

Fabrication of Poly(3-hexylthiophene) Thin Films by Vapor-Phase Polymerization for Optoelectronic Device Applications

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ABSTRACT The vapor-phase polymerization (VPP) of poly(3-hexylthiophene) (P3HT) was achieved successfully as an alternative method to conventional solution-based thin film fabrication. Using $\text{Fe}^{\text{III}}\text{Cl}_3 \cdot 6\text{H}_2\text{O}$, a spontaneous reaction of 3-hexylthiophene monomers resulted in the rapid formation of conducting P3HT thin films directly on substrates, such as glass, indium–tin-oxide, and poly(ethylene terephthalate), at thicknesses ranging from 50 to 1000 nm. The VPP of P3HT was achieved using ferric chloride hexahydrate and a 1:1 ratio of a methanol/ethanol mixture as the solvent system. The developed VPP technique can provide good processing consistency with an electrical conductivity, a transmittance, and a surface roughness of $\sim 10^{-2}$ S/cm, $>90\%$, and <10 nm, respectively.

KEYWORDS: poly(3-hexylthiophene) • vapor-phase polymerization • conducting polymers • thin films • electronic devices

INTRODUCTION

Since π -conjugated polymers were first reported (1, 2), they have been studied extensively primarily for their high conductivity and potential applications in semiconductors of electronic devices. For example, polythiophenes (PTs) (2) and poly(3-alkylthiophene)s (P3ATs) (3–5) with a higher regioregularity (2, 6) have been examined for use in light-emitting diodes (LEDs), field-effect transistors (FETs), all-polymer integrated circuits, and thin-film transistors (TFTs) (7–13). Similarly, photovoltaic cells have adopted P3ATs as an electron-donor layer because they are one of the most promising active components for this application (14, 15). Furthermore, P3AT polymer lasers are attractive devices because of their light weight, flexibility, and low-cost fabrication (16).

Among the P3ATs, such as poly(3-octylthiophene) and poly(3-dodecylthiophene) (3, 4), poly(3-hexylthiophene) (P3HT) is used most widely because of its low cost and superb properties. P3HT is the best of the P3ATs, even though it is either annealed to improve the degree of crystallinity or exposed to oxygen (4, 5). Depending on the

doping level, P3HT thin film has many potential uses in applications such as biosensors, wettability switch devices, flexible electronic displays, microchips, electrochromic windows, and radiation screens (17–20). P3HT is also one of the most indispensable materials in organic TFTs, providing solution-processable capability and relatively high mobility ($0.1 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$) (11, 21). In particular, in organic solar cells, P3HT has recently been used as an active matrix material incorporated with CuPc, TiO_2 , perylene, or fullerene (C_{60}) derivatives, such as methanofullerene and phenyl C_{61} -butyric acid methyl ester (14, 15).

P3HT is usually synthesized in powder form by chemical oxidative polymerization, electrochemical polymerization, Grignard polycondensation, oxidative coupling, and quasi-living polymerization (22–26). However, there are several drawbacks to be addressed with this polymer. For example, the light transmittance is not usually enhanced up to 90% and the surface morphology of fabricated P3HT films is generally poor because film formation is usually achieved by a wet-coating process, which is also limited by the wetting characteristics of the P3HT solution with the substrate materials (27, 28).

This study investigated P3HT thin films deposited using a vapor-phase polymerization (VPP) technique (29, 30), which desirably ensures thin-film formation in various substrate materials without the additional processes to liquefy polymers. To the best of the authors' knowledge, there are no reports showing the successful VPP of P3HT even though this method is simple, easy to use, and inexpensive. It is believed that the VPP technique for P3HT will allow the

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fabrication of thin coatings over a large surface area of various substrate materials. Although organic active materials are sensitive to water and oxygen during their long-term use, it may be more important to adopt an easy process for commercializing electronic devices than to enhance those properties that may be resolved in the future (31–33).

In this study, the appropriate fabrication conditions for the VPP of P3HT on various substrate materials were determined. It was found that the component ratio of the solvents and the pretreatment conditions of the substrates were the key issues for the successful VPP of P3HT. It was also found that choosing an appropriate iron(III) solution was the most important factor. Among the various dopant materials, only ferric chloride hexahydrate ensured high-quality thin films of vapor-phase-polymerized P3HT. It is believed that this method is easy to use and ensures high-quality thin coatings on a variety of substrate materials.

