63–99 pm V–1 at 1.31 µm), excellent temporal stability (the original r33 values remain unchanged at 100 °C for more than 500 h), and blue-shifted near-IR absorption. With these combined desirable properties, a poled EOD2/TMI film could be processed through multiple lithographic and etching steps to fabricate a racetrack-shaped micro-ring resonator. By coupling this ring resonator with a side-polished optical fiber, a novel broadband electric-field sensor with high sensitivity of 100 mV m–1 at 550 MHz was successfully demonstrated.

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phile. Through this modified surrounding, it has led to a considerable blue shift of the dendrimer’s main absorption peak, therefore, its near-IR optical attenuation was reduced.[11] In one of the dendrimers (EOD3), two different diene groups could be functionalized on the donor-end and the bridge-center of the chromophore. It was crosslinked later with trismaleimide through a binary processing temperature window (110 and 200 °C) to achieve the optimal processibility and temporal stability. All of these new NLO dendrimers showed large and thermally stable EO activities (63–99 pm V–1 at 1.31 μm, nearly unchanged at 85 or 100 °C after 500 h), excellent processibility and solvent resistance, and large hypsochromic shift (up to 52 nm) of the π-π* charge-transfer absorption peaks. These desirable properties enable the successful fabrication of a novel broadband electric field (E-field) sensor. This E-field sensor was fabricated by coupling a side-polished fiber with microring resonators made of the abovementioned dendrimer. It showed high sensitivity (100 mV m–1) of E-field sensing at 550 MHz.

2. Results and Discussion

2.1. Synthesis of Dendrimers

Dendrons D1-3 were synthesized by first reacting the diene-containing reagents with 3,5-dihydroxylbenzoate (Scheme 1) using the literature reported conditions for esterification or etherification. These intermediates were then hydrolyzed to afford diene-containing dendrons that have a benzoic acid group. The overall yield from these two steps is ranging from 50 to 83 % depending on the different systems. As shown in Scheme 2, dendrimers EOD1-4 were obtained by the carbodiimide-mediated esterification between D1-4 and the dihydroxylated chromophore 11. The reaction mixture was activated by adding 1,3-dicyclohexylcarbodiimide (DCC) and a catalytic amount of 4-(dimethylamino)pyridinium-4-toluene-sulfonate (DPTS). Because of the poor solubility of 11 in dichloromethane, tetrahydrofuran (THF) was added as a co-solvent to enhance the efficiency of the reaction. The hydroxymethyl group on the thiophene ring of compound 11


Scheme 2. Synthesis of Diels–Alder Crosslinkable NLO Dendrimers EOD1-4 (also inserted the structure of dendrimer EOD5 with Fréchet-and trimaleimide dienophile crosslinker TMI).
was successfully condensed with the carboxylic acid on the
diene dendrons without causing the chromophore decomposi-
tion. However, the hydroxyethyl group at the donor-end could
not be reacted under the same condition due to its low reactiv-
ity. This phenomenon was further confirmed through thin layer
chromatography (TLC) and structural characterization of the
isolated chromophoric intermediates, which only contain a
mono-dendron situated on the thiophene ring of the conjugat-
ing bridge. To complete the reaction, another equivalent of a
stronger coupling reagent, 1-(3-(dimethylamino)propyl)-3-
ethyl-carbodiimide hydrochloride (EDC), was added to the re-
action mixture together with 4-(dimethylamino)pyridine
(DMAP). The sequential addition of these reagents minimizes
the time and amount of the very reactive EDC/DMAP used in
this reaction. This not only helps preserve the chemically-sensi-
tive chromophore but also ensures that EOD1, 2, and 4 could
be obtained in good yields. Moreover, the reaction proceeds
without any the need of using protecting or deprotecting steps
for sequential functionalization of two different diene moieties
onto the same chromophore. This process allows us to generate
crosslinkable dendrimers such as EOD3 in one step. Because it
bears two different crosslinking moieties, it can be processed
using a binary temperature condition.

2.2. Thermal Properties and Near-IR Absorption of
Dendrimers

Dendrimers EOD1-4 possess much improved solubility in
common organic solvents such as THF, chloroform, 1,1,2-tri-
chloroethane (TCE), and cyclopentanone compared to the diol
of chromophore 11. These amorphous solids possess clear glass
transition temperatures as shown in their thermo-diagrams
measured by differential scanning calorimetry (DSC) (Fig. 1).
Their decomposition temperatures are defined as the onset
temperature points where the large exothermic peaks arise in
the DSC curves and this was further confirmed by thermo-
gravimetric analysis (TGA). It is apparent that each dendrimer
possesses quite different glass transition and decomposition
temperatures. This is a clear indication that different dendrons
can offer substantially different surrounding environments to
the central chromophore.

