Solution-Processable Field-Effect Transistors Fabricated Using Aryl Phenoxazine Based Polymers as the Active Layer

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Three phenoxazine-based conjugated polymers, namely, the aryl substituted phenoxazine homopolymer (P1) as well as the dimeric phenoxazine-fluorene (P2) and phenoxazine-bithiophene (P3) copolymers, were synthesized *via* the Ni(0) mediated Yamamoto reaction and the palladium-catalyzed Suzuki coupling reaction. The weight-averaged molecular weights (M_w) of P1, P2, and P3 were found to be 27,000, 22,000, and 15,000, respectively, and their polydispersity indices were 3.6, 1.8, and 2.1. All the polymers were soluble in common organic solvents such as chloroform, toluene, and so on. The UV-visible absorption maxima for P1, P2, and P3 in the film state were located at 421, 415 and 426 nm, respectively, and the ionization potentials of the polymers ranged between 4.90 and 5.12 eV. All the studied phenoxazine-based polymers exhibited amorphous behavior, as confirmed by X-ray diffraction (XRD) and atomic force microscopy (AFM) studies. Thin film transistors were fabricated using the top-contact geometry. P1 showed much better thin-film-transistor performance than P2 or P3: A thin film of P1 gave a saturation mobility of 0.81×10^{-3} cm²V⁻¹s⁻¹ and an on/off ratio of about 10^2 .

Key Words: Organic transistors, Semiconducting polymers, Phenoxazine

Introduction

Interest in organic thin-film transistors (OTFTs) based on solution-processable organic semiconductors has increased considerably in recent years because of their various applications. which include low-cost memory devices, large-area displays, and flexible electronics.¹⁻⁸ Recently, the charge-carrier mobility and stability of solution-processable organic semiconductors have sharply improved, enabling the achievement of thin-film-transistor (TFT) performances as high as those of vacuum-deposited oligomers.⁹⁻¹² To obtain a high mobility, the charge carriers must be transported through the TFT channel with a minimum of trap states. It is important that the polymer semiconductors are well-ordered on the substrates to reduce the number of trap states. In recent years, polythiophene derivatives containing long regioregular alkyl side chains for good intermolecular alignment have been mainly studied.¹³⁻¹⁵ Most of the organic semiconductors used for the fabrication of organic transistors are derived from thiophene based conjugated materials. Herein, we study various classes of organic semiconductors in order to obtain highly stable compounds exhibiting good performance. Phenoxazine and phenothiazine derivatives are well-known tricyclic heteroaromatic materials with a high electron density in their aromatic rings. Polymers containing phenoxazine or phenothiazine moieties have attracted great interest because of their hole-transporting properties.¹⁶ N-Alkyl substituted phenoxazine compounds and their analogues containing fluorene and thiophene rings in their polymer backbone have been reported by Jenekhe *et al.*¹⁷ Most of those polymers were synthesized by means of random copolymerization, and we have been interested in the alternating incorporation of phenoxazine groups.

Here, we report the synthesis, characterization, and fieldeffect mobility of an N-aryl-substituted phenoxazine-based homopolymer (P1) and alternating copolymers containing fluorene and bithiophene units. The alternating copolymers reported here are the phenoxazine-fluorene (P2) and phenoxazine-bithiophene (P3) copolymers, in which the phenoxazine unit is used as the dimeric phenoxazine structure to enhance the rigidity of the molecules.

Experimental Section

Synthesis of monomens. 1-(2-Ethylhexyloxy)-4-bromobenzene (1): 4-Bromophenol (20 g, 124.3 mmol). 2-ethylhexyl bromide (21.5 g, 103.6 mmol) and K_2CO_3 (24.1 g, 174.0 mmol) were dissolved in acetone. The mixture was stirred at 80 °C for 72 h. The reaction was then quenched with water and the solution was extracted with CHCl₃. The organic layer was dried over anhydrous MgSO₄ and the solvent removed by rotary evaporation. The crude product was purified by silica gel column chromatography with *n*-hexane to yield compound 1 as a colorless oil (86.9%). ¹H NMR (CDCl₃, 400 MHz): $\hat{0}$ 7.35 (m. 2H), 7.77 (m. 2H), 3.79 (d. 2H), 1.70 (m. 1H), 1.48-1.28 (m. 6H), 0.91 (m. 6H). ¹³C NMR (CDCl₃, 75.5 MHz): $\hat{0}$ 158.56, 132.18, 116.34, 112.53, 70.76, 39.40, 30.57, 29.15, 23.91, 23.13, 14.17, 11.17. Fab+-Mass m/e 285.