EXPERIMENTAL METHODS

The main chemicals used in this study were 3-hexylthiophene (3HT; 99%, Aldrich, St. Louis, MO), iron(III) chloride hexahydrate ($\text{Fe}^{\text{III}}\text{Cl}_3 \cdot 6\text{H}_2\text{O}$; 97%, Aldrich, St. Louis, MO), anhydrous methyl alcohol (MeOH; 99.9%, Carlo Erba Reagenti, Rodano, Italy), and anhydrous ethyl alcohol (EtOH; 99.9%, Carlo Erba Reagenti, Rodano, Italy). Indium–tin-oxide glass (ITO glass; 10 Ω , 185 \pm 20 nm ITO, 1.1 mm ITO glass, UID, Korea), plane glass (1.1 mm, Paul Marienfeld GmbH & Co. KG, Lauda-Koenigshofen, Germany), and poly(ethylene terephthalate) (PET; 100 μm , HwaSung Co. Ltd., Seoul, Korea) were used as substrate materials in this study.

The substrates were washed and rinsed with deionized water and acetone while being sonicated for 10 min to remove any organic contaminants. The glass substrates were plasma-treated (KSC Korea switching, Kyungki-Do, Korea) for 10 min (10 kHz, 10 V, and 7 A at a speed of 50 cm^3 of helium/min), and the ITO glass substrates were ultraviolet (UV)-treated for 20 min. The iron(III) solution used in this study was a mixture of MeOH and EtOH at a 1:1 ratio with 5 wt % $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$. After the iron(III) solution was sonicated for 2 min at 40 $^\circ\text{C}$, it was spin-coated onto the substrates at a speed of 500 rpm for 5 s and then at 1400 rpm for 5 s. Subsequently, the substrates coated with an iron(III) solution were placed in the VPP chamber containing 3HT monomers to evaporate and fill therein under a nitrogen purge, which was similar to that reported elsewhere (27, 28). The 3HT monomers in the VPP chamber were polymerized for 1 h at 50 $^\circ\text{C}$. The sample was soaked and washed sequentially with MeOH to eliminate iron(III) remaining on the substrate. The washed P3HT film was further dried for 10 min in an ambient atmosphere to remove the residual solvents.

The contact angles were measured using a Digi-drop (Sin Jin Tech, Kyonggi-do, Korea). The thickness of the vapor-phase-polymerized P3HT was measured by field-emission scanning electron microscopy (FE-SEM; JEOL JSM-7000F FESEM, voltage of 5.0 kV) and α -step IQ (KLA-Tencor Corporation, The Yield Management Co., San Jose, CA). The highest occupied molecular orbital (HOMO) was measured using a UV photoelectron spectrometer (UPS; surface analyzer model AC-2, Riken-Keiki Co., Tokyo, Japan) (9, 34), and the band gap was determined by UV–vis–NIR spectrophotometry (UV-3600, Shimadzu, Kyoto, Japan) (2, 35, 36). The electrical conductivity of the P3HT films was measured using a four-point probe (Jandel Engineering Ltd., Beds, U.K.) connected to a Keithley 2400 source meter, where the probe was equipped with four spring-loaded tungsten carbide needles spaced 1 mm apart, and two-point probe methods (U1252A, Agilent Technologies, Santa Clara, CA). The

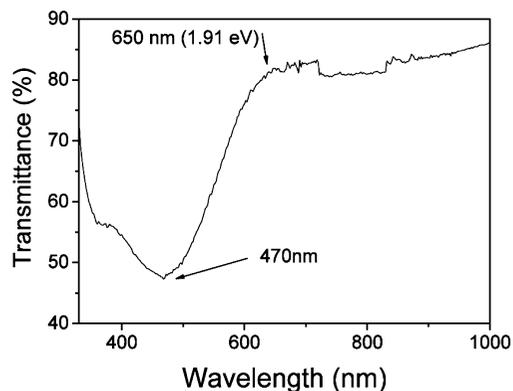


FIGURE 1. UV–vis spectrum of the P3HT thin coating formed at 50 $^\circ\text{C}$ for 1 h.

conductivity of the P3HT coating on the glass plate was calculated from the surface resistivity and film thickness. Atomic force microscopy (AFM; Nanoscope IIIa, Digital Instruments, Plainview, NY) was used to examine the surface morphology of the P3HT coating, where a 5 $\mu\text{m} \times 5 \mu\text{m}$ area was analyzed to determine the surface roughness in all cases. The molecular weight of vapor-phase-polymerized P3HT was measured by matrix-assisted laser desorption/ionization mass spectrometry (MALDI-MS; Voyager-DE STR biospectrometry Workstation, Applied Biosystems Inc., Foster City, CA) equipped with a dinitrogen UV laser radiating at 337 nm wavelength using tetrahydrofuran (THF) as the solvent and dithranol as the matrix.

