

Chain Length Effect of Dialkoxynaphthalene End-Capped Divinylbenzene for OTFT

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The new organic semiconductors which are composed of divinylbenzene core unit and alkoxy-naphthalene on both sides, 1,4-bis-2-(6-octyloxy)naphthalen-2-ylvinylbenzene (BONVB), 1,4-bis-2-(6-decyloxy)naphthalen-2-ylvinylbenzene (BDNVB) and 1,4-bis-2-(6-dodecyloxy)naphthalen-2-ylvinylbenzene (BDDNVB) were synthesized by Wittig reaction. The structures of obtained BONVB, BDNVB and BDDNVB were confirmed by FT-IR and mass spectroscopy. UV-absorption of thin film showed H-aggregates and J-aggregates due to closely packed structure between adjacent molecules. The characterization of vacuum-evaporated films by X-ray diffraction (XRD) and atomic force microscopy (AFM) showed that the chain length of alkoxy group affects the crystallinity and morphology. BONVB with octyloxy group showed the mobility of $0.011 \text{ cm}^2/\text{V}\cdot\text{s}$, on/off ratio of 1.31×10^5 , and a subthreshold slope of 0.93 V .

Key Words : Organic semiconductor, TFT (thin film transistor), Hole mobility, Chain length

Introduction

Organic thin film transistors (OTFTs) based on oligomers and polymers have recently received considerable attention because of their fundamental optoelectronic properties and potential applications for organic integrated circuit sensors,¹⁻⁴ low-cost memories, smart cards, and driving circuits for large-area display device applications such as active-matrix flat-panel liquid-crystal display (AMFPDs), organic light-emitting diodes, and electronic paper displays.⁵⁻¹⁷ The field-effect mobility of OTFTs is gradually increased as well as the advantages of cheap manufacturing and simple processing make them suitable for commercial applications.

Oligomers are of particular interest because they generally possess good molecular ordering properties and can be easily purified. A large number of oligomers have demonstrated interesting performances in OTFTs, such as fluorene, acenes and carbazole derivatives.¹⁸⁻²¹ The synthesis of new π -conjugated units that could be incorporated in oligomeric structures could be desirable for significant optimization of OTFT performances and for a better understanding of the structure-property relationship.²²⁻²⁷

For low-cost manufacturing, the processing should be carried out in ambient conditions, which requires the materials to be stable with respect to ambient environmental effects.²⁸ Recently, several groups have reported significant enhancements of the stability of OTFTs under ambient conditions with semiconducting small molecules,²⁹⁻³³ oligomers,^{22,24,34-39} and polymers.

Recently, our group reported anthracene main core with dialkoxynaphthalene end-capper had good oxidation stability and exhibited a field-effect performance with a mobility of $0.64 \text{ cm}^2/\text{V}\cdot\text{s}$, a subthreshold slope of 0.8 V and an on/off

ratio of 7.5×10^5 .²⁴

In the present study, divinylbenzene with dialkoxynaphthalene end-capper was synthesized and characterized to create a high performance semiconducting material with high oxidation stability. We also studied the dependence of alkoxy chain length to photophysical, the film morphology, crystallinity and OTFT performance.

Experiment

Measurements. The FT-IR spectra, gas chromatography (GC) mass spectrometry, and ¹H-NMR spectra were recorded on a Bruker IFS66 spectrometer, a JEOL GC Mate2, and a Bruker Avance 300-MHz NMR spectrometer, respectively. UV-visible absorption and photoluminescence (PL) spectra were obtained using a Perkin Elmer LAMBDA-900 UV-visible spectrometer and a Perkin Elmer LS50B luminescence spectrophotometer, respectively. Cyclic voltammograms were recorded using Epsilon E3 at room temperature in a 0.1 M solution of tetrabutylammonium perchlorate (Bu₄NClO₄) in acetonitrile under nitrogen gas protection at a scan rate of 50 mV/s. A Pt wire was used as a counter-electrode and an Ag/AgNO₃ electrode was used as the reference electrode. A thermogravimetric analysis (TGA) was performed under nitrogen using TA Instruments 2050 thermogravimetric analyzer. The sample was heated at a 10 °C/min heating rate from 30 °C to 700 °C. Differential scanning calorimetry (DSC) was conducted under nitrogen on a TA Instruments 2100 differential scanning calorimeter. The sample was heated at 10 °C/min from 30 °C to 400 °C.