10-(4-(2-Ethylhexyloxy)phenyl)-10*H*-phenoxazine (2): In a frame-dried flask. Pd₂(dba)₃ (1.2 g. 136.4 mmol). tricyclohexyl-phosphine (0.5 g, 191.0 mmol) and *t*-BuONa (3.9 g, 40.9 mmol) were added in the glove box. After taking out the flask from glove box. compound 1 (9.3 g. 32.4 mmol), and phenoxazine (5 g, 27.3 mmol) in anhydrous toluene was injected under N_2

gas. The mixture was stirred at 120 °C for 2 h. After the reaction was completed, the product was extracted with CHCl₃. The organic layer was dried over anhydrous MgSO₄ and the solvent removed by rotary evaporation. The crude product was purified by column chromatography on silica gel (hexane) to yield compound **2** as a white solid (75.7%). ¹H NMR (CDCl₃, 400 MHz): δ 7.21 (d, 2H), 7.07 (d, 2H), 6.63 (d, 2H), 6.55 (d, 2H), 3.89 (d, 2H), 1.75 (m, 1H), 1.53-1.31 (m, 6H), 0.97-0.88 (m, 6H). ¹³C NMR (CDCl₃, 75.5 MHz): δ 159.16, 143.97, 134.82, 131.67, 130.97, 123.19, 121.03, 116.68, 115.26, 113.21, 70.75, 39.42, 30.55, 29.15, 23.88, 23.05, 14.01, 11.16. Fab+-Mass m/e 387.

10-(4-(2-Ethylhexyloxy)phenyl)-3-bromo-10H-phenoxazine (3): Compound 2 (2 g, 5.2 mmol) was dissolved CHCl₃. The solution was cooled to -78 °C with a dry ice bath. Solution of Br₂ (2.5 mL, 1.5 mmol) in CHCl₃ (75 mL) was added dropwise to the solution. After the reaction was finished, the product was extracted with CHCl₃. The organic layer was dried over anhydrous MgSO4 and the solvent removed by rotary evaporation. The crude product was purified by column chromatography on silica gel (hexane) to yield compound 3 as a colorless oil (30%). ¹H NMR (CDCl₃, 400 MHz): δ 7.24 (d, 2H), 7.07 (d, 2H), 6.77 (s, 1H), 6.69 (m, 4H), 5.92 (d, 2H), 5.78 (d, 2H), 3.88 (d. 2H). 1.75 (m. 1H), 1.53-1.25 (m. 6H), 0.97-0.89 (m, 6H). ¹³C NMR (CDCl₃, 75.5 MHz): § 159.33, 144.67, 143.56, 134.29, 134.21, 131.46, 130.52, 125.81, 123.60, 121.34, 118.33, 116.81, 115.42, 114.26, 113.39, 112.27, 70.76, 39.43, 30.56, 29.13, 23.90, 23.08, 14.13, 11.18. Fab+-Mass m/e 465.

