



Original Article

Synthesis and Properties of Some Polythiophenes Containing Benzo[*d*]thiazole Heterocycle

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Abstract: In this paper, some new polythiophenes containing benzo[*d*]thiazole heterocycle were synthesized by oxidative polymerization in chloroform using iron(III) chloride as a catalytic oxidant. Infrared (IR) spectroscopy and ultraviolet-visible (UV-Vis) spectroscopy analyses confirmed that the polymerization reaction successfully took place and the absorption band at 497 nm is typical for π - π^* transition in the conjugated polythiophenes. The polymers are composed of relatively uniform particles using field emission scanning electron microscopy (FESEM) method. Based on thermal gravimetric analysis (TGA), the polymers had average thermal stability in the atmosphere when decomposed completely at 500 °C. The polymers exhibited fluorescence emission at about 516 nm due to the conjugated chain in the polymers.

Keywords: Polythiophene, benzo[*d*]thiazole, chemical oxidative polymerization, heterocyclic side groups, electrical conductivity.

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1. Introduction

In recent years, polythiophenes containing heterocycles have had many advanced applications based on their high environmental sustainability, structural flexibility, optical stability, and electrochemical characteristics [1-5]. They are potential functional materials used for organic field-effect transistors [6, 7], organic light-emitting diodes [8, 9], organic photovoltaic cells [10], and other optoelectronic devices [11, 12]. Among these, benzothiazole-based polythiophenes have much attractive by their wide range of biological activities [13, 14]. In addition, functional groups on the side chains can also affect the structure, electrochemical and optical characteristics of polythiophenes [15]. As a result, it is interesting to prepare the polymers from thiophene monomers having benzo[d]thiazole heterocycle. A novel poly 3-(benzothiazole-2-yl) thiophene-based conductive polymer has been synthesized by chemical and electrochemical polymerization [16]. This polymer was studied for its photoabsorption and photoluminescence characteristics and was applied in polymeric light-emitting diodes. Some synthetic methods developed for preparing 3-(benzothiazol-2-yl) thiophene are available in which a mixture of thiophene-3-carbaldehyde and *o*-aminothiophenol are refluxed in ethanol [17] or a mixture of 3-bromothiophene, 2-chlorobenzothiazole, and magnesium turnings are refluxed in diethyl ether [18].

Herein, the synthesis and fluorescent properties of new polythiophenes containing the benzo[d]thiazole heterocycle on the side chain are presented. These polythiophene derivatives were synthesized by polymerization of monomers, which were synthesized from a solvent-free microwave-assisted reaction of the starting materials without catalyst in a short time, leading to a simple purification and a high yield [19]. Among the obtained polymers, two have a new structure with polythiophene containing a benzothiazole heterocycle attached to a benzo[d]oxazole heterocycle. The morphology, thermal stability, optical, and

conductivity features of the obtained polythiophenes were studied and discussed.

2. Experimental

2.1. Chemicals and Methods

The starting materials containing (3-thiophenecarboxaldehyde (Sigma Aldrich), methanol (Merck), and *o*-aminothiophenol (Merck) were used without further purification.

The melting points of polymers were measured using a Gallenkamp Melting Point Apparatus (England). The IR spectra of polymers were recorded using a Nicolet Impact 410 FTIR Spectrometer (USA). The nuclear magnetic resonance (NMR) spectra of the polymers were recorded on a Bruker XL-500 Spectrometer (USA) at 500 MHz using DMSO-*d*₆ and CDCl₃ as solvents. The thermogravimetric analysis/differential thermal analysis (TGA/DTA) of the polymers was determined on a DTG-60/60H Shimadzu Simultaneous Measuring Instrument under the atmosphere with a heating rate of 10 °C/minute. The scanning electron microscopy (SEM) analysis was performed using an S-4800 SEM instrument (Hitachi, Japan). The UV-Vis spectra of the polymers were recorded using a UV-visible spectrophotometer (Jasco V670, Japan). The photoluminescence emission spectra of polymers were measured on a Horiba Fluoromax Series (USA).

2.2. Synthesis of Polythiophenes Containing Benzo[d]thiazole Heterocycle

2.2.1. Synthesis of Monomers Containing Benzo[d]thiazole Heterocycle (1, 2 and 3)

Synthesis of 3-(2-benzothiazolyl)thiophene (1)

A reaction mixture of 3-thiophenecarboxaldehyde (1 mmol) and *o*-aminothiophenol (2 mmol) was heated in a domestic microwave at 400 W.