The film microstructure and morphology of a vapor-deposited thin film of BONVB, BDNVB and BDDNVB, fabricated at a substrate temperature of 90 °C, grown on

SiO₂/Si were investigated by x-ray diffraction (XRD) and atomic force microscopy (AFM).

For the fabrication of the OTFTs, a highly doped *p*-Si wafer with a 300 nm thermally grown oxide layer was used as a substrate. The wafer served as a gate electrode and the oxide layer acted as a gate insulator. Prior to the surface treatment of the silicon oxide layer, the wafer was cleaned with deionized (DI) water, acetone, and isopropyl alcohol (IPA). As an organic interlayer material between the organic active material and the dielectric layer, 1,1,1,3,3,3-hexamethyldisilazane (HMDS, Aldrich) was applied to the SiO₂ using a dipping method to improve the field-effect mobility of the OTFTs by enhancing the quality of the organic active material/dielectric interface. After a HMDS treatment, BONVB, BDNVB and BDDNVB were deposited onto the substrates at a thickness of 60 nm under a pressure of 3×10^{-6} Torr at a rate of 6 Å/s. Gold (50 nm) was used for the source and drain electrodes and deposited on the organic semiconductor layer through a shadow mask with a channel width (W) of 1000 μm and a channel length (L) of 50 μm. The device was fabricated at a substrate temperature (*T*_{sub}) of 90 °C.

Materials. 6-Bromo-2-naphthanol, 1-bromooctane, 1-bromodecane, 1-bromododecane, *n*-Butyllithium, 1-formylpiperidine, α,α'-dibromo-*p*-xylene, triphenylphosphine, were purchased from Aldrich Co. and they were used as received. All the solvents were dried over sodium/benzophenone ketyl and freshly distilled prior to use. The other chemical reagents were common commercial level and used as received.

Synthesis of 2-Bromo-6-octyloxy-naphthalene (1). The 6-bromo-2-naphthanol (10 g, 44.8 mmol), 1-bromooctane (9.1 g, 47.1 mmol), KOH (3.27 g, 58.3 mmol) and NaI (0.67 g, 4.5 mmol) were mixed in ethanol (100 mL) and refluxed for 24 h. After refluxing, the flask was cooled to the room temperature, and removed ethanol using rotary evaporator. The mixture was stirred with MgSO₄ for drying, and filtered. The crude product was purified by column chromatography by hexane as eluent and recrystallized from methanol. Yield: 7.75 g (44.6%); ¹H-NMR (300 MHz, CDCl₃, ppm) δ 7.91 (d, 1H), 7.65-7.67 (d, 1H), 7.58-7.60 (d, 1H), 7.49-7.52 (dd, 1H), 7.16-7.18 (dd, 1H), 7.1 (d, 1H), 4.05-4.08 (t, 2H), 1.84-1.87 (m, 2H), 1.48-1.55 (m, 2H), 1.28-1.38 (m, 8H), 0.86-0.91 (t, 3H).

Synthesis of 2-Bromo-6-decyloxy-naphthalene (2). The procedure used is the same as that for preparation of compound (1). Yield: 6.01 g (37.3%); ¹H-NMR (300 MHz, CDCl₃, ppm) δ 7.91 (d, 1H), 7.64-7.66 (d, 1H), 7.59-7.60 (d, 1H), 7.49-7.52 (dd, 1H), 7.16-7.2 (dd, 1H), 7.11 (d, 1H), 4.05-4.08 (t, 2H), 1.82-1.85 (m, 2H), 1.49-1.55 (m, 2H), 1.28-1.38 (m, 12H), 0.88-0.92 (t, 3H).