10-(4-(2-Ethylhexyloxy)phenyl)-3-(10-(4-(2-ethylhexyloxy)phenyl)-10*H*-phenoxazin-7-yl)-10*H*-phenoxazine (4): In a frame-dried flask, Ni(COD)₂(1.4 g, 5.1 mmol) and 2,2'-bipyridyl (0.8 g, 5.1 mmol) were added in the glove box. After taking out the flask from the glove box, compound **3** and anhydrous THF were injected under N₂. The mixture was stirred at 65 °C for 2 h. After the reaction was finished, the product was extracted with CHCl₃. The organic layer was dried over anhydrous MgSO₄ and the solvent removed by rotary evaporation. The crude product was purified by silica gel column chromatography with hexane : CHCl₃(10 : 1) to yield compound **4** as a yellow solid (45.3%). ¹H NMR (CDCl₃, 400 MHz): δ 7.19 (d, 4H), 7.08 (d, 4H), 6.81 (d, 4H), 6.64 (m, 4H), 3.89 (d, 4H). 1.76 (m, 2H), 1.54-1.32 (m, 12H), 0.98-0.89 (m, 12H). Fab+-Mass m/e 773.

10-(4-(2-Ethylhexyloxy)phenyl)-3-(10-(4-(2-ethylhexyloxy)phenyl)-3-bromo-10*H*-phenoxazin-7-yl)-7-bromo-10*H*-phenoxazine (5): To a solution of compound 4 (0.7 g, 1.9 mmol) in a mixture of CHCl₃ (20 mL) and Acetic acid (40 mL) was cooled to 0 $^{\circ}$ C with an ice-water bath. And then *N*-bromosuccinimide (0.6 g, 2.0 mmol) was added. After the reaction was finished, the product was extracted with CHCl₃. The organic layer was dried over anhydrous MgSO₄ and the solvent removed by rotary evaporation. The crude product was purified by silica gel column chromatography with hexane : CHCl₃(3 : 1) to yield compound 5 as a yellow solid (86.3%). ¹H NMR (CDCl₃, 400 MHz): δ 7.18 (d, 4H), 7.16 (d, 4H), 6.77 (d, 4H), 6.68 (m, 4H), 5.93 (d, 2H), 5.79 (d, 2H), 3.89 (d, 4H). 1.74 (m, 2H). 1.53-1.32 (m, 12H), 0.97-0.88 (m, 12H). Fab+-Mass m/e 931.

Synthesis of 4,4,5,5-tetramethyl-2-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-9,9-dioctyl-9*H*-fluoren-7-yl)-1,3,2dioxaborolane (6): Compound 6 was prepared according to a **Synthesis of dialkyl bithiophene monomer (7):** Compound 7 was similarly prepared according to a previously reported method.¹⁹¹H NMR (CDCl₃, 400 MHz): δ 7.78 (s. 2H), 2.51 (t, 4H), 1.55 (m, 4H), 1.35 (m, 36H), 0.86 (m, 6H). Fab+-Mass m/e 586.

Polymerization of homopolymer (P1). The homopolymer of phenoxazine unit was synthesized by nickel(0)-mediated polymerization. The 100 mL Schlenk flask containing anhydrous DMF (5 mL), bis(1.5-cyclooctadienvl) nickel(0), 2.2'-dipyridyl and 1.5-cvclooctadiene (the last three in a molar ratio of 1:1:1) was kept under nitrogen atmosphere at 78 °C for 15 min. Compound 5 dissolved in anhydrous toluene (15 mL) were added to the reaction mixture. The polymerization was maintained at 78 °C for 36 h. When the reaction had finished, each polymer was precipitated from an equi-volume mixture of HCl. methanol and acetone. The polymers were dissolved in small amount of toluene and precipitated in methanol. The resulting polymers were purified with column chromatography using toluene as the eluent. Finally, the resulting polymers were further purified by Soxhlet extraction using methanol and then dried in vacuum. The polymer yield was 28.3% after purification. ¹H NMR (CDCl₃, 400 MHz): ô aromatic; 7.22-5.79 (10H), aliphatic; 3.89 (2H), 1.67-0.92 (15H).

