



Journal of Nanoscience and Nanotechnology Vol. 16, 10331–10336, 2016 www.aspbs.com/jnn

p-type carrier mobilities ($\leq 0.1 \text{ cm}^2/\text{Vs}$).¹² Therefore, it

is desirable to explore new arylacetylene derivatives that

lene derivative based on a benzothiadiazole core end-

functionalized with thiophene moieties (Fig. 1). Thiophene

and phenyl groups exhibit electron-donating characteris-

tics, while benzothiadiazoles represent electron-accepting

moieties. Donor-acceptor-donor units with connecting

carbon-carbon triple bonds enhance molecular rigidity and

pi-polarization, possibly resulting in large pi-conjugation

with high film crystallinity.¹³ The thermal, optical, and

electrochemical properties of the new compound were

measured. The molecular structure and HOMO/LUMO

energy levels of the compound were determined via den-

sity functional theory (DFT) calculations. Furthermore,

the new compound was employed as a semiconductor

film, as prepared by a solution process [solution shearing

(SS)], in a top-contact/bottom-gate OTFT, and the result-

ing devices were characterized. Finally, the morphology

and microstructure of the deposited films were character-

ized by atomic force microscopy (AFM) and X-ray diffraction (XRD). Our results revealed that films of **5** were

active as *p*-channel organic semiconductors with a carrier

To this end, we have synthesized a novel arylacety-

show high electrical performance.

Synthesis and Characterization of Arylacetylene Derivative as Solution Processable Organic Semiconductors for Organic Thin-Film Transistors

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New arylacetylene derivative (4,7-bis((4-(thiophen-2-yl)phenyl)ethynyl)benzo[*c*][1,2,5]thiadiazole; **5**) functionalized with thiophene and benzothiadiazole was synthesized and characterized as organic semiconductors for organic thin-film transistors (OTFTs). Compound **5** exhibited *p*-channel characteristics with carrier mobility as high as 10^{-4} cm²/Vs and a current on/off ratio >10⁵ for top-contact/bottom-gate OTFT devices.

Keywords: Organic Thin-Film Transistors, Arylacetylene, Benzothiadiazole, Self-Assembled Monolayer. Description of the second se

> : 141.101.201.88 On: Mon, 29 Aug 2016 21:03:2 Copyright: American Scientific Publishers

1. INTRODUCTION

Organic semiconductors based on pi-conjugated small molecules and polymers are widely studied because of their potential application as essential components for organic thin-film transistors (OTFTs).¹⁻⁴ Numerous chemical cores, including pentacene,⁵ oligothiophene,⁶ and (hetero)acenes,7 have been developed and employed in OTFT applications. Despite recent achievements, it is desirable to develop new organic semiconductors. To this end, conjugated arylacetylenes are a promising class of semiconductors suitable for OTFT applications. Arylacetylenes have been less investigated as compared to other molecular cores. Conjugated triple-bond-containing systems have gained much interest as a promising class of semiconductors. This is due to the availability of efficient synthetic protocols for alkynylation reactions, and favorable steric and conformational advantages for orbital delocalization.⁸⁻¹¹ Appropriate functionalization of these cores with electron-donating or electron-withdrawing functional groups has been known to modulate their physical and electronic properties. For example, Silvestri et al. reported solution-processable arylacetylenes with good

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Figure 1. Chemical structure of 4,7-bis((4-(thiophen-2-yl)phenyl) ethynyl)benzo[c][1,2,5]thiadiazole (**5**).

mobility as high as 10^{-4} cm² V⁻¹ s⁻¹, and a current on/off ratio >10⁵.

2. EXPERIMENTAL DETAILS

2.1. General Methods

Air and/or moisture sensitive reactions were carried out under an N₂ atmosphere in oven-dried glassware and with anhydrous solvents. All chemicals were purchased from commercial sources unless otherwise noted and used without further purification. Solvents were freshly distilled or dried by passing through an alumina column. Thin layer chromatography was carried out on glass plates coated with silica gel SiO₂ 60 F254 from Merck; visualization with a UV lamp (254 nm) or by staining with a *p*-anisaldehyde or potassium permanganate solution. Flash chromatography was performed with silica gel SiO₂ 60 (0.040-0.063 µm, 230-400 mesh), technical solvents, and a head pressure of 0.2-0.4 bar. Proton (¹H) and carbon (¹³C) nuclear magnetic resonance (NMR) spectroscopy was performed on a JEOL ECP-400 spectrometer at 400 MHz (¹H) and 100 MHz (¹³C) at 294 K. Chemical shifts are reported in ppm relative to the residual non-deuterated solvent (CDCl₃: $\delta H = 7.26$ ppm, $\delta C =$ 77.16 ppm). All ¹³C NMR spectra are proton decoupled. The resonance multiplicity is described as s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), dd (doublet of doublet), dt (doublet of triplet), td (triplet of doublet), *m* (multiplet), and *br* (broad). High-resolution mass spectrometry (HRMS) was measured on a JEOL JMS-700 spectrometer. Mass peaks are reported in m/z units.