Thin films with good optical quality could be obtained by
spin-coating EOD1-4, from their TCE or cyclopentanone solu-
tions onto glass substrates. These thin films showed quite dif-
ferent absorption maxima ($\lambda_{\text{max}}$) (Fig. 2), which indicate that
the environment of the core chromophore was influenced sig-
ificantly by its surrounding dendrons.

Because EOD1-4 molecules have a diene moiety, they can
react with TMI, a dienophile crosslinker via the Diels–Alder
cycloaddition. The temperature window for processing these
dendrimers could be controlled by tuning the electronic prop-
ties of the diene branches.[12] In addition, the lattice-harden-

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Figure 1. Left: Thermal analysis for EOD1-4 by differential scanning calorimetry (DSC) at 10 °C min–1 under nitrogen; Right: Thermal analysis of EOD2
with and without the addition of trimaleimide, before and after curing.

Figure 2. UV-visible absorption spectra of EOD1-4 films. All these thin
films were baked at 50 °C overnight in a vacuum oven after spin-coating.
The thickness of the films was controlled to be around 0.2–0.3 µm. Note
that these spectra are normalized to their peaks.
ing of EOD1-4 also converts the π-electron-rich dendrons into rigidified cyclic structures with much less π electrons. This leads to a less polarizable environment. As a result, a considerable blue shift could be observed in their absorption spectra. This phenomenon is very similar to the strong solvatochromism observed for AJL8 chromophore in different solvents (Fig. 3).

Compared to the λmax for EOD4 (776 nm), the λmax of the blend system (EOD4 and TMI) blue-shifted 40 nm to 736 nm even before the thermal curing. This is attributed to the partial reaction between the reactive anthryl and maleimido groups. By baking at 50 °C under vacuum overnight, the λmax of the resultant chromophore further blue-shifted to 724 nm (Fig. 4). This phenomenon is worth further explore because it offers a promising way to decrease the attenuation of EO dendrimers in the near-IR region.

2.3. Poling and EO Properties of Dendrimers

To study the EO property, a dendrimer solution (7.0 wt % solid content) with same equivalent of TMI in TCE was filtered through a 0.2 μm PTFE-syringe filter and spin-coated onto indium tin oxide (ITO) glass substrates. Micron-thick thin films could be easily obtained by a single spin-coating. The resulting films were baked in vacuum at 50 °C overnight to ensure the removal of any residual solvent. Then, a thin layer of gold was sputtered onto the films as a top electrode for contact poling. The r33 values were measured using the Teng-Man simple reflection technique at the wavelength of 1.31 μm.[13] Although a baking temperature of 50 °C is well below the Tg of the dendrimers, all of the films pre-reacted to a certain extent during this step because of highly reactive nature of the dienes and TMI. This reaction actually increases molecular weight and dielectric strength of the materials and lead to films with moderate solubility in organic solvents such as acetone. Moreover, the interchain Diels–Alder adducts can be thermally dissociated through the retro-Diels–Alder reaction. This can be tapped to further enhance the alignment of chromophores during poling. With all these dynamics encountered, the optimal poling temperature is strongly dependent on proper balance between their Tg's (after pre-crosslinking), Tdiss (dissociation), and Tx (crosslinking) to accomplish high poling efficiency. High temperature and high voltage poling could also be applied concurrently to increase lattice hardening.

Upon the removal of electric field and cooling to room temperature, the poled films of EOD1-3 showed large EO coefficients (r33 = 63–99 pm V−1 at 1.31 μm) (Table 1) which are much higher than that from the guest-host EO polymer (AJL8/APC) or the uncrosslinked EOD5 which contains the similar chromophore. Over 90 % of the original EO activities (EOD1/TMI with an r33 of 99 pm V−1 stayed nearly unchanged) could be retained at 100 °C for more than 500 h (Fig. 5).