After each minute of the reaction time, the reaction mixture in a sealed 250 mL flask was cooled to room temperature in the atmosphere for 2 minutes. The reaction progress was

monitored with thin-layer chromatography (TLC) using a hexane–ethanol mixture (1:1 v/v) as the developing solvents. The reaction was completed in 4 minutes. The reaction mixture was cooled to room temperature and then added to a cold *n*-hexane–ethyl acetate mixture (5:1 v/v) to obtain a solid product, which was further crystallized in the same solvent mixture to give 0.19 gram (yield of 87%) of the product (**1**) in yellow crystal (m.p. of 113 °C). IR (Nicolet Impact 410 FT–IR, KBr, cm⁻¹): 3067 (C–H), 1634 (CH=N), 1581 (C=C). ¹H-NMR [Bruker XL-500, 500 MHz, DMSO-*d*₆, ppm, *J* (Hz)]: 8.36 *dd J* = 1 Hz, *J* = 2.5 Hz (H²); 7.72 *dd J*₂₋₄ = 1.0 Hz, *J*₅₋₄ = 5.0 (H⁴); 7.77 *dd J*₂₋₅ = 2.5, *J*₄₋₅ = 5.0 (H⁵); 8.02 *d J*₁₁₋₉ = 1.0, *J* = 8.0 Hz (H⁹); 7.52 *td J* = 1.0 Hz, *J* = 7.5 Hz, *J* = 8.0 Hz (H¹⁰); 7.44 *td J* = 1.0 Hz, *J* = 7.5 Hz, *J* = 8.0 Hz (H¹¹); 8.11 *dd J* = 1.0 Hz, *J* = 8.0 Hz (H¹²). ¹³C-NMR [Bruker XL-500, 500 MHz, DMSO-*d*₆, ppm]: 127.5 (C²), 135.1 (C³), 126.1 (C⁴), 128.3 (C⁵), 162.1 (C⁶), 134.1 (C⁷), 153.3 (C⁸), 122.5 (C⁹), 126.5 (C¹⁰), 125.3 (C¹¹), 122.2 (C¹²).

Synthesis of 5-(benzo[d]thiazol-2-yl)-7-methoxy-2-(thiophen-3-yl)benzo[d]oxazole (**2**)

A mixture of 3-thiophene carboxaldehyde (1 mmol), 2-amino-4-(benzo[d]thiazol-2-yl)-6-methoxy phenol hydrochloride (1 mmol), and acetic acid (2 mL) was heated in a domestic microwave at 400 W. The mixture was cooled down after each minute. The reaction progress was monitored with TLC using a hexane–ethanol mixture (1:1 v/v). The reaction was performed within 17 minutes. The reaction mixture was cooled to room temperature and dissolved in an ethanol-water mixture (4:1 v/v) to obtain a solid product. It was further crystallized in the same solvent mixture to give 0.31 grams (yield of 85%) of the product (**2**) in grey-black powder (m.p. of 197 °C). IR (cm⁻¹): 3083 (C–H alkene), 2954, 2923 (C–H alkane), 1621 (CH=N), 1575, 1508 (C=C). ¹H-NMR [DMSO-*d*₆, ppm, *J* (Hz)]: 8.55 *d J* = 2 Hz (H²); 7.77 *m* (H⁴); 7.38 *dd J*₂₋₅ = 3.0, *J*₄₋₅ = 5.0 (H⁵); 8.00 *d J*₁₁₋₉ = 1.0 (H⁹); 7.75 *dd* (H¹¹); 8.09 *d J* = 8.5 (H¹⁶); 7.56 *t J* = 7.5 (H¹⁷); 7.47 *t J* = 7.5 (H¹⁸); 8.15 *d J* = 8.0 (H¹⁹); 4.14 *s*

(H²⁰). ¹³C-NMR [500 MHz, DMSO-*d*₆, ppm]: 130.2 (C²), 130.7 (C³), 126.2 (C⁴), 128.8 (C⁵), 160.2 (C⁶), 140.7 (C⁷), 143.5 (C⁸), 111.2 (C⁹), 127.6 (C¹⁰), 106.8 (C¹¹), 144.7 (C¹²), 167.0 (C¹³), 134.6 (C¹⁴), 153.4 (C¹⁵), 122.8 (C¹⁶), 126.6 (C¹⁷), 125.5 (C¹⁸), 122.2 (C¹⁹), 56.4 (C²⁰).