2.2. Synthesis

2.2.1. Synthesis of ((4-bromophenyl)ethynyl) trimethylsilane(1)

1-Bromo-4-iodobenzene (1a, 300 mg, 1.06 mmol), Pd(PPh₃)₄ (61.2 mg, 0.053 mmol), and CuI (20 mg, 0.11 mmol) were dissolved in 10 mL of triethylamine and trimethylsilyl acetylene (1b, 176 mL, 1.27 mmol) was added slowly at 0 °C. Then, reaction mixture was stirred under N₂ atmosphere for 5 h and trimethylamine was distilled off under vacuo. The residue obtained was taken up in ethyl acetate and washed with distilled water and brine. The organic layer was next dried over anhydrous MgSO₄ and evaporated *in vacuo* to give the crude product. Then the crude product was purified by flash column chromatography on silica gel to give ((4-bromophenyl)ethynyl)trimethylsilane(1) as a white solid (192 mg, 71.5%).¹¹ ¹H NMR (400 MHz, CDCl₃): δ 7.40 (*d*, 8.4 Hz, 2H), 7.30 (*d*, 8.4 Hz, 2H), 0.24 (*s*, 0.22 Hz, 9H).

2.2.2. Synthesis of Trimethyl((4-(thiophen-2-yl) phenyl)ethynyl)silane(2)

A mixture of ((4-bromophenyl)ethynyl)trimethylsilane (1, 300 mg, 1.18 mmol), 2-thiophenboronic acid (2a, 180 mg, 1.4 mmol), and $Pd(PPh_3)_4$ (68 mg, 0.059 mmol) was dissolved in tetrahydrofuran (10 mL) and 2 M aq. Na₂CO₃ (1.8 mL, 3.6 mmol) was added. The reaction mixture was refluxed for 12 h under N2 atmosphere. The crude mixture was diluted with ethyl acetate, and filtered, and then the residue was washed with ethyl acetate. The combined filtrates were washed with distilled H₂O. Organic layer separated, dried over anhydrous MgSO₄ and evaporated in vacuo. Then the crude product was purified by column chromatography on silica gel to give trimethyl((4-(thiophen-2-yl)phenyl)ethynyl)silane(2) as a pale yellow solid (183 mg, 60.4%).¹² ¹H NMR (400 MHz, CDCl₃): δ 7.52 (d, 8.04 Hz, 2H), 7.44 (d, 8.44 Hz, 2H), 7.31 (d, 3.64 Hz, 1H), 7.28 (d, 4.76 Hz, 1H), 7.06 (t, 8.8 Hz, 1H), 0.23 (s, 9H).

2.2.3. Synthesis of 2-(4-ethynylphenyl)thiophene(3)

Trimethyl((4-(thiophen-2-yl)phenyl)ethynyl)silane (**2**, 100 mg, 0.39 mmol) and 2 M KOH (5 mL) in methanol were dissolved in 5 mL of tetrahydrofuran. The reaction mixture was stirred at room temperature for 1 h under N₂ atmosphere. After completion of the reaction (indicated by TLC), H₂O and ethyl acetate were added. The organic layer was separated, dried over anhydrous MgSO₄ and evaporated *in vacuo*. Then the crude product was purified by column chromatography on silica gel to give 2-(4-ethynylphenyl)thiophene(**3**) as a yellow solid (68 mg, 94.7%).¹³ ¹H NMR (400 MHz, CDCl₃): δ 7.56–7.54 (*m*, 2H), 7.49–7.47 (*m*, 2H), 7.32–7.28 (*m*, 2H), 7.07 (*q*, 4.76, 5.12 Hz, 1H), 3.12 (*s*, 1H).