RESULTS AND DISCUSSION

For the VPP of P3HT, $\text{Fe}^{\text{III}}\text{Cl}_3 \cdot 6\text{H}_2\text{O}$ was chosen as the iron(III) solution, which is commercially available and behaves as an oxidant with a relatively high vapor pressure. Iron(III) tosylate and other iron(III) materials were also attempted, but the VPP of P3HT was unsuccessful, which is in contrast to the case using ferric salts of organic sulfonates for poly(3,4-ethylenedioxythiophene) (37). It should be noted that the expected life span of the $\text{Fe}^{\text{III}}\text{Cl}_3 \cdot 6\text{H}_2\text{O}$ solution was less than 3 days in air.

Figure 1 shows the UV–vis transmittance spectra of a vapor-phase-polymerized P3HT film formed on a glass substrate after being polymerized at 50 $^\circ\text{C}$ for 1 h. Taking the isosbestic point at 650 nm as the energy gap, the band gap was estimated to be 1.91 eV, which is similar to that of conventional PTs corresponding to the 70% regioregular head-to-tail (HT) P3HT case (35, 36). The measured value in this study is believed to be a proper band gap in organic LEDs (OLEDs) and photovoltaic cells (8, 15). The regioregularity of P3AT is also considered to be extremely important for the manifold properties, such as conductivity and the extent of crystal growth (2, 6, 36). The regioregular HT P3ATs form well-defined and well-organized 3D structures in the form of π stacks, which usually lead to both better material characteristics and enhanced device performance in almost all categories ranging from electrical conductivity to stability (2, 6). The minimum peak (λ_{min}) at 470 nm in Figure 1 indicates the regioregularity of P3HT corresponding to a 70% rr-P3HT of the π – π^* transition (35, 36).

Figure 2A shows a vapor-phase-polymerized P3HT coating on glass with a coating thickness, conductivity, and

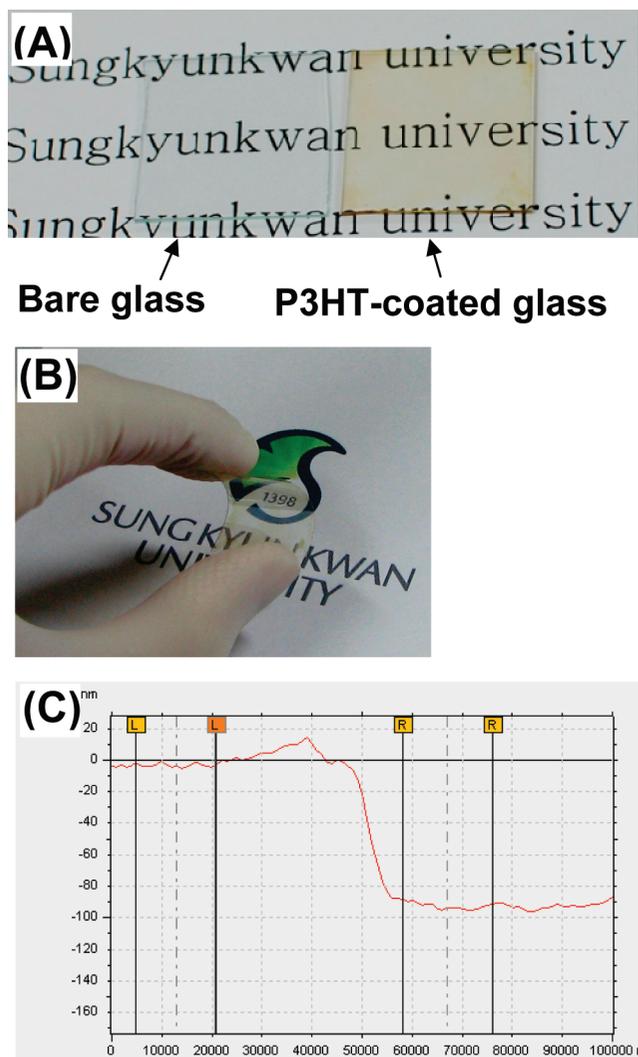


FIGURE 2. Images and an α -step image of P3HT polymerized at 50 °C for 1 h: (A) comparison between vapor-phase-polymerized P3HT on glass and bare glass; (B) vapor-phase-polymerized P3HT coating with 90% optical transmittance on a flexible PET film; (C) α -step image showing the thickness and morphology. Low levels were scraped off by a pincette.