Synthesis of 2-Bromo-6-dodecyloxy-naphthalene (3). The procedure used is the same as that for preparation of compound (1). Yield: 7.24 g (41.3%); ¹H-NMR (300 MHz, CDCl₃, ppm) δ 7.92 (d, 1H), 7.64-7.67 (d, 1H), 7.58-7.61 (d, 1H), 7.48-7.52 (dd, 1H), 7.16-7.19 (dd, 1H), 7.1 (d, 1H), 4.05-4.07 (t, 2H), 1.83-1.86 (m, 2H), 1.48-1.54 (m, 2H), 1.28-1.38 (m, 12H), 0.88-0.92 (t, 3H).

Synthesis of 6-Octyloxy-2-naphthaldehyde (4). *n*-Butyllithium (7.16 mL, 17.9 mmol) was added dropwise to a solution of 2-bromo-6-octyloxy-naphthalene (5 g, 14.9 mmol) in 75 mL of THF at -78 °C. After 1 h upon addition, 1-formylpiperidine (2.2 g, 19.5 mmol) was added to the solution and stirred for 3 h at room temperature. Finally, aqueous solution of NH₄Cl (20%) was added to quench the reaction. The product was extracted with diethyl ether, and then dried with MgSO₄. After the solvent was evaporated, the crude product was purified by recrystallized from hexane. Yield: 3.18 g (75%); ¹H-NMR (300 MHz, CDCl₃, ppm) δ 10.11 (s, 1H), 8.27 (d, 1H), 7.95-7.89 (t, 2H), 7.82-7.79 (d, 1H), 7.27-7.23 (dd, 1H), 7.19-7.18 (d, 1H), 4.15-4.10 (t, 2H), 1.91-1.84 (m, 2H), 1.53-1.50 (m, 2H), 1.29 (m, 8H), 0.92-0.88 (t, 3H); FT-IR (KBr) (cm⁻¹): 2919 (aromatic C-H), 2845 (aldehyde C-H), 1695 (C=O), 1633 (aromatic C=C), 1476 (CH₂), 1382 (CH₃).

Synthesis of 6-Decyloxy-2-naphthaldehyde (5). The procedure used is the same as that for preparation of compound (4). Yield: 3.42 (73.4%); ¹H-NMR (300 MHz, CDCl₃, ppm) δ 10.11 (s, 1H), 8.27 (d, 1H), 7.95-7.89 (t, 2H), 7.82-7.79 (d, 1H), 7.27-7.23 (dd, 1H), 7.19-7.18 (d, 1H), 4.15-4.10 (t, 2H), 1.91-1.84 (m, 2H), 1.53-1.50 (m, 2H), 1.29 (m, 12H), 0.92-0.88 (t, 3H); FT-IR (KBr) (cm⁻¹): 2920 (aromatic C-H), 2851 (aldehyde C-H), 1695 (C=O), 1629 (aromatic C=C), 1477 (CH₂), 1383 (CH₃).

Synthesis of 6-Dodecyloxy-2-naphthaldehyde (6). The procedure used is the same as that for preparation of compound (4). Yield: 3.66 g (84.14%); ¹H-NMR (300 MHz, CDCl₃, ppm) δ 10.11 (s, 1H), 8.27 (d, 1H), 7.95-7.89 (t, 2H), 7.82-7.79 (d, 1H), 7.27-7.23 (dd, 2H), 7.19-7.18 (d, 1H), 4.15-4.10 (t, 2H), 1.91-1.84 (m, 2H), 1.53-1.50 (m, 2H), 1.29 (m, 16H), 0.92-0.88 (t, 3H); FT-IR (KBr) (cm⁻¹): 2915 (aromatic C-H), 2845 (aldehyde C-H), 1694 (C=O), 1623 (aromatic C=C), 1474 (CH₂), 1382 (CH₃).

Synthesis of (1,4-Phenylenebis(methylene))bis(triphenylphosphonium)bromide (7). A mixture of α,α'-dibromo-*p*-xylene (10 g, 37.9 mmol) and triphenylphosphine (20 g, 75.8 mmol) in DMF (30 mL) was stirred for 12 h in an oil bath at 160 °C. The mixture was cooled to room temperature and was filtered. The precipitation was recrystallized from diethyl ether to obtain the product of a white solid. Yield: 15.53 g (52%); ¹H-NMR (300 MHz, CDCl₃, ppm) δ 7.80-7.77 (t, 6H), 7.72-7.23 (m, 24H), 6.92 (s, 4H), 1.67 (s, 4H).