2.2.4. Synthesis of 4,7-Diiodobenzo[c] [1,2,5]Thiadiazole(4)

Benzo[*c*][1,2,5]thiadiazole (**4a**, 1.0 g, 7.34 mmol), I₂ (4.12 g, 16.23 mmol), and Ag₂SO₄ (1.29 g, 7.34 mmol) were dissolved in 10 mL of conc. H₂SO₄. Resulting mixture was stirred at 110 °C for 14 h. After cooling to room temperature, the reaction mixture was poured into ice water and the precipitate was collected by filtration. The precipitate was dissolved in CH₂Cl₂ and washed with aq. NaHSO₃ solution and bine. The organic layer was separated, dried over anhydrous MgSO₄ and evaporated *in vacuo*. The crude product was purified by recrystallization in ethanol to afford 4,7-diiodobenzo[*c*][1,2,5]thiadiazole(**4**) as a yellow crystal (1.5 g, 52.6%).¹⁴ ¹H NMR (400 MHz, CDCl₃): δ 8.16 (*s*, 2H).

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2.2.5. Synthesis of 4,7-Bis((4-(thiophen-2-yl)phenyl) ethynyl)benzo[c][1,2,5]thiadiazole(5)

2-(4-Ethynylphenyl)thiophene (3, 100 mg, 0.54 mmol), 4,7-diiodobenzo[c][1,2,5]thiadiazole (4, 104 mg. 0.26 mmol), $Pd(PPh_3)_4$ (16 mg, 0.013 mmol), and CuI (5.4 mg, 0.028 mmol) were dissolved in 10 mL of triethylamine/THF (1:1). The reaction mixture was heated at 60 °C for 24 h under N₂ atmosphere. After completion of the reaction (indicated by TLC), solvent was removed by reducing pressure. The crude mixture was dissolved in CH₂Cl₂ and washed with water and brine solution. Then, organic layer was separated, dried over anhydrous MgSO₄ and evaporated in vacuo. Then the crude product was purified by column chromatography on silica gel to give compound 5 as a vellow solid (84.8 mg, 66%).^{11,15} ¹H NMR (400 MHz, CDCl₃): δ 7.79 (s, 2H), 7.64 (q, 12.84, 12.12 Hz, 8H), 7.37 (d, 3.68 Hz, 2H), 7.31 (dd, 4.76, 5.12 Hz, 2H), 7.09 (q, 4.76 Hz 2H). ¹³C NMR (100 MHz, CDCl₃): δ 155.09, 144.22, 135.84, 135.72, 133.76, 133.26, 133.16, 128.98, 127.01, 126.54, 126.43, 124.65, 124.58, 122.03, 117.88, 98.24, 87.02, 125.9, 124.5, 124.4, 120.3, 120.1, 109.6. HRMS-EI(*m*/*z*): $[M + Na^+]$ calcd. for $C_{30}H_{16}N_2S_3Na^+$, 523.0368; found, 523.0376.

2.3. Theoretical Calculation

Density functional theory (DET) calculations on the A present semiconductor were performed using the B3LYP (Becke's 3 parameters employing the Lee-Yang-Parr) functional and the 6-31G(d) basis set as implemented in Gaussian 03W program.

2.4. Device Fabrication

Top-contact/bottom-gate organic thin-film transistors (OTFTs) were fabricated on a heavily n-doped (100) silicon wafers (resistivity <0.005 $\Omega \cdot cm$) with a thermally grown silicon dioxide (300 nm SiO₂) as dielectric layer. The Si/SiO₂ substrate was washed in an ultrasonic bath using acetone for 10 min, dried using a N₂ gun and cleaned by air plasma for 5 min (Harrick Plasma, 18 W). The general recipes were employed for the treatment of PS-brush layer9 and OTS self-assembled monolayer20 on the Si/SiO₂ substrates. Hydroxyl end-functionalized polystyrene (Mn = 10 kg/mol, Polymer Source) was employed for the formation of PS-brush layer. Semiconducting layers were formed via solution-shearing (SS) method. For the SS process, a few drops of semiconductor solution were cast onto a heated substrate $(1 \times 2 \text{ cm}^2)$, and the substrate was covered with a dewetting OTS-modified top substrate.²¹ For the optimization of film-forming process, various solvents including toluene, chlorobenzene, p-xylene, 1,2,4-trichlorobenzene with concentration (1 mg/mL) were employed. Solvent evaporation was controlled by a deposition temperature (60% of the solvent boiling point in centigrade) and a solution shearing

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speed (6 mm/min). The solution-sheared substrates were placed in a vacuum oven at 90 °C for 3 hr to remove the residual solvent. Film thicknesses were characterized by profilometer (DEKTAK-XT, Brucker) with a thickness in the 20–50 nm range. Gold source and drain electrodes (40 nm) were thermally evaporated through a shadow mask with various channel lengths length (L; 50 μ m) and width (W; 1000 μ m).