transmittance in the visible light between 400 and 800 nm of 100 nm, 10^{-5} S/cm, and 87%, respectively. Figure 2B shows the vapor-phase-polymerized P3HT coating on a PET substrate, demonstrating that this method can be used in flexible electronic devices. Figure 2C shows an example of the coating thickness measurement for a vapor-phase-polymerized P3HT coating on ITO, giving a coating thickness of 95 nm.

Figure 3A shows the solubility tests of P3HT, monomer (3-HT), and iron(III) solution, demonstrating that VPP was successfully carried out in this study. Figure 3A, parts a–c, shows that the vapor-phase-polymerized P3HT coating (200 nm of thickness in a blue color) still remains on the glass as a red-brown layer after being washed with MeOH for 3 h, 1 h, and 5 min, respectively. Because THF is known to be a good solvent of P3HT, Figure 3A, part d, shows that the vapor-phase-polymerized P3HT coating is completely dissolved in THF after 1 min of washing, leaving a yellow solution. Although P3HT is not dissolved in MeOH, the

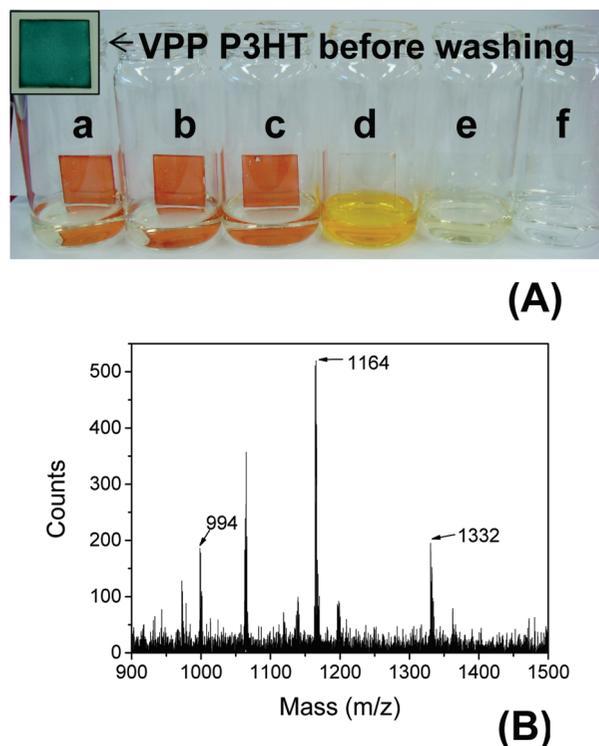


FIGURE 3. (A) Solubility test of a vapor-phase-polymerized P3HT coating on glass after being washed with MeOH for 3 h (a), 1 h (b), and 5 min (c) and washed with THF for 1 min (d), also exhibiting a $\text{Fe}^{\text{III}}\cdot\text{Cl}\cdot 6\text{H}_2\text{O}$ coating (e) and monomer coating (f) on glass after being washed with MeOH for 1 min. (B) MALDI-MS spectrum of vapor-phase-polymerized P3HT at 55 °C for 1 h.

iron(III) or 3-HT monomer is entirely dissolved in MeOH. In order to confirm the solubility of the iron(III) and 3-HT in MeOH, the iron(III) and monomer coatings are shown in Figure 3A, parts e and f, after being washing in MeOH for 1 min. As can be seen, these coatings are immediately dissolved in MeOH. Consequently, the solubility test proves that P3HT was successfully synthesized by VPP in this study because the synthesized P3HT coating remains intact on the glass substrate after being thoroughly washed with MeOH, which may very well remove the iron(III) solution from the coating layer.