Synthesis of 1,4-Bis-2-(6-octyloxy)naphthalen-2-ylvinylbenzene (BONVB). A mixture of (1,4-phenylenebis(methylene)) bis(triphenylphosphonium) bromide (7) (2.05 g, 2.6 mmol) and NaH (0.62 g, 26 mmol) in toluene (50 mL) was stirred for 4 h at 120 °C. The mixture was cooled to room temperature and 6-octyloxy-2-naphthaldehyde (4) (2 g, 7 mmol) was added. This mixture was stirred for 12 h at 120 °C and quenched in 2 N HCl. The compound was filtered and washed with H₂O, ethanol, and methylene chloride. The pure product was obtained as soxhlet with chlorobenzene in the form of a powder. Yield: 0.75 g (50%); EI-MS: *m/z* = 638 (M⁺); FT-IR (KBr) (cm⁻¹): 2923-2851 (aromatic, vinyl C-H), 1602 (vinyl C=C), 1474 (aromatic

C=C), 1383 (CH₂), 1247 (CH₃). Anal. Calcd. for C₄₆H₅₄O₂: C, 86.47; H, 8.52. Found: C, 86.45; H, 8.58.

Synthesis of 1,4-Bis-2-(6-decyloxy)naphthalen-2-ylvinylbenzene (BDNVB). The procedure used is the same as that for preparation of compound BONVB. Yield: 0.72 g (48.6%); EI-MS: m/z = 694 (M⁺); FT-IR (KBr) (cm⁻¹): 2915-2851 (aromatic, vinyl C-H), 1595 (vinyl C=C), 1468 (aromatic C=C), 1382 (CH₂), 1247 (CH₃). Anal. Calcd. for C₅₀H₆₂O₂: C, 86.40; H, 8.99. Found: C, 86.35; H, 9.01.

Synthesis of 1,4-Bis-2-(6-dodecyloxy)naphthalen-2-ylvinylbenzene (BDDNVB). The procedure used is the same as that for preparation of compound BDDNVB. Yield: 0.81 g (55.1%); EI-MS: m/z = 750 (M⁺); FT-IR (KBr) (cm⁻¹): 2915-2851 (aromatic, vinyl C-H), 1623-1620 (vinyl C=C), 1468 (aromatic C=C), 1381 (CH₂), 1247 (CH₃). Anal. Calcd. for C₅₄H₇₀O₂: C, 86.35; H, 9.39. Found: C, 86.27; H, 9.32.