Synthesis of 5-(benzo[d]thiazol-2-yl)-2-(thiophen-3-yl)benzo[d]oxazole (**3**)

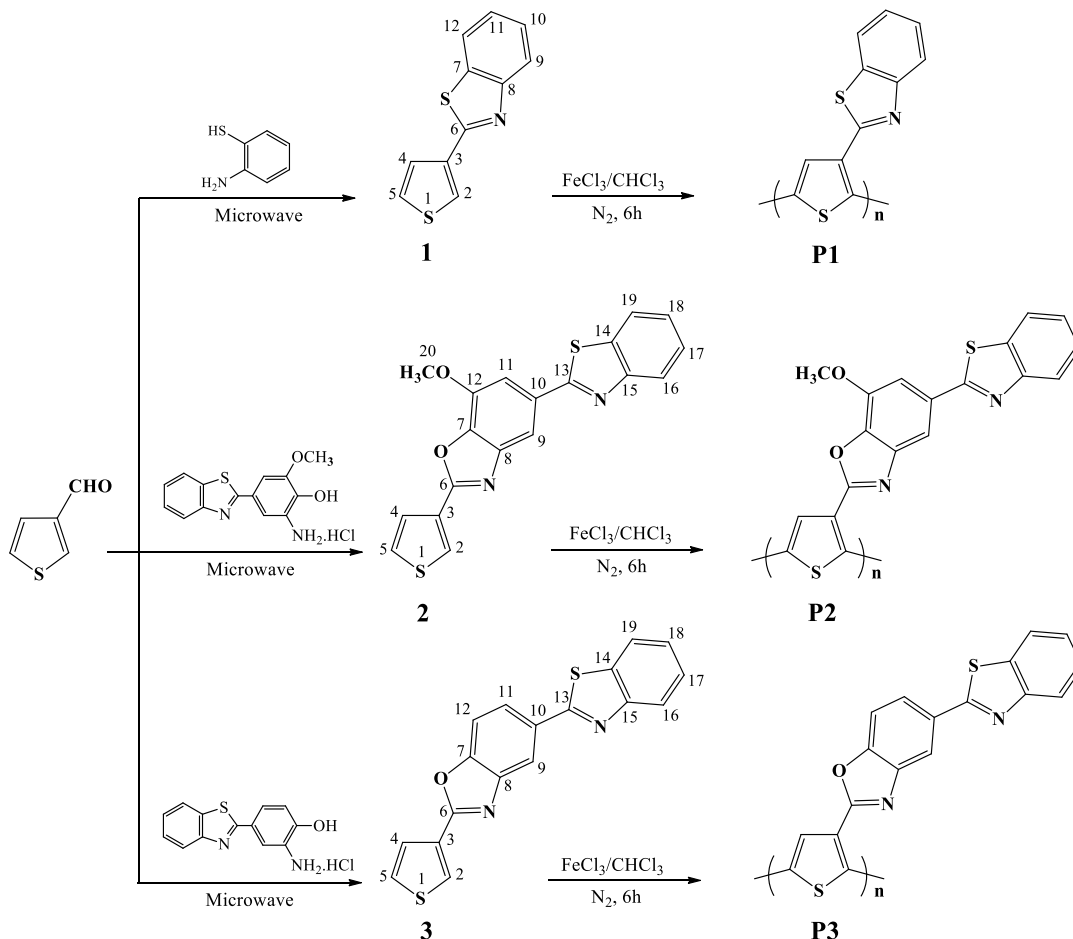
A reaction mixture of 2-amino-4-(benzo[d]thiazol-2-yl)phenol hydrochloride (1 mmol), 3-thiophene carboxaldehyde (1 mmol), and acetic acid (2 mL) was heated for 14 minutes in a domestic microwave at 400 W. The reaction mixture was cooled to room temperature and then dissolved in an ethanol-water mixture (4:1 v/v) to obtain a solid product which was further crystallized in the same solvent mixture to give 0.29 grams (yield of 87%) of the product (**3**) in pale-grey powder (m.p. of 213 °C). IR (cm⁻¹): 2926 (C–H), 1623 (CH=N), 1581, 1515 (C=C). ¹H-NMR [500 MHz, DMSO-*d*₆, ppm, *J* (Hz)]: 8.25 *m* (H²); 7.82 *d J*₅₋₄ = 5.0 (H⁴); 7.47 *dd J*₂₋₅ = 3.0, *J*₄₋₅ = 5.0 (H⁵); 8.42 *d J*₁₁₋₉ = 1.0 (H⁹); 8.18 *dd J* = 1.5, *J*₁₂₋₁₁ = 7.5 (H¹¹); 7.65 *d J*₁₁₋₁₂ = 7.5 (H¹²); 8.09 *d J*₁₇₋₁₆ = 7.0 (H¹⁶); 7.51 *dd J*₁₆₋₁₇ = 7.5, *J*₁₈₋₁₇ = 8.0 (H¹⁷); 7.39 *dd J*₁₉₋₁₈ = 7.5, *J*₁₇₋₁₈ = 8.0 (H¹⁸); 7.39 *d J*₁₈₋₁₉ = 7.5 (H¹⁹). ¹³C-NMR [500 MHz, DMSO-*d*₆, ppm]: 128.7 (C²), 128.8 (C³), 126.7 (C⁴), 127.2 (C⁵), 160.9 (C⁶), 152.1 (C⁷), 142.7 (C⁸), 119.3 (C⁹), 130.8 (C¹⁰), 124.8 (C¹¹), 111.0 (C¹²), 167.6 (C¹³), 135.2 (C¹⁴), 154.2 (C¹⁵), 123.2 (C¹⁶), 126.4 (C¹⁷), 125.2 (C¹⁸), 121.6 (C¹⁹).