Polymerization of P2. The synthesis of the copolymer was carried out using palladium-catalyzed Suzuki coupling. Compound 5 (0.25 g, 0.278 mmol) and compound 6 (0.187 g, 0.292 numol) were dissolved in 8 mL of anhydrous toluene. Pd(OAc)2 (1.9 mg, 0.008 mmol) and tricyclohexylphosphine (3.9 mg, 0.014 mmol) were added to the mixture under N₂ condition and then aqueous tetraethylammonium hydroxide (2.0 mL, 2.778 mmol) was added to the mixture rapidly. The reaction mixture was stirred and maintained at 90 °C for 6 h until the reaction mixture became viscous. An excess of 2-bromo-5-methylthiophene (0.005 mL, 0.028 mmol) was added as an end capper. After further stirring for 2 h, the reaction mixture was poured into the methanol/HCl mixture solution. The polymer fibers were collected by filtration and reprecipitation from methanol. Finally, the resulting polymers were further purified by Soxhlet extraction using methanol and then dried in vacuum. The polymer vield was 35.0% after purification. ¹H NMR (CDCl₃, 400 MHz): δ aromatic: 7.66-5.89 (26H), aliphatic; 3.91 (2H). 1.96 (2H). 1.55-0.75 (60H)

Polymerization of P3. P3 was synthesized with the same method used for P2, with 0.5 g (0.555 mmol) of compound 5 and 0.358 g (0.611 mmol) of compound 7. The final product was obtained after drying in vacuo with a yield of 46.3%. ¹H NMR (CDCl₃.400 MHz): δ aromatic; 7.11-5.92 (22H), aliphatic; 3.92 (d, 4H), 2.53 (m, 4H), 1.78-0.82 (52H).

Instruments. ¹H and ¹³C NMR spectra were recorded on Bruker AVANCE 400 spectrometer, with tetramethylsilane as an internal reference. Fast atom bombardment (FAB) mass spectra were recorded on a JEOL, JMS-AX505WA, HP5890 seris II. The optical absorption spectra were measured by a shimadzu UV-3100 UV-VIS-NIR spectrometer. FT-IR spectra were recorded on Perkin Elmer Spectrum 2000 system. The number- and

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weight-average molecular weights of polymers were determined by gel permeation chromatography (GPC) on Viscotek T60A instrument, using tetrahydrofuran (THF) as eluent and polystyrene as standard. The differential scanning calorimetry (DSC) and thermal gravimetric analysis were made using TA Q100 instrument and operated under nitrogen atmosphere at a heating rate of 10 °C/min. Cyclic voltammetry was performed on an AUTO-LAB/PG-STAT12 model system with a three-electrode cell in a solution of Bu₄NBF₄ (0.10 M) in acetonitrile at a scan rate of 50 mV/s. A film of each polymer was coated onto a Pt wire electrode by dipping the electrode into a solution of the polymer. X-ray diffraction analyses of the polymer films were performed on XPET-PRO MRD. Electrical characteristics of the TFTs were measured in ambient conditions using both Keithley 2400 and 236 source/measure units.

Fabrication of the organic thin film transistor devices. Thinfilm transistors were fabricated on silicon wafer using the top contact geometry (channel length L = 100 μ m, width W = 1000 µm) in ambient conditions without taking any special precautions to exclude air, moisture and light. A heavily n-doped silicon wafer with a 300 nm thermal silicon dioxide (SiO₂) was used as the substrate/gate electrode, with the top SiO₂ layer serving as the gate dielectric. The SiO2 surface of the wafer substrate was first cleaned with pirana solution and the cleaned wafer was immersed in a solution of octyltrichlorosilane (OTS-8) in toluene at room temperature for 2 h. The semiconductor layer was spin-coated at 2000 rpm from 1.0 wt% toluene solution. with a thickness of 70 nm. Subsequently, a series of gold source/ drain electrode pairs were deposited by vacuum evaporation through a shadow mask. Silicon oxide at the backside of the silicon wafer of the TFT device was removed with HF solution to provide a conductive gate contact.