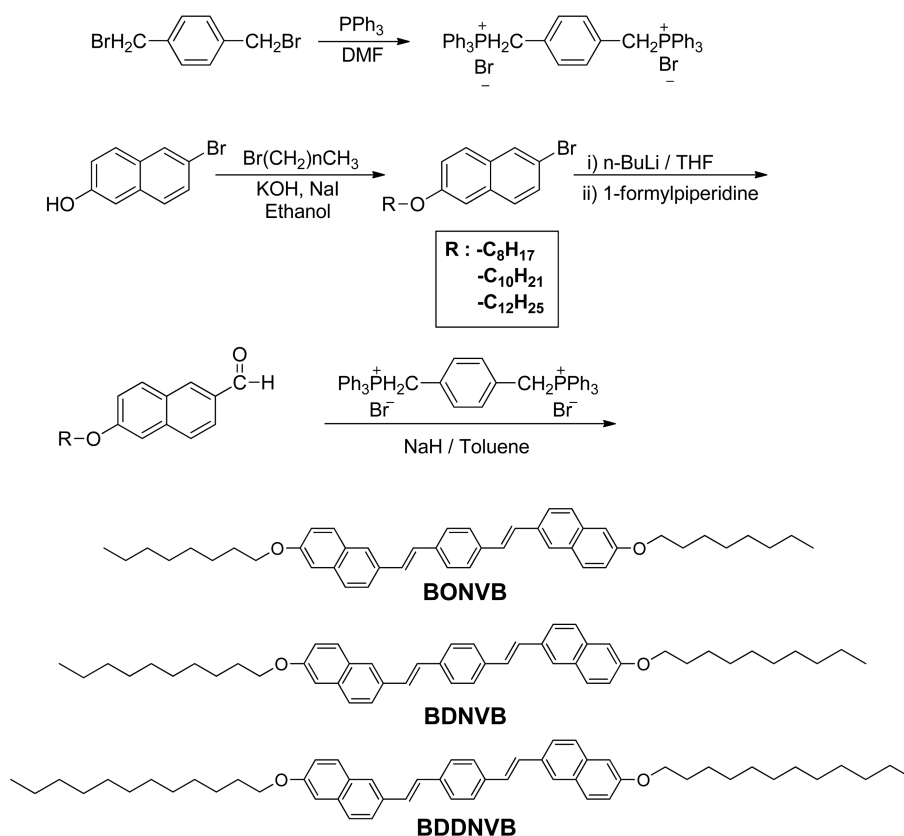
Results and Discussion

The methods used to prepare dialkoxynaphthalene end capped divinylbenzene are depicted in Scheme 1. The obtain-

ed materials were synthesized through alkylation, formylation and Wittig reaction. The obtained materials were purified by soxhlet following recrystallization. Further purification was carried out twice by sublimation under a vacuum. The obtained BONVB, BDNVB and BDDNVB were confirmed by Mass, IR and elemental analysis.

The UV-vis absorption and photoluminescence (PL) properties of the obtained materials in CHCl₃ solution and the thin films are summarized in Table 1. The thin films of BONVB, BDNVB and BDDNVB show the 20-30 nm blue shifted main absorption peaks and 30-40 nm red shifted shoulder peaks. These phenomena may explain by H-aggregates or J-aggregates, which are usually related to excitonic coupling between adjacent molecules in a closely packed structure.⁴¹ In the PL spectra, the thin films of all compounds showed a large degree of red-shift relative to their solution spectra. The results may also originate from the aggregation or excimer formation due to $\pi \rightarrow \pi^*$ stacking or intermolecular interaction caused by their planar structures (Figure 1).

The thermal properties of BONVB, BDNVB and BDDNVB were evaluated by means of thermogravimetric analysis



Scheme 1. Synthetic scheme of BONVB, BDNVB and BDDNVB.

Table 1. Photophysical and electrochemical properties of BONVB, BDNVB and BDDNVB

	λ_{abs} (nm)		λ_{em} (nm)		HOMO (eV)	LUMO (eV)	E_g (eV)
	Solution	Film	Solution	Film			
BONVB	385	355, 397, 422	429, 462, 487	467, 498, 533	5.4	2.50	2.90
BDNVB	375	350, 390, 407	427, 461, 488	470, 499, 534	5.35	2.55	2.80
BDDNVB	355	310, 375, 425	427, 450	469, 499, 533	5.34	2.54	2.80