These poled films also showed very good solvent resistance toward THF, TCE, and acetone. The combined dendritic site-isolation and well-balanced processing protocols for pre-crosslinking, poling, and lattice hardening contribute to these desirable properties.
2.4. Demonstration of Broadband Electric-Field Sensor by EOD2/TMI

The combined high EO activity and excellent solvent resistance of these EO dendrimers facilitate the fabrication of a novel EO electric field (E-field) sensor. These devices were fabricated by coupling a side-polished optical fiber with micro-ring resonators made of the EO dendrimer (i.e., EOD2/TMI) (Fig. 6). The complete description of this device is reported elsewhere.[14] The device was fabricated using a crosslinked dendrimer (EOD2/TMI) that possesses both good temporal stability and solvent resistance. Quite amazingly, it can tolerate all the chemicals and solvents used for waveguide fabrication (such as AZ1512 positive resist, AZ352 solution, AZ1512 developer, SU-8 electron beam resist, and SU-8 developer). The poled EOD2/TMI film can also be patterned with electron-beam lithography and reactive ion etching (RIE) to obtain a racetrack-shape ring resonator. This is the first example of dendrimer-based EO device that can meet the stringent fabrication challenges and achieve superior performance to the devices made from its polymer counterparts. The operation of this E-field sensor is based on evanescently coupling light from an optical fiber into the resonator to generate resonant modes for certain wavelengths and produce notches in the output intensity (using a single wavelength positioned at the slope of a resonance) or the transmission spectrum (in the wavelength domain).

By using the poled EOD2/TMI, a sensitivity of 100 mV m⁻¹ could be achieved at frequencies up to 550 MHz (limit of the measurement system) in a proof-of-concept device. This all-dielectric EO sensor possesses high sensitivity, low invasiveness, and high bandwidth that can be used for applications in the semiconductor industry.

3. Conclusion

A series of highly efficient, crosslinkable EO dendrimers have been synthesized by functionalizing a standardized AJL8-type chromophore with diene-containing dendrons and a carboxylic acid substituted anthryl moiety. By introducing trismaleimide (TMI) dienophile, these dendrimers could be efficiently in-situ crosslinked and poled under high electric field. Under this dynamic process, the spatially voided and maleimide-substituted chromophore core changed into a denser and more aliphatic network with the dipolar chromophore embedded and aligned inside. The resultant materials exhibit large EO coefficients (63–99 pm V⁻¹ at 1.31 μm), excellent long-term stability ($r_{33}$ values nearly unchanged at 85 °C for over 500 h), and a blue-shifted near-IR absorption. By taking advantage of these combined properties, a poled EOD2/TMI film can be processed through stringent lithographic and etching steps to fabricate a racetrack-shaped micro-ring resonator. By coupling this micro-ring resonator with a side-polished optical fiber, we have demonstrated a novel broadband electric-field sensor with high sensitivity of 100 mV m⁻¹ at 550 MHz. This all-dielectric EO sensor possesses high sensitivity, low invasiveness, and high bandwidth that can be used for applications in the semiconductor industry.
4. Experimental

**General Methods:** 'H and 13C NMR spectra were recorded on Bruker AV-300 and AV-500 spectrometers, respectively. Absorption spectra were obtained on a Perkin-Elmer Lambda-9 spectrophotometer and ESI-MS spectra were recorded on a Bruker Daltonics Esquire ion trap mass spectrometer. Glass transition temperatures (T_g) were measured by differential scanning calorimetry (DSC) using a DSC2010 in TA instruments with a heating rate of 10°C/min.

**Materials:** Dichloromethane (CH2Cl2) and tetrahydrofuran (THF) were distilled over phosphorus pentoxide and sodium benzophenone ketyl, respectively, under nitrogen prior to use. Chromophore 11 [15], EOD5 [16], and TMI [17] were prepared according to the methods described in the literature. All other chemicals were purchased from Aldrich and were used without further purification. All reactions were carried out under an inert atmosphere unless otherwise specified.

**Preparation of Compound 3:** The solution of methyl 3,5-dihydroxybenzoate (compound 1, 5.1 g, 30 mmol), diethyl azocarboxylate (62 mL, 133 mmol) and triphenylphosphine (36.8 g, 133 mmol) in freshly distilled THF (60 mL) was cooled to 0 °C followed by dropwise addition of dicyclohexylcarbodiimide (DCC, 0.09 g, 0.53 mmol) in dry THF (12 mL) at room temperature under an inert atmosphere. The reaction mixture was warmed up to room temperature slowly and freshly distilled THF (60 mL) was added 3-furoic acid (compound 2, 8.39 (d, J = 7.8 Hz, 4H), 7.61 (s, 2H), 7.58–7.49 (m, 8H), 7.10 (s, 1H), 6.02 (s, 4H), 3.97 (s, 3H). **Preparation of Diene dendron D3:** The solution of compound 8 (2.36 g, 4.3 mmol), potassium carbonate (1.38 g, 10 mmol) and MeOH/H2O (15 mL + 5 mL) was kept stirring at room temperature overnight, and removed MeOH under reduced pressure. The reminder was dissolved with CH2Cl2, and the organic phase was washed with HCl (2 M), brine, and dried over Na2SO4. The solvent was evaporated under the reduced pressure. The residue was distilled over phosphorus pentoxide and sodium benzophenone ketyl, respectively, under nitrogen prior to use.

