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Research Article

SYNTHESIS OF NOVEL FLUORESCENT CONJUGATED POLYMERS BASED ON PHENOXAZINE DERIVATIVES AND 9,9-DIOCTYL-9*H*-FLUORENE

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ABSTRACT

In this research, the new fluorescent conjugated polymers based on 10-(Perylene-3-yl-10H-Phenoxazine (PyP) and 9,9-dioctyl-9h-fluorene (PF) have been synthesized via direct arylation polymerization using $Pd(OAc)_2$, PivOH and $PCy_3.HBF_4$ as catalyst system. The monomer based on phenothiazine has been synthesized via C-N coupling to obtain the 4-(10H-phenothiazine-10-yl)-N,N-diphenylaniline (PyP). The synthesized novel conjugated polymers based on PyP and PF have been characterized via ^{1}H NMR, GPC, FTIR, DSC, XRD, PL, and UV-Vis spectrum. The obtained conjugated polymers exhibited a narrow bandgap of 2.85 eV and polymer exhibited the fluorescent emission at 350 nm. Based on the optical properties of PFPyP polymer, PFPyP can be applied for organic solar cell and fluorescent chemosensor devices.

Keywords: conjugated polymers; Direct arylation polymerization; organic solar cells

1. Introduction

In the last few years, one pressing concern in anti-terrorism, global security, and military issues is explosive detection. The reliable and accurate detection of explosives is an issue of global concern. The recent increase in global terrorism has stimulated the necessity for the followed methods to detect explosive should be remote, sensitive and low-cost. For all these reasons, training canines (Furton, & Myers, 2001), metal detection, ion mobility spectrometry (IMS) (Eiceman & Stone, 2004; Griffin et al., 2005), mass spectrometry (Hakansson et al., 2000) and X-ray imaging (Hallowell, 2001) have been used or proposed as appropriate methods for the detection. Trained canines are reliable for

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the detection of explosives due to the powerful olfactory system of dogs. Well-trained canines have been widely used in the field test and transportation sites such as airports to identify and discriminate between different explosives. However, canine training is very expensive and dogs easily get tired of continuous sensing. Metal detectors are an indirect technique and very efficient for landmine and weapon detection packaged in metals, however, this technique is not sensitive for explosives chemical finger-print properties and thus cannot be applied for transportation site screening. IMS is a commonly used explosive detection system in airports, and has sensitivity down to nanograms or picograms for common explosives, but this technique lacks enough sensitivity for a broad range of explosives, such as PETN and RDX, which greatly limits its overall utility. Moreover, IMS requires sophisticated protocols with time-consuming calibration, along with its poor portability and high cost, making it not suitable for real-time field detection (Hill, & Simpson, 1997). Although all the above method have advantages, none of these is ideal because of some features such as lack of mobility, expensive, not sensitive enough for a broad range of explosives. Besides, due to the widespread use of explosive formulations, the analysis of explosives has also been critical in landmine detection, forensic research, and in the study of environmental issues associated with explosive residues (Germain, & Knapp, 2009; Salinas et al., 2012). Furthermore, nitroaromatic explosives in short-term or long-term exposure can cause some serious health threats, including carcinogenicity, cataract development, abnormal liver function, and anemia… disease for both animals and humans. Therefore, fast, selective and sensitive explosive detection could provide quick warning in case of terrorist attacks, helping to track and locate explosives to minimize the constant fatalities of civilians from landmines (Salinas et al., 2012; Sun et al., 2015). Recently, optical detection techniques based on the design of colorimetric and fluorimetric assays, which offer many advantages over other common detection techniques, such as low-cost, good mobility, high sensitivity, and selectivity, have attracted great attention (Germain, & Knapp, 2009). Typically, fluorescence-based detection is one to three orders of magnitude more sensitive and has wider linear ranges compared to absorbance-based methods. Furthermore, the source and detector of the fluorescence method could be easily incorporated into a handheld device for the field detection of explosives. Therefore, the fluorescence-based method has the great promise to be applied in detecting explosives quickly, sensitively and selectively.