2.5. Characterization

Thermogravimetric analyses (TGA, TA Instrument O50-1555) were performed on sample in a platinum crucible; the sample was heated from 40 to 700 °C at a heating rate of 20 °C min⁻¹, while the chamber was purged continuously with N₂ gas at a rate of 100 mL min⁻¹. Differential scanning calorimetry (DSC) analyses (TA instrument Q20-2487) were performed at a scan rate of 20 °C min⁻¹ from 40 to 350 °C. The UV-visible spectra of the compound in chloroform were obtained using JASCO V-530 spectrometer with quartz cuvette over the special range 200-800 nm. Cyclic voltammetry (CV) experiments were performed with a conventional three-electrode configuration (glassy carbon working electrode, platinum-wire counter electrode, and Ag/AgCl reference electrode) with supporting electrolyte of tetrabutylammonium tetrafluoroborate in the specified dry solvent on AUT302N Electrochemical Analyzer (Autolab). All electrochemical potentials were referenced to an Fc+/Fc internal standard. The currentvoltage characteristics of fabricated OTFT devices were measured at room temperature under vacuum using a Keithley 4200 SCS. Carrier mobilities (μ) were calculated in the saturation regime by the formula, $\mu_{\rm sat} = (2I_{\rm DS}L)/$ $[WC_i(V_{\rm G}-V_{\rm T})^2]$, where $I_{\rm DS}$ is the source-drain current, L is the channel length, W is the channel width, C_i is the areal capacitance of the gate dielectric (C_i = 11.4 nF cm⁻²), $V_{\rm G}$ is the gate voltage, and $V_{\rm T}$ is the threshold voltage.

The surface morphology and film microstructure of thin films was characterized using an atomic force microscope (AFM, NX10, Park Systems) and X-ray diffraction (XRD, Miniflex, Rigaku), respectively.

3. RESULTS AND DISCUSSION

3.1. Synthesis

The synthetic pathway for the preparation of **5** is illustrated in Scheme 1. First 2-(4-ethynylphenyl)thiophene (**3**) was prepared in three steps, namely: Sonogashira coupling of 1-bromo-4-iodobenzene (**1a**) and TMS-acetylene (**1b**) to afford ((4-bromophenyl)ethynyl)trimethylsilane (**2**);¹⁴ followed by Suzuki coupling of this compound and thiopheneboronic acid (**2a**) to afford trimethyl((4-(thiophen-2-yl)phenyl)ethynyl)silane (**2**);¹⁵ after which the TMS group was deprotected to afford 2-(4-ethynylphenyl)thiophene (**3**).¹⁶



Scheme 1. Synthesis of 5.

Next, 4,7-diiodobenzo[c][1,2,5]thiadiazole (**4**) was synthesized by direct iodination of benzo[c][1,2,5]thiadiazole (**4a**).¹⁷

Finally, **3** and **4** were coupled by Sonogashira reaction to afford **5**.¹⁸ Delivered by Ingenta to: A IP: 141.101.201.88 On: M

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3.2. Thermal, Optical, and Electrochemical Properties The thermal properties of the new arylacetylene derivative were characterized by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). A sharp endotherm at 251 °C and weight loss (~5%) upon heating above 237 °C were observed (Table I), indicating thermal stability of **5**. The optical absorption spectra in chloroform solution display λ_{max} values at 249, 328, and 438 nm (Fig. 2). Hence, the HOMO–LUMO energy gap calculated from the onset of the experimental optical absorption was determined as 2.54 eV.

Cyclic voltammetry (CV) measurements of **5** were performed in o-C₆H₄Cl₂ at 25 °C. The compound exhibits an oxidation peak at around +1.50 V (using ferrocene/ferrocenium as an internal standard at +0.73 V). Hence, the HOMO energy level, determined by the conversion of the oxidation potential using ferrocene as the standard, was -5.51 eV. The LUMO energy levels were calculated from the HOMO (CV measurements) and the bandgap (UV-vis measurements), as illustrated in Table I.

Table I. Physical and electrochemical properties of the compound 5.