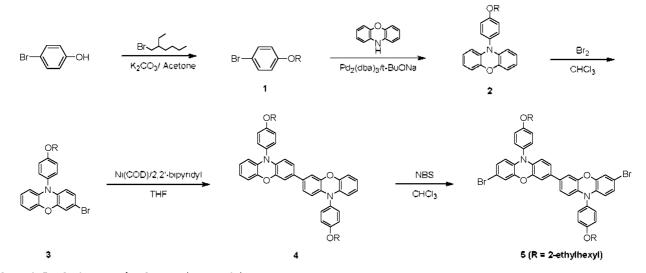
Results and Discussion

Synthesis. The synthetic routes to the monomers and polymers are shown in Schemes 1 and 2, respectively. The aryl substituted phenoxazine homopolymer (P1) was prepared *via* the Ni (0) mediated Yamamoto reaction whereas the dimeric

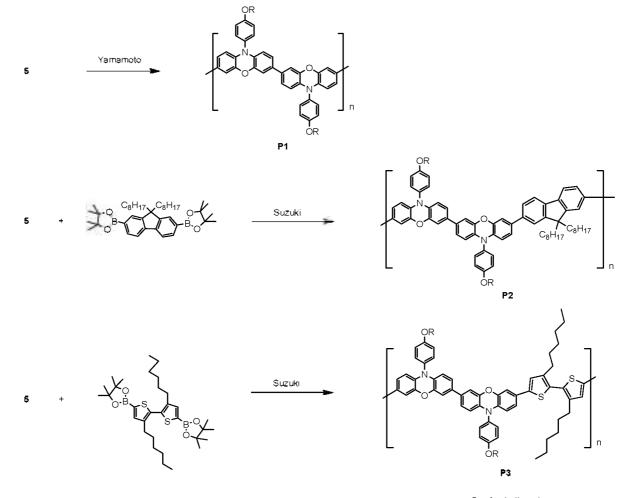
phenoxazine-fluorene (P2) and phenoxazine-bithiophene (P3) copolymers were synthesized by using the palladium-catalyzed Suzuki coupling method. These polymers were soluble in common organic solvents, such as chloroform and toluene, without any evidence of gel formation at room temperature. The molecular weights and polydispersity indices of the polymers were determined *via* gel permeation chromatography (GPC) using tetrahydrofuran (TFT) as the eluent and polystyrene as the standard. The weight-averaged molecular weights (Mw) of P1, P2, and P3 were 27,000, 22,000, and 15,000, respectively, and their polydispersity indices were 3.6, 1.8, and 2.1.

Thermal properties of the polymens. The thermal properties of the polymers were evaluated by means of thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC) measurements. All the polymers showed very good thermal stability and did not decompose at temperatures up to 400 °C under nitrogen atmosphere. The glass-transition temperatures (T₂) of P1, P2, and P3 were found to be 253.7, 158.0, and 100.8 °C, respectively (Figure 1). As can be seen, P1 exhibits the highest glass-transition temperature among all the studied polymers, presumably due to the incorporation of the rigid phenoxazine moiety into the structure of this polymer. None of the polymers showed melting- or crystalline-transition behaviors: therefore, it is thought that they might have an amorphous structure - or that the transition temperature is at higher temperatures than those studied. The results obtained for the synthesized polymers are summarized in Table 1.

Optical and electrochemical properties. UV-visible absorption spectra of the polymers were recorded both in a toluene solution and in the thin-film state (see Figure 2). The UV-visible absorption maxima of P1, P2 and P3 appeared at 406, 413, and 419 nm. respectively. in solution, and at 421, 415, and 426 nm, respectively, in the film state. The absorption spectra of the polymer thin films show a shape similar to that of the spectra obtained in dilute solution, except for the red shift observed in the case of the thin films. The wavelength differences between the absorption maxima observed for P1, P2, and P3 in the film state and in solution ($\Delta\lambda$) were found to be 15, 2, and 7 nm, respectively, whereby P1 showed the largest $\Delta\lambda$ value among all



Scheme 1. Synthetic routes for phenoxazine containing monomer.