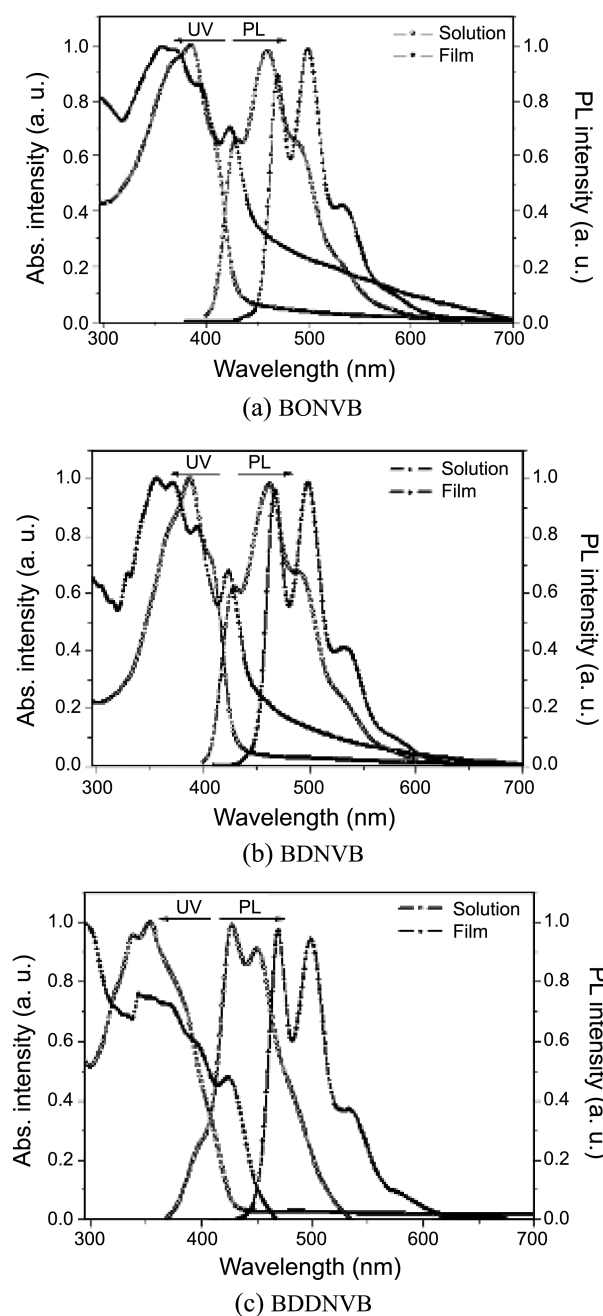


Figure 1. UV-vis absorption and photoluminescence (PL) spectra of BONVB, BDNVB and BDDNVB in CHCl_3 solution and the thin film.

(TGA) and differential scanning calorimetry (DSC) in the nitrogen atmosphere. The 5% weight loss of BONVB, BDNVB and BDDNVB was observed at 410 °C regardless chain length of alkoxy group. DSC reveals reversible melting features for BONVB, BDNVB and BDDNVB with each material exhibiting one or two thermal transitions before melting. From the results, the obtained compounds are crystalline and well ordered thin films could be formed by thermal evaporation processes (Figure 2, Table 2).

In order to understand the charge transport properties and assess the ionization potentials and electrochemical stability of BONVB, BDNVB and BDDNVB, cyclic voltammetry

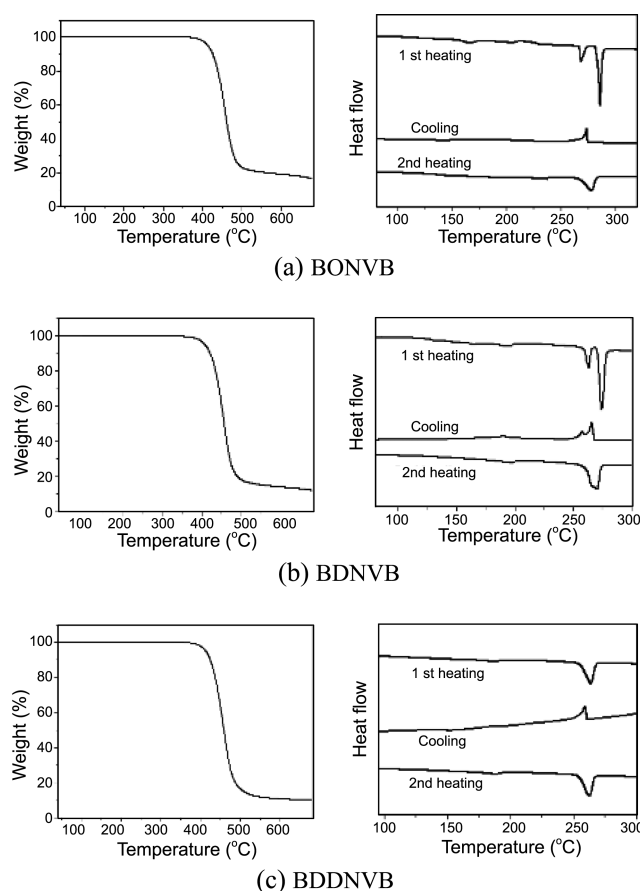


Figure 2. TGA and DSC thermogram of BONVB, BDNVB and BDDNVB.