Fluorescent conjugated polymers (CPs) have recently been used successfully in nitrated explosive detection. Compared with small molecule fluorophores, they have an extended exciton migration pathway and efficient electronic communication between quenchers along the polymer backbone. CPs are excellent electron donors, and their donor ability is enhanced by the delocalized excited state, which facilitates exciton migration and hence increases the electrostatic interaction between the polymer and electron-deficient nitroaromatic analytes. For CPs fluorescent sensors, Swager et al. proposed that binding one receptor site resulted in an efficient quenching of all emitting units in an entire conjugated polymeric molecule relative to single molecule systems. This amplification is known as the molecular wire effect, or the one-point contact, multi-point response effect (Swager, 1998; Rochat et al., 2013). In 2012, Lijuan Feng and co-worker introduced poly(p-phenylenevinylene) (PPV) into mesoporous silica nanoparticles (MSNs) to fabricate the hybrid nanoparticles (PPV@MSNs) by an ion exchange and in situ polymerization route. And they have demonstrated that the PPV@MSN-NH_2 can be used as a nanosensor for TNT sensing. It shows good fluorescence quenching sensitivity towards TNT through FRET because of the good energy matching between TNT–amine complexes and PPV (Feng, 2012).

The general aim of this work is to investigate the effective method to synthesize the new conjugated polymers based on phenoxazine derivatives and 9,9-dioctyl-9*H*-fluorene and to investigate their optical properties. In a further study, this novel conjugated polymer would be developed into a chemical sensor that helps recognize explosives compound.

2. Experiment

2.1. Materials

Perylene (99%), Phenoxazine (99%), $Pd(OAc)_2$ (99%) were purchased by Sigma Aldrich. NBS (99%), $P(t\text{-}Bu)_3$ (98%), NaOt-Bu (97%), MMA (99%) were purchased by Merck. Tetrakis(triphenylphosphine) palladium(0) ($Pd(PPh_3)_4$) (99%), palladium(II) acetate ($Pd(OAc)_2$, 98%), tricyclohexylphosphine tetrafluoroborate ($P(Cy)_3.HBF_4$, 97%), cesium carbonate (Cs_2O_3, 99%), pivalic acid (PivOH, 99%) were purchased from Sigma-Aldrich (St. Louis, MO, USA). Potassium acetate (KOAc, 99%) and potassium carbonate (K_2CO_3, 99%) were purchased from Acros and used as received. Chloroform ($CHCl_3$, 99.5%), toluene (99.5%) and tetrahydrofuran (THF, 99%) were purchased from Fisher/Acros and dried using molecular sieves under N_2. Dichloromethane (DCM, 99.8%), hexane (99%), absolute ethanol (99%), ethyl acetate (EA, 99%) and 1,4-dioxane were purchased from Fisher/Acros and used as received. 2-bromo-3-hexylthiophene, dioxaborolane-TPA and 1-bromopyrene are synthesized by our group in National Key Laboratory For Polymer and Composite Materials (PCKLAB).

2.2. Measurements

^1H-NMR spectra were recorded on a Bruker Avance 300 MHz NMR spectrometers using deuterated chloroform ($CDCl_3$) as a solvent with TMS ($\delta = 0.00$ ppm). The following abbreviations are used to describe the NMR signals: s (singlet), d (doublet), t (triplet), q (quartet), and br (broad). UV-visible absorption spectra of oligomer in solution and thin film were recorded on a Shimadzu UV-2450 spectrometer over the wavelength range of 250-800 nm. Photoluminescence measurements (PL) is obtained by photon excitation (usually a laser) and is commonly observed with III-V semiconductor materials. The emission spectra were recorded in the wavelength range of 350-900 nm. Solutions of all fluorophores were prepared in THF. Size exclusion chromatography (SEC) measurements were performed on a Polymer PL-GPC 50 gel permeation chromatography system equipped with an RI detector, with tetrahydrofuran (THF) as the eluent at a flow rate of 1.0 mL/min. Molecular weight and molecular weight distribution were calculated with polystyrene standards as a reference.