Scheme 2. Synthetic routes for P1, P2 and P3 polymers.

the studied polymers. This result may be explained by considering that the planar structures of the phenoxazine units play an important role in increasing the π - π stacking during the aggregation of the polymer chains. In the case of the alternating copolymers, P2 and P3, the long alkyl substituents of the fluorene

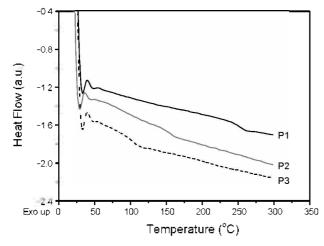


Figure 1. DSC thermograms of P1, P2 and P3 with a heating rate 10 °Cmin⁻¹ under inert atmosphere.

(R = 2-ethylhexyl)

and bithiophene units decrease the intermolecular interaction. which can lead to less efficient π - π stacking. The maximum absorption peak of the dimeric phenoxazine-bithiophene copolymer (P3) exhibited a 5 nm red shift, whereas the dimeric phenoxazine-fluorene copolymer (P2) exhibited a 6 nm blue shift, compared to the homopolymer (P1) in the solid state. The small red-shifted absorption peak of P2 may be due to an elongation of the effective conjugation length by introducing the unhindered 2.2'-dialkylbithiophene unit. To investigate the electrochemical properties of the polymers, we carried out cyclic voltammetry (CV) measurements. The ionization potentials (IPs) of P1, P2, and P3 were estimated to be 4.90, 5.12, and 5.12 eV, respectively. From these results, we can conclude that the phenoxazine moiety efficiently increases the electron density in the polymer backbones, which may facilitate the carrier transport within the thin films. These energy levels of the polymers

Table 1. Summary of the characteristics of the synthesized polymers.

Polymer	$M_{\rm w}(g/mol)$	$M_n(\text{g/mol})$	PDI	$T_{g}(^{\circ}C)$	$T_d(^{\circ}\!C)$
P1	27,300	7,500	3.6	253.7	417.9
P2	22,400	12,400	1.8	158.1	430.0
P3	15,100	7,100	2.1	100.8	461.7

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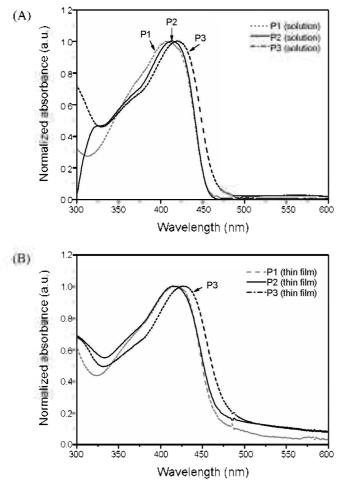


Figure 2. UV-visible spectra of P1, P2 and P3 in toluene solution (A) and in solid thin films (B).

matched well with that of the source/drain Au (5.1 eV) contact. The results of the optical and electrochemical studies performed on P1, P2, and P3 are summarized in Table 2.

TFT Characteristics of the Polymer Thin Films. OTFTs based on P1, P2, and P3 were fabricated by means of solution processing. Top-contact OTFTs were fabricated on a common gate consisting of highly n-doped silicon with a 300-nm-thick thermally grown SiO₂ dielectric layer. The substrates were modified with OTS-8. Organic-semiconductor films with a thickness of 70 nm were spin-coated at 2000 rpm from a 1.0 wt.% toluene solution, followed by drying at 100 °C (for 5 min) to remove the solvent. Gold was deposited on top of the thin-film surface through a shadow mask to give the source and drain electrodes. The channel length L was $100 \,\mu\text{m}$ and the channel width W was 1000 µm. The OTFT characteristics were determined under ambient conditions, and all the OTFTs containing P1, P2, and P3 were found to exhibit typical p-channel characteristics. Figure 3 shows the typical output and transfer curves of P1 polymer under ambient conditions without taking any precautions to insulate the materials and devices from exposure to air, moisture, and light. The field-effect mobility was extracted from the following equation:²⁰

Saturated regime $(V_{\rm D} > V_{\rm G})$: $I_{\rm D} = (W/2L)\mu C_{\rm i} (V_{\rm G} - V_{\rm T})^2$

 Table 2. Optical and electrochemical properties of the synthesized polymers.