(CV) measurements were carried out on a thin film of BONVB, BDNVB and BDDNVB (Table 1). The oxidation potential E_{ox} was 1.0 V for BONVB, 0.95 V for BDNVB and 0.96 V for BDDNVB, respectively, showing good electrochemical stability. The HOMO level was estimated to be 5.4 eV, 5.35 eV and 5.34 eV for BONVB, BDNVB and BDDNVB, respectively, using a reported empirical equation.^{42,43} From the results, it is expected that BONVB, BDNVB and BDDNVB will have good stability, which is a major concern with organic semiconductor such as pentacene.

OTFT was fabricated by using thin film of BONVB, BDNVB and BDDNVB as active layer, and their electrical properties were characterized. The mobility (μ_{FET}) and threshold voltage (V_{TH}) of the OFETs were obtained from equation 1 for saturation regimes.

$$I_{\text{D}}^{\text{sat}} = (W/2L)\mu_{\text{FET}}C_{\text{i}}(V_{\text{G}} - V_{\text{TH}})^2$$

Table 2. Thermal properties of BONVB, BDNVB and BDDNVB

	Transition temperature (°C)			T_{d} (°C)
	1st heating	Cooling	2nd heating	
BONVB	268, 286	273	278	410
BDNVB	263, 274	257, 265	266, 270	410
BDDNVB	263	259	262	410

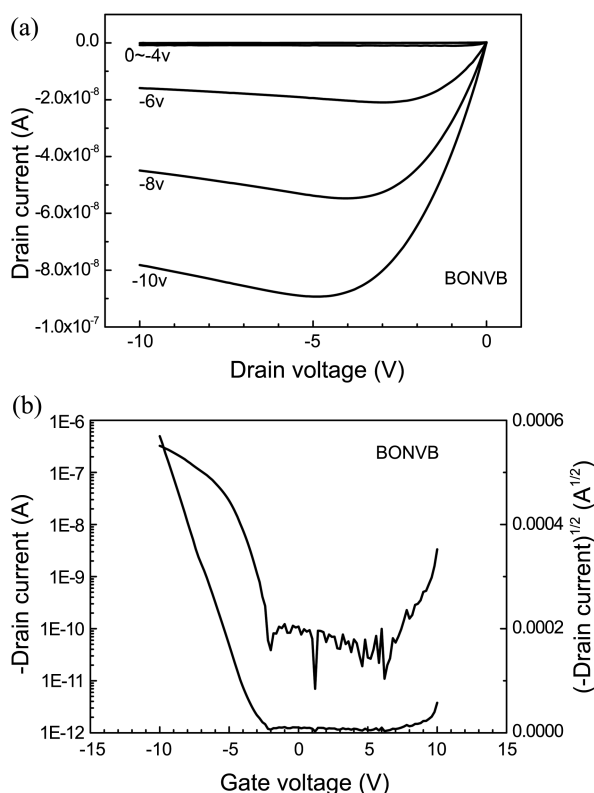


Figure 3. Field effect transistor device characteristic with BONVB semiconductor. (a) Output characteristics and (b) transfer characteristics of BONVB.

Where L is the channel length, W is the channel width, and C_i is the capacitance per unit area of the gate dielectric layer ($C_i = 10 \text{ nF/cm}^2$ for 300 nm thick SiO_2). The output characteristics showed very good saturation behavior and clear saturation currents that are quadratic to the gate bias.

The BONVB thin-film transistor was found to exhibit typical p-channel FET characteristics. Figure 3 shows the drain current (I_D) versus drain-source voltage (V_{DS}) characteristics of fabricated devices using BONVB as semiconducting material. Clearly, a significant change in the drain current can be observed for devices when a gate voltage (V_G) is applied. Figure 3 shows a plot of the $I_D^{1/2}$ versus the V_G characteristics as well as a plot of the I_D versus the V_G characteristics for the device using BONVB as semiconductor. The mobility in the saturation regime was determined using above Equation. Table 3 summarizes the field effect mobilities as well as on/off current ratios and the threshold voltages obtained with BONVB, as active semiconductor