**Preparation of Compound 4:** A mixture of 9-anthracenemethanol (compound 9, 1.04 g, 5.0 mmol), succinic anhydride (compound 10, 2.0 g, 20.0 mmol), and 4-dimethylamino-pyridine (4-DMAP, 0.61 g, 5.0 mmol) was placed in a 100 mL round-bottom flask and dissolved in 50 mL anhydrous CH2Cl2. To this solution was added anhydrous pyridine (2.0 mL, 25.0 mmol) at room temperature and the reaction was stirred for 16 h. The reaction mixture was poured into an ice/water mixture, and warmed to room temperature. The organic layer was separated and the aqueous layer was washed with CH2Cl2 (2 × 50 mL). The combined organic layers were washed with 5% HCl, NaHCO3, brine, dried over MgSO4, filtered and concentrated in vacuo to afford pure D4 (1.42 g, 4.85 mmol, yield, 97%) as a yellow solid. Mp 112–113 °C. **Preparation of EO Dendrimer EOD1:** The solution of compound 11 (0.30 g, 0.176 mmol). D1 (0.17 g, 0.53 mmol), and DPTS (0.02 g, 0.0206 mmol) in THF (15 mL) was kept stirring overnight after adding dicarboxylosorbodiimide (DCC, 0.036 g, 0.176 mmol) slowly. 1-(di-methylamino)propyl-3-ethyldiaminobis hydrochloride (EDC, 0.033 g, 0.176 mmol) and 4-dimethylamino-pyridine (DMAP, 0.01 g, 0.088 mmol) was added into the reaction solution successively and the solution was kept stirring for another 12 h. After filtration of the resultant urea carefully, all the solvent was evaporated under the reduced pressure. The crude product was purified by column chromatography using ethyl acetate and hexane (1:6, v/v) as the eluent to afford EOD1 as a deep green solid (0.14 g, 0.12 mmol, 68%). **Preparation of EO Dendrimer EOD2:** The solution of compound 12 (0.25 g, 0.176 mmol), D1 (0.17 g, 0.53 mmol), and DPTS (0.02 g, 0.0206 mmol) in THF (15 mL) was kept stirring overnight after adding dicarboxylosorbodiimide (DCC, 0.036 g, 0.176 mmol) slowly. 1-(di-methylamino)propyl-3-ethyldiaminobis hydrochloride (EDC, 0.033 g, 0.176 mmol) and 4-dimethylamino-pyridine (DMAP, 0.01 g, 0.088 mmol) was added into the reaction solution successively and the solution was kept stirring for another 12 h. After filtration of the resultant urea carefully, all the solvent was evaporated under the reduced pressure. The crude product was purified by column chromatography using ethyl acetate and hexane (1:6, v/v) as the eluent to afford EOD1 as a deep green solid (0.14 g, 0.12 mmol, 68%). 1H NMR (CDCl3, TMS, δ ppm): 8.21 (s, 7H), 8.19 (s, 2H), 8.10 (d, J = 15.3 Hz, 1H), 7.82 (d, J = 2.4 Hz, 2H), 7.77 (d, J = 7.8 Hz, 2H), 7.57–7.52 (m, 4H), 6.75 (m, 4H), 6.56 (t, J = 2.1 Hz, 2H).
Preparation of EO Dendrimer EO D3: To a solution of compound 11 (0.1 g, 0.176 mmol), D1 (0.055 g, 0.176 mmol), and DPTS (0.02 g, 0.0206 mmol) in THF (15 mL) was added dicyclohexylcarbodiimide (DCC, 0.036 g, 0.176 mmol). The reaction mixture was allowed to stir at room temperature for 12 h, and then 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (EDC, 0.051 g, 0.264 mmol) and 4-dimethylamino-pyridine (DMAP, 0.01 g, 0.088 mmol) and D4 (0.085 g, 0.264 mmol) was added into the reaction solution successively, and the reaction solution was kept stirring for another 12 h. After filtration of the resultant urea carefully, all of the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography using ethyl acetate and hexane (1:5, v/v) as an eluent to afford EO D3 (0.107 g, 0.091 mmol, 52 %) as deep green solid. 