2.3. Synthesis of 10-(Perylene-3-yl)-10H-phenoxazine (PyP)

A 50 ml storage flask was charged with a magnetic stir bar, flamed under vacuum and back-filled with nitrogen three times. The flask was then charged with phenoxazine (183.21 mg), NaOtBu (144.15 mg), $Pd(OAc)_2$ catalyst (4.49 mg), $P(tBu)_3$ (8.09 mg) and dry toluene (45 ml). The flask was evacuated and back-filled three times with nitrogen before 3-bromoperylene (331.21 mg) was added. The flask was then placed in an oil bath at 110^oC while stirring for 24 hours. The flask was then cooled to room temperature and diluted with $CHCl_3$, wash with water, brine, dried with K_2CO_3 and purified using column chromatography (3.3% EtOAc/hexane). The product was dried under reduced pressure to yield 78% of a red orange solid.

^1H NMR (500 MHz, $CDCl_3$), δ (ppm): 8.38 - 8.06 (m, 9H), 7.06 (d, 2H), 6.78 (t, 2H), 6.67 (t, 2H), 5.98 (d, 2H).

2.4. Synthesis of 3,7-dibromo-10-(pyren-1-yl)-10H-phenothiazine

10-(Perylene-3-yl)-10H-phenoxazine (1 equiv.) and *N*-bromosuccinimide (2 equiv.) were added to anhydrous THF (10 mL) at 0 °C under nitrogen. The mixture was stirred 50 °C for 24 h. After that, 10 mL of distilled water was added to the reaction mixture, which was extracted with diethyl ether. The organic layer was washed with 10 % solution o $Na_2S_2O_3$ and 10%solution of KOH (10 %), dried over anhydrous$MgSO_4$ and concentrated. The product was precipitated in cold *n*-heptane and dried under vacuum to give a light yellow powder ($R_f = 0.6$; Yield: 50 %).

^1H NMR (500 MHz, $CDCl_3$): δ (ppm): 8.38 - 8.00 (m, 9H), 7.14 (d, 2H), 6.73 d, 2H), 5.77 (d, 2H).