Table 3. Characteristics of field-effect transistor

	Mobility (cm^2/Vs)	V_{th} (V)	Ss (V/dec)	I_{off} (A)	I_{on}/I_{off}
BNDV ²⁵	0.062	-0.61	0.4	1.38×10^{-11}	2.45×10^5
BHNDV ²⁶	0.015	-2.52	0.69	4.11×10^{-11}	1.18×10^5
BONVB	0.011	-2.91	0.93	1.52×10^{-11}	1.31×10^5
BDNVB	-	-	-	-	-
BDDNVB	-	-	-	-	-

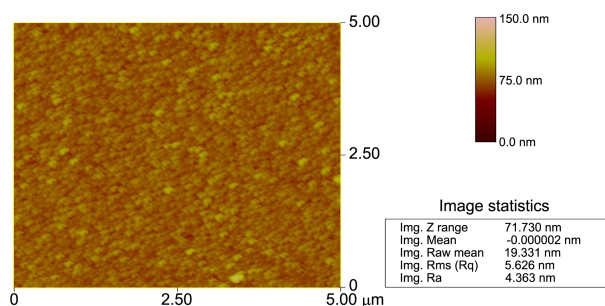


Figure 4. AFM image of BONVB.

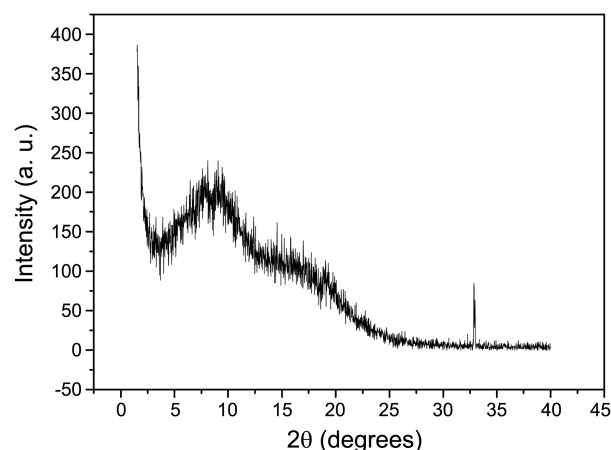


Figure 5. X-ray diffractogram of BONVB.

layer deposited on 1,1,1,3,3,3-hexamethyldisilazane (HMDS)-treated SiO_2 . BONVB had the mobility of $0.011 \text{ cm}^2/\text{V}\cdot\text{s}$, on/off ratio of 1.31×10^5 , and a subthreshold slope of 0.93 V. However, the devices using BDNVB and BDDNVB as semiconductor did not show the mobility characteristics while naphthalene end capped divinylbenzene (BNDV) and hexyloxy naphthalene end capped divinylbenzene (BHNDV) showed mobility of 0.062 and 0.015, respectively.^{24,43} From the results, the mobility of dialkoxy naphthalene end capped divinylbenzene depends on their chain length.

In order to understand of morphological properties, the film of BONVB on the 1,1,1,3,3,3-hexamethyldisilazane (HMDS)-treated substrates were examined by atomic force microscopy (AFM) (Figure 4). The film morphology of BONVB consists of grains which are 0.06–0.05 μm in diameter while those of reported BHNDV and BNDV consists of grains which are 0.1–0.08 μm and 1–2 μm in diameter, respectively, although film show that the surface morphology is smooth and the substrate is fully covered with the materials.^{24,43}

The orientation of the thin films was also investigated by means of X-ray diffraction (XRD) analysis. Figure 5 shows X-ray diffractograms of the BONVB. Thin film XRD characterization of BONVB revealed amorphous nature without ordered crystallinity while the reported BHNDV and BNDV showed highly ordered crystalline structure.

Conclusion

We fabricated OTFT based dialkoxy naphthalene end-

capped divinylbenzene. BONVB, BDNVB and BDDNVB showed not only good thermal stability and oxidation stability with deep HOMO level but also H-aggregates and J-aggregates due to closely packed structure between adjacent molecules in UV-absorption of thin film and solution, regardless the chain length of alkoxy group. However, the field effect performances of dialkoxy naphthalene end capped divinylbenzene depends on chain length of dialkoxy groups. As the chain length increase, the thin film transistor performance was decreased. As the chain length decrease, the morphology with relatively large grain size and the ordered crystallinity were increased, leading increased mobility.

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