2.2.2. Synthesis of Polythiophenes Containing Benzo[d]thiazole Heterocycle P1-P3

The polymers were synthesized by oxidative coupling polymerization method utilizing anhydrous chloroform using four molar equivalents of anhydrous ferric chloride. FeCl₃ (4 mmol) was stirred in chloroform (40 mL) for 15 minutes under a nitrogen atmosphere. Afterward, the solution containing one mmol of monomer in 20 mL of chloroform was prepared and added slowly to the solution

containing FeCl_3 . The polymerization mixture was further stirred at room temperature for 6 hours under a nitrogen atmosphere. The precipitate was filtered and washed with deionized water and methanol.

The residual iron (III) chloride and oligomers were removed from the polymer by Soxhlet extraction with methanol for 48 hours to get a brown-red-colored powder (yield of 68% (**P1**), 60% (**P2**) and 62% (**P3**)).



Scheme 1. Monomer and polymer synthesis reactions.

3. Results and Discussion

3.1. FT-IR Spectra

The monomer was chemically polymerized using FeCl_3 as an oxidant to synthesize the corresponding polymer **P3**. The structure and formation of conjugated- π polythiophene of polymer were determined based on FT-IR spectra.

The synthesized polymer **P3** exhibits absorption bands in the original locations as its

precursor monomer (Figure. 1). For example, in the IR spectrum of polymer **P3**, a high intensity peak at 1633 cm^{-1} is assigned to the formation of a new conjugated $\text{C}=\text{C}-\text{C}=\text{C}$ in the polymer. The bands at 1468 cm^{-1} and 1437 cm^{-1} correspond to carbon-carbon asymmetric vibrations in the thiophene ring. The peak at 729 cm^{-1} is attributed to carbon-carbon out-of-plane bending vibrations in the aromatic ring. The bands at 1297 cm^{-1} and 1070 cm^{-1} are attributed to C-C and C-S vibrations,

respectively. However, the wide of bands are extended compared to those of monomers. It depended on the wide chain dispersity of the resulting oligomers and polymers [20]. The bands at about 3087–2923 cm⁻¹ do not appear in the IR spectrum of polymer **P3**. It indicates that the C–H bonds of the monomer **3** have disappeared. During the free radical polymerization, the thiophene ring unchanges and the polymerization reaction takes place at the C-2 and C-5 positions of the thiophene ring [21, 22].