Determination of the molecular weights of polymers is almost invariably found by gel permeation chromatography (GPC) using polystyrene standards, which indicates relative molecular weights. Conjugation systems such as PTs, however, are known to have a more rodlike conformation in solution, which tends to overestimate the molecular weights of such systems (38). More specifically, in P3HT systems, the molecular weight by GPC is a factor of 1.2–2.3 times higher than that by MALDI-MS (39). Consequently, MALDI-MS was used in this study in order to determine the molecular weight of vapor-phase-polymerized P3HT. Figure 3B shows the molecular weight of vapor-phase-polymerized P3HT measured by MALDI-MS, providing peaks at around 994, 1166, and 1333 corresponding to six through eight repeat units of 3-HT (0.74 nm) (40), giving chain lengths of 4.44–5.92 nm. This is within the effective conjugation length of P3HT (41) reported differently in different applications as 9–10 thiophene units in polymer LEDs (42) and 5–7

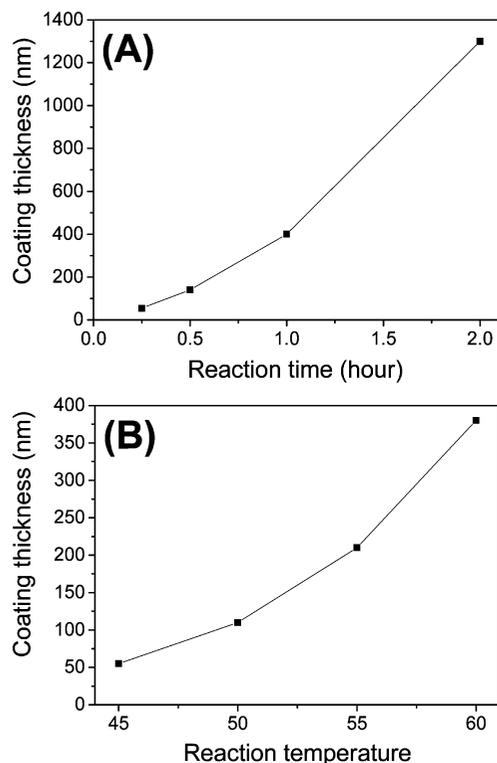


FIGURE 4. Vapor-phase-polymerized P3HT coating thickness as a function of the reaction time at 50 °C (A) and the reaction temperature for a 1 h reaction (B).

thiophene units in FETs (43). To apply the thin vapor-phase-polymerized P3HT coatings to many optoelectronic devices, further work will be needed on controlling the molecular weights of vapor-phase-polymerized P3HT.

The transmittance at 700 nm through a 50-nm-thick vapor-phase-polymerized P3HT sample was measured to be 91 %, showing compatibility with commercially available ITO (ca. 93 % transmittance) optimized for transparent applications, such as organic photovoltaic cells and OLEDs. For a vapor-phase-polymerized P3HT thin coating, the electric conductivity was measured in the range of 10^{-2} – 10^{-4} S/cm for coating thicknesses ranging from 50 to 500 nm. These conductivities are higher than those of other semi-conducting FeCl_3 -doped polymers, which are usually between 10^{-3} and 10^{-9} S/cm (2, 33). A clear dependence of the conductivity on the film thickness was not observed, seemingly depending more strongly on the surface morphology. Additional experiments to optimize the relationship between the conductivity, thickness, and transmittance of vapor-phase-polymerized P3HT are currently underway.

Figure 4 shows the coating thickness as a function of the polymerization time and temperature. As shown in Figure 4A, the coating thickness increased gradually with an increase in the reaction time at 60 °C from 55 nm after 15 min to 1300 nm after 2 h. Similarly, the coating thickness increased with an increase in the polymerization temperature for 1 h, for example, from 55 nm at 45 °C to 380 nm at 60 °C (Figure 4B). It should be noted that the slight accelerating feature of the thickness with time in Figure 4A likely stems from the fact that it is needed for the 3HT vapor

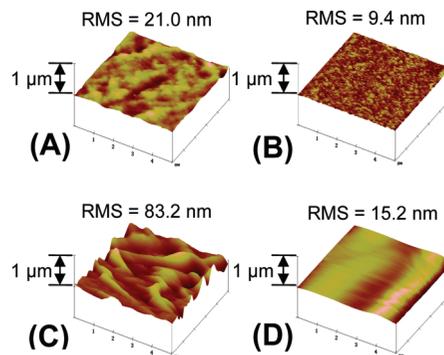


FIGURE 5. AFM topography images of the thin P3HT coatings with $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ on the glass substrate in cases of 5% EtOH/MeOH (A, before washing; B, after washing), 20% of the same solution with washing (C), and a 5% solution including isopropyl alcohol (D).

to be completely saturated in the chamber, especially the early stage of the experiments.