2.5. Synthesis of Poly(3-(9-heptyl-9-octyl-9H-fluoren-2-yl)-alt-10-(pyren-1-yl)-10H-phenothiazine) (PFPyP)

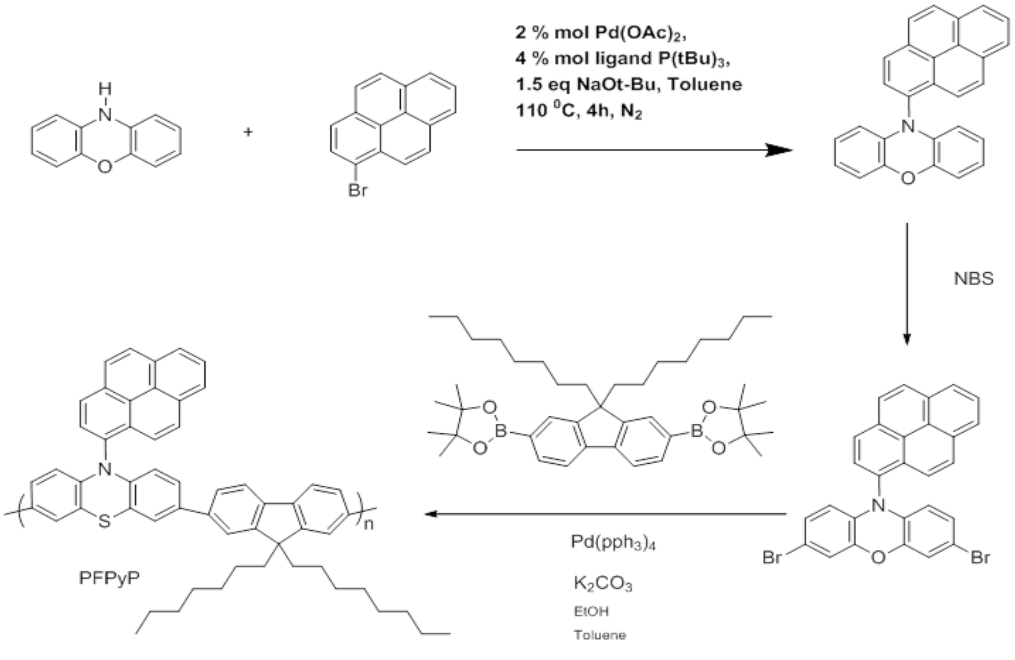
A 50 mL Schlenk flask was charged with compounds 3,7-dibromo-10-(pyren-1-yl)-10H- phenothiazine (139.33 mg, 0.25 mmol), or 3,7-dibromo-10-(pyren-1-yl)-10H-phenoxazine (135.31 mg, 0.25 mmol), and 2,2'-(9,9-dioctyl-9H-fluorene-2,7-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane (M5) (160.64 mg, 0.25 mmol) were mixed in 12.5 mL of toluene at room temperature. The reaction was purged with N_2 for about 20 min. The septum was opened and K_2CO_3 (172.75 mg, 1.25 mmol), 0.6 mL of distilled water, 0.8 mL of EtOH and *tetrakis*(triphenylphosphine)palladium(0) $[Pd(PPh_3)_4]$ catalyst (28.89 mg, 0.025 mmol) were added. Oxygen was removed from the reaction solution by three freeze–pump–thaw cycles, and at the end the reactor was then back-filled with N_2 to preserve an inert atmosphere. Polymerization was initiated by heating the reactor to 100°C and allowed to proceed for 48 h. The reaction mixture was allowed to cool to 55°C, then 50 mL of $CHCl_3$ was added to dissolve any precipitated polymer, and the mixture was eluted with chloroform through a Celite plug. After precipitation into the 200 mL solution of (50:1) mixture of methanol and water, the product was purified by Soxhlet extraction with methanol (24h), acetone (24h), hexane (24h) and finally with chloroform. The chloroform fraction was collected and concentrated and poured into 200 mL of methanol. The precipitated polymer was filtered and dried under vacuum at 50 °C for 24 h to obtain the desired polymer.

Yellow solid (Yield = 157.86 mg, 82%). ^{1}H NMR (500 MHz, $CDCl_3$), δ (ppm): 8.62-7.84 (td, 9H, Py-H), 7.84-7.26 (td, 6H, fluorene -H), 7.22-6.96 (s, 2H), 6.94-6.35 (d, 2H), 5.92-5.55 (d, 2H), 2.15–1.78 (m, 4H), 1.50–0.84 (m, 24H), 0.82–0.48 (m, 6H).

GPC: M_n =25.200 g/mol. Đ= M_w/M_n =2.56.

3. Results and Discussion

The synthetic route of the monomers and conjugated polymers are outlined in Scheme 1. In the first, 10-(pyren-1-yl)-10H-phenothiazine (PyP) has been synthesized via Buchwald-Hartwig C-N coupling amination where Palladium(II) acetate ($Pd(OAc)_2$ and tri-tert-butylphosphine have been used as catalyst and ligand respectively. The resulting monomers were purified via column chromatography with hexane as an eluent to obtain pure monomers. The chemical structures of PyP were presented in Fig. 1.



Scheme 1. *Synthesis of Poly (3-(9-heptyl-9-octyl-9H-fluoren-2-yl)-alt-10-(pyren-1-yl)-10H-phenothiazine) (PFPyP)*

In Figure 1, the ^{1} H NMR exhibited all characteristic peaks of PyP monomer, the peaks from 8.38 - 8.00 ppm assigned for the aromatic proton of pyrene moieties of PyP. The peaks at 7.06 (2H), 6.78 (2H), 6.67 (2H) and 5.98 (2H) are corresponding to phenoloxazine moieties. These results confirmed that the 10-(pyren-1-yl)-10H-phenothiazine (PyP) has been synthesized successfully via the Buchwald-Hartwig C-N coupling reaction.