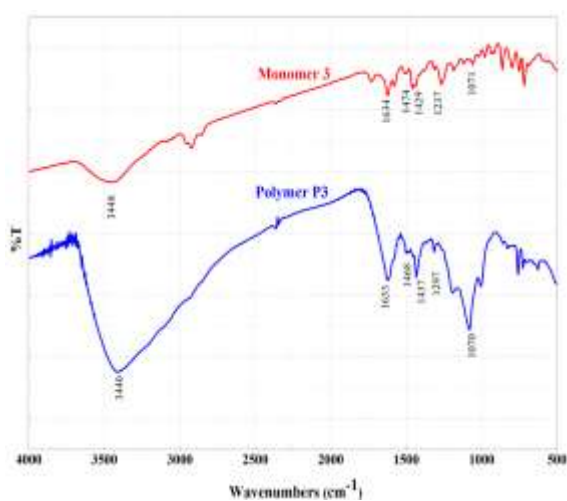


Figure 1. IR spectra of monomer **3** and polymer **P3**.

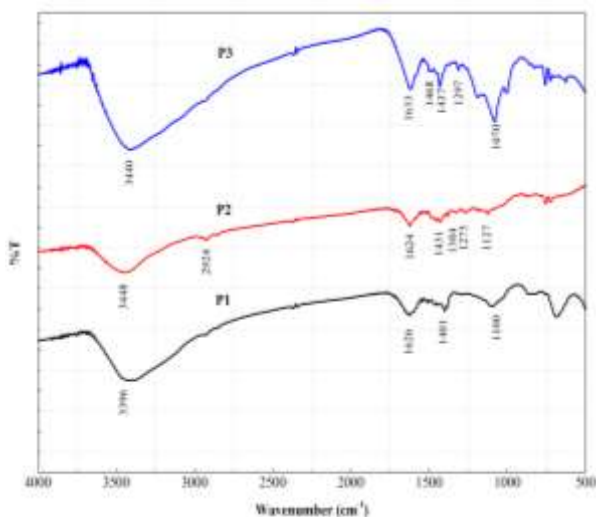


Figure 2. FT-IR spectrum of polymers **P1-P3**.

Figure 3 and Table 2 present the TGA diagrams and thermal parameters of polymers **P1-P3**. The polymers **P2** and **P3** have medium thermal stability in the atmosphere with a completed degradation temperature of about 525–550 °C. On the other hand, the polymer **P1** has low thermal stability. It can be due to the high number of side groups, increasing the spatial arrangement in the polymer side chains and enhancing the effective π -conjugation. As obtained from the DTG curves, the polymer **P1** had the lowest maximum degradation temperature ($T_{max} = 297$ °C) and the polymer **P2** had the highest T_{max} ($T_{max} = 424$ °C).

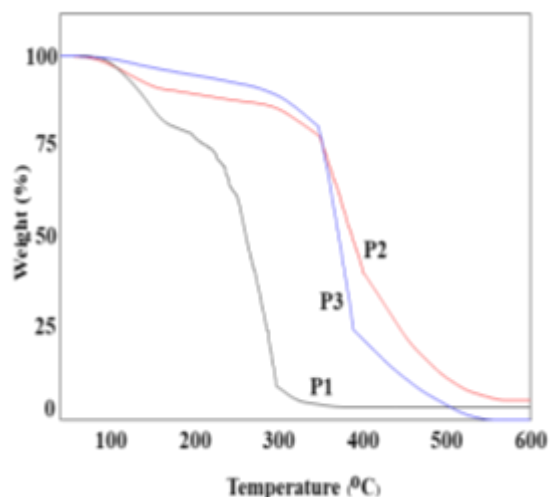


Figure 3. TGA thermograms of polymers **P1-P3**.

Table 2. Thermal parameters of polymers **P1-P3**

	Endothermic temperature (°C)	Complete degradation temperature (°C)	Weight remaining (%)
P1	137; 297	330	3.00
P2	118; 424	550	4.55
P3	120; 404	525	0

Among the 03 synthesized polymers, polymer **P2** has the best thermal stability. It may come from polymer **P2** owing a higher degree of π -conjugation than polymer **P3**.

3.3. Morphology of Polymers

Figure 4 shows the morphology of the synthesized polymers **P1-P3**. The polymers exhibit uniform particles. These particles adhered together during the polymerization. The structures of **P2** and **P3** are tighter than that of **P1**, affecting the thermal stability of these polymers. The **P2** and **P3** polymers are more stable than **P1** as discussed above.