Surface morphology is important when P3HT is used for thin coating applications because it influences a large number of properties, such as double-layer capacitance and adhesion (27, 28). It was reported that the polymerization rate of conjugated polymers, surface treatment, and iron(III) solution removal conditions substantially affect the surface morphology of thin film coatings (27, 28). For example, Winther-Jensen et al. (30) made a variety of attempts to remove the residual iron(III) and monomers successfully in order to make the surfaces of thin conducting polymers smoother. Figure 5 shows the surface morphology of the P3HT thin coating for different types of solvents for iron(III) and washing conditions. Parts A and B of Figure 5 show the coating surfaces before (A) and after (B) the washing steps for 5% $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in EtOH/MeOH, respectively. The washing process improved the root-mean-square (rms) roughness from 21.0 to 9.4 nm. In general, the surface roughness of P3HT thin film coatings should not exceed ca. 10 nm for most optoelectronic applications (27), which was satisfied in this study. Figure 5C shows the coating surface after removal of iron(III) with EtOH/MeOH. The surface roughness of the coating increased with an increase in the iron(III) concentration, as shown in Figure 5B,C, giving a rms roughness as 9.47 and 83.2 nm for 5% and 20% $\text{Fe}^{\text{III}}\text{Cl} \cdot 6\text{H}_2\text{O}$, respectively. Although the result is not included here, the rms roughness is increased with an increase in the polymerization time or temperature, mainly because of the increased polymerization rate. It should be noted that other $\text{Fe}^{\text{III}}\text{Cl}$ solutions incorporating isopropyl alcohol, propanol, or butanol provided rougher surfaces than the MeOH/EtOH mixture, one of which is shown in Figure 5D for the isopropyl alcohol case. On the other hand, thicker films tend to have higher electric conductivity, which is strongly dependent on the surface roughness. When the thickness of P3HT was more than 300 nm, the surface appeared rougher, making it quite difficult to remove the remaining iron(III) from the coating formed at high concentrations. Moreover, a long polymerization time at low concentration produced a better surface morphology than a short polymerization time at high concentrations, even though the film thicknesses are the same.

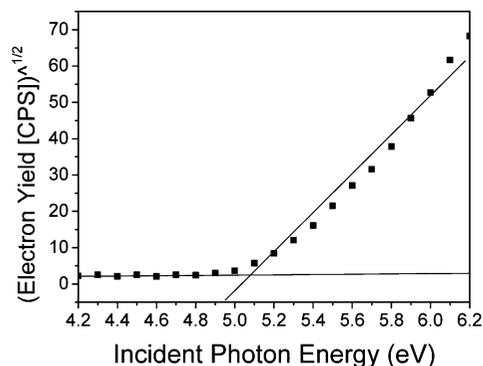


FIGURE 6. HOMO of a P3HT thin coating on an ITO glass substrate.

The HOMO of the vapor-phase-polymerized P3HT coating was approximately 5.07 eV (Figure 6), which is similar to previous studies on OLEDs and solar cells (8, 15). Because the HOMO of P3HT can be adjusted using techniques such as thermal annealing, the HOMO of the vapor-phase-polymerized P3HT in this study can be adjusted for applications in a variety of optoelectronic devices to meet different electron-donor materials.

CONCLUSION

A conducting P3HT thin coating was successfully fabricated on various substrates with a thickness ranging from 50 nm to 1 μm by VPP of a 3HT monomer. The most appropriate iron(III) solution was found to be 5% $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in MeOH/EtOH (1:1), providing good conductivity (10^{-2} – 10^{-4} S/cm) and transmittance (<91%). As anticipated, the coating thickness increased with an increase in the reaction time and temperature, but the relationship between the conductivity and other conditions, such as the coating thickness, was not clarified because of the rugged surface. The rms roughness of vapor-phase-polymerized P3HT was 9.4 nm. The band gap and HOMO of vapor-phase-polymerized P3HT were found to be 1.91 eV (650 nm) and 5.07 eV, respectively, which may be suitable for applications in optoelectronic devices.

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