Polymers	$\lambda \max(nm)$			Eg (eV)	ID (N) ⁴
	solution	film	$\Delta \lambda$. Tā(ev)	IF (EV)
Pl	406	421	15	2.67	4.90
P2	413	415	2	2.67	5.12
P3	419	426	7	2.62	5.12

^aIP was obtained by the ferrocene standard.

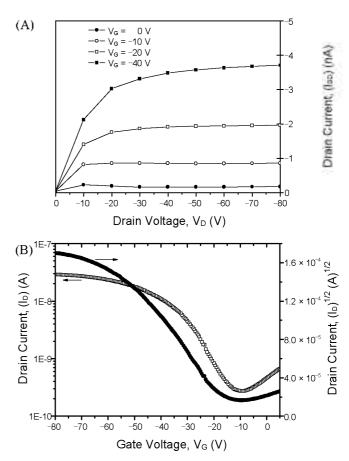


Figure 3. Output characteristics of the Pl device at different gate biases (A) and transfer curves at constant $V_D = -60$ V for devices using Pl (B).

where $I_{\rm D}$ is the drain current in the saturated region, W and L are the channel width and length, respectively, u is the field-effect mobility, C_1 is the capacitance per unit area of the gate dielectric layer, and V_0 and $V_{\rm T}$ are the gate and threshold voltages, respectively. The saturation mobilities of P1, P2, and P3 were found to be 0.81×10^{-3} , 1.4×10^{-4} , and 9.0×10^{-5} cm² V⁻¹ s⁻¹, respectively, and the on/off ratio was about 10^2 for all these polymers. The dimeric phenoxazine-fluorene and bithiophene alternative copolymers showed a relatively low mobility compared to the homopolymer, whereas P1 showed the highest field-effect mobility among the studied polymers.

To investigate the structural characteristics and the surface morphology of the thin-film state. X-ray diffraction (XRD) analysis and atomic force microscopy (AFM) experiments were performed. No reflection peaks were observed in any of the thin-film XRD patterns obtained for P1. P2. and P3 (data not

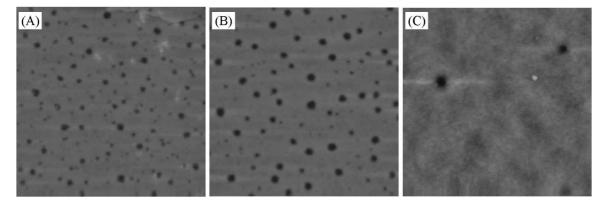


Figure 4. Atomic force microscopy height images for P1 (A), P2 (B) and P3 (C) thin films spin-coated onto OTS treated SiO₂ substrate. (Image size: $3 \ \mu m \times 3 \ \mu m$).

shown here). These findings indicate that the P1, P2, and P3 films are amorphous collections of randomly oriented polymer chains. The AFM height images of the polymers, obtained on oc-tyltrichlorosilane (OTS-8) modified wafer substrates, are shown in Figure 4. In all cases, the images indicate a smooth and flat surface with a root-mean-square (RMS) surface roughness of 2.04 - 2.26 nm. These results indicate that all the polymer thin films are amorphous, and thus, carrier transportation is attributed to hopping between the polymer chains. These results suggest that the rigid-planar phenoxazine groups play a major role in increasing the carrier mobility due to efficient π - π stacking.

Conclusions

In summary, we have synthesized solution-processable polymers containing dimeric aryl-phenoxazine groups as semiconducting materials and have investigated their physical behavior and FET characteristics. XRD and AFM studies demonstrated that the synthesized polymers exhibit amorphous behavior. The field-effect mobilities of P1, P2, and P3 were found to be 0.81×10^{-3} , 1.4×10^{-4} , and 9.0×10^{-5} cm² V⁻¹s⁻¹, respectively, and the on/off ratio was approximately 10^{2} for all the studied polymers. The FET mobility of the phenoxazine homopolymer (P1) exhibited the highest value, which may be due to an increment in the π - π stacking upon incorporation of the rigid-planar phenoxazine groups.

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