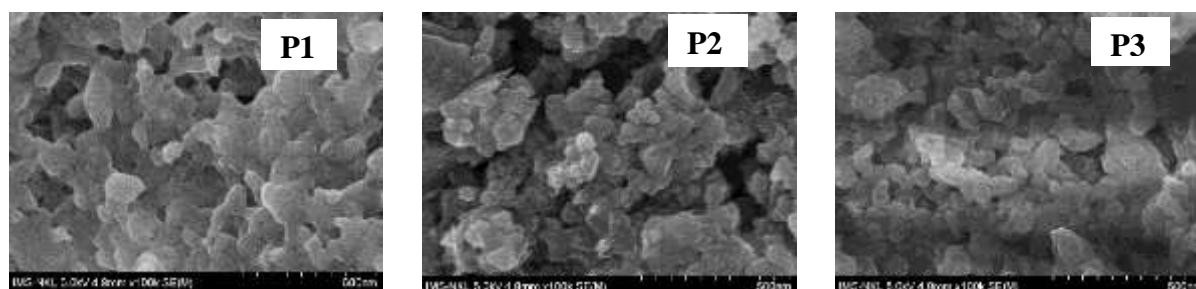


Figure 4. FE-SEM micrographs of polymers **P1-P3**.

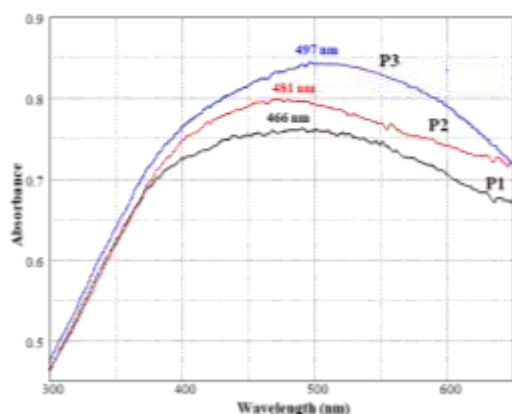


Figure 5. UV-Vis spectra of polymers **P1-P3** in solid film.

3.5. Photoluminescence Spectra of Polymers

Figure 6 presents the photoluminescence spectra of polymers **P1-P3**. Polymer **P3** has the maximum photoluminescence intensity based on longer π -conjugated polythiophene chains. All polymers display maximum fluorescence emission at about 516 nm under 415 nm excitation. The length of the π -conjugated polymer has almost no influence on the photoluminescence wavelength of the polymers.

3.4. UV-Vis Spectra of Polymers

Figure 5 shows the UV-Vis spectra of the polymers in the solid film. Thin solid films without coating on a glass substrate were directly placed in the sample holder box. In solid films, the polymers present a maximum absorption band at 466–497 nm attributed to $\pi \rightarrow \pi^*$ transition of the π -conjugated polythiophene [23-25].

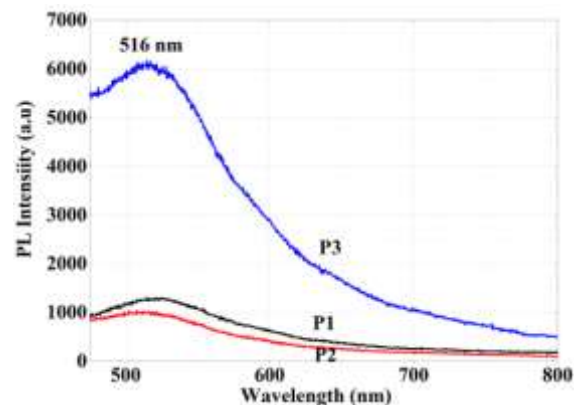


Figure 6. Photoluminescence spectra of polymers **P1-P3**.

4. Conclusion

In this work, a novel series of polythiophenes containing benzo[*d*]thiazole from thiophene-3-carbaldehyde were synthesized by oxidative polymerization with anhydrous FeCl_3 in anhydrous CHCl_3 . The three monomers were synthesized via a microwave-assisted solvent-free reaction without a catalyst in a short time, leading to high conversion yields. The UV-Vis spectroscopy combined with the infrared (IR)

spectroscopy analyses suggest the success of polymerization. The thermal gravimetric analysis indicates that these polymers have medium thermal stability in the atmosphere at about 330-550 °C. The maximum fluorescence emission of these polymers is at about 516 nm, which shows the potential application of the studied polymers for fluorescent dyes or transport materials.

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6. References

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