1H NMR (CDCl3, TMS, δ ppm): 8.51 (s, 1H), 8.37 (d, J = 9.0 Hz, 2H), 8.05 (d, J = 8.4 Hz, 3H), 7.59–7.39 (overlap, 8H), 7.32 (d, J = 2.4 Hz, 2H), 7.19 (d, J = 3.9 Hz, 1H), 6.82 (s, 1H), 6.65 (d, J = 8.7 Hz, 2H), 6.58 (d, J = 15.6 Hz, 1H), 6.43 (d, J = 3.0 Hz, 2H), 6.38 (d, J = 1.8 Hz, 4H), 6.20 (s, 2H), 5.40 (s, 2H), 4.99 (s, 4H), 4.14 (t, J = 6.9 Hz, 2H), 3.42 (overlap, 4H), 2.64 (overlap, 4H), 1.95 (s, 3H), 0.94 (t, J = 7.8 Hz, 3H). 

13C NMR (CDCl3, TMS, δ ppm): 176.64, 172.91, 172.78, 166.20, 163.78, 160.59, 154.15, 151.10, 150.11, 144.47, 144.37, 142.11, 141.99, 137.45, 137.26, 136.79, 132.91, 132.47, 132.43, 131.98, 131.95, 130.53, 130.00, 127.69, 125.79, 125.15, 125.62, 125.07, 125.02, 124.75, 114.71, 112.88, 112.77, 112.19, 111.62, 111.54, 111.47, 111.36, 109.42, 107.76, 63.14, 62.43, 60.36, 59.56, 59.40, 49.21, 45.85, 18.94, 12.58. ESI-MS (m/z): Calcd: 1152.3; Found: 1152.3.

Preparation of EO Dendrimer EO D4: The solution of compound 11 (0.14 g, 0.25 mmol), D3 (0.30 g, 0.56 mmol), and DPTS (0.02 g, 0.0206 mmol) in THF (15 mL) was kept stirring at room temperature overnight after addition of dicyclohexylcarbodiimide (DCC, 0.058 g, 0.28 mmol) slowly 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (EDC, 0.056 g, 0.264 mmol) and 4-dimethylamino-pyridine (DMAP, 0.02 g, 0.176 mmol) was added into the reaction solution successively, and the solution was kept stirring for another 12 h. After filtering of the resultant urea carefully, all of the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography using ethyl acetate and hexane (1:5, v/v) as the eluent to afford EO D4 (0.09 g, 0.18 mmol, 72 %) as a deep green solid. 

1H NMR (CDCl3, TMS, δ ppm): 8.51 (s, 2H), 8.44 (s, 2H), 8.22 (d, J = 8.7 Hz, 8H), 8.08–7.98 (overlap, 9H), 7.61–7.45 (overlap, 26H), 7.31 (d, J = 9.0 Hz, 2H), 7.08 (d, J = 21.6 Hz, 2H), 6.80 (d, J = 15.5 Hz, 1H), 6.71 (d, J = 8.5 Hz, 2H), 6.65 (d, J = 15.5 Hz, 1H), 5.92 (s, 4H), 5.83 (s, 4H), 5.10 (s, 2H), 4.43 (t, 2H), 3.58 (t, 2H), 3.36 (m, 2H), 1.92 (s, 3H), 0.54 (t, J = 6.9 Hz, 3H). 

13C NMR: 175.17, 166.51, 166.07, 162.17, 160.60, 160.53, 154.13, 149.44, 140.92, 140.74, 136.49, 136.39, 135.23, 132.05, 131.94, 131.60, 131.59, 131.15, 131.13, 129.68, 129.41, 129.34, 126.89, 126.87, 126.47, 126.28, 125.29, 125.26, 123.97, 123.91, 123.52, 123.01, 113.01, 112.11, 111.20, 110.98, 110.67, 108.83, 108.58, 107.94, 107.45, 97.31, 63.21, 62.64, 59.26, 58.78, 55.52, 48.09, 44.79, 29.91, 19.37, 12.27. ESI-MS (m/z): Calcd: 1598.5; Found: 1599.6.
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