DSC T _m (°C)	TGA (°C; 5%)	UV-Vis $\lambda_{\max} \ (nm)^a$	$E_{ m ox}^{ m peak} \ ({ m V})^b$	$E_{\rm gap} \ ({ m eV})^a$	HOMO (eV) ^b	LUMC (eV) ^c
251	237	249, 328, 438	1.50	2.54	-5.51	-2.97

3.3. Theoretical Calculation

The molecular structure and HOMO/LUMO energy distribution of the arylacetylene derivative were determined by DFT (B3LYP) calculations with 6-31G(d) basis sets (Fig. 3). In **5**, with arylacetylene moieties on both ends, the HOMO was dispersed over the whole molecule. On the other hand, the LUMO was localized on the benzothiadiazole moiety. These results demonstrate the electron-accepting character of the benzothiadiazole. The theoretically calculated band gap energy was 2.56 eV. This value is similar to that determined from the UV-vis spectrum.

3.4. Thin-Film Transistor Characterization

Top-contact/bottom-gate OTFTs were fabricated using thin films of **5** as semiconducting layers, via a solution process method (SS). PS brush-coated SiO₂/Si substrates were used as the dielectric and gate. For the SS process,



Figure 2. Optical spectra of 5 in chloroform solution.

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Figure 3. Molecular orbital surfaces of the HOMO and LUMO energy levels of 5 determined from DFT calculations.

various conditions for the formation of thin films were optimized. These include the solvents (toluene, chlorobenzene, *p*-xylene, 1,2,4-trichlorobenzene), concentration of solution, temperature of substrate, and shearing speed. Gold electrodes were deposited through a shadow mask to define the source and drain. Table II summarizes the OTFT data (charge carrier mobility, current on/off ratio, and threshold voltage). Solution-sheared films of **5** exhibited *p*-channel activity with a carrier mobility of up to 1.0×10^{-4} cm²/Vs, and a current on/off ratio > 10^5 . Solution-sheared films of **5** exhibited *p*-channel activity with a carrier mobility of up to 1.0×10^{-4} cm²/Vs, and a current on/off ratio > 10^5 . Solution-sheared films of **5** exhibited *p*-channel activity with a carrier mobility of up to 1.0×10^{-4} cm²/Vs, and a current on/off ratio > 10^5 .

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Table II. TFT device performance parameters based on thin films of compound **5** (μ : carrier mobility, I_{on}/I_{off} : current on/off ratio, V_{T} : threshold voltage).

Method	Surface treatment	Solvent	Surface temperature (°C)	μ (cm ² /Vs)	$I_{\rm on}/I_{\rm off}$	$V_{\rm T}$ (V)
SS	PS-brush	Toluene	60	$1.0 imes 10^{-4}$	4.7×10^5	-11



Figure 4. Representative transfer curve of the OTFT device based on thin films of 5. (Inset: representative output curve of the corresponding OTFT device).

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Figure 5. (a) AFM topographic image $(10 \times 10 \ \mu\text{m}^2)$ of a thin film fabricated by SS of **5** (Inset: $\theta - 2\theta$ XRD scan of the corresponding thin film). Scale bar = 2 μ m. (b) Microscopy image of a solution-sheared thin film. The white arrow represents the shearing direction; scale bar = 8 μ m.

3.5. Thin-Film Microstructure and Morphology

The film microstructure and surface morphology of the new solution-processed semiconductor thin films were characterized using wide-angle θ -2 θ XRD and AFM to evaluate device performance. Figure 5(a) illustrates the AFM surface morphology and conventional $\theta/2\theta$ XRD scan of the organic semiconductor thin film based on 5. The XRD scan (Inset of Fig. 5(a)) reveals that the solution-sheared thin films did not exhibit any significant Bragg reflections. This is an indication of poor film texture. Furthermore, the AFM image demonstrates a relatively oriented crystalline morphology with a surface roughness of ~11.0 nm for a 10.0 μ m × 10.0 μ m scan area. Specifically, grains in these thin films displayed preferred growth direction on a locale scale, forming microscale rods with evident terraced islands.

4. CONCLUSION

New arylacetylene derivative with benzothiadiazole core end-functionalized with phenylthiophene was synthesized and characterized. Thin-film transistors fabricated from the compound exhibited *p*-channel device characteristics with mobility as high as 1.0×10^{-4} cm²/Vs.

Acknowledgment: This study was supported by a Research Grant of Pukyong National University (2015).

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Received: 28 November 2015. Accepted: 23 March 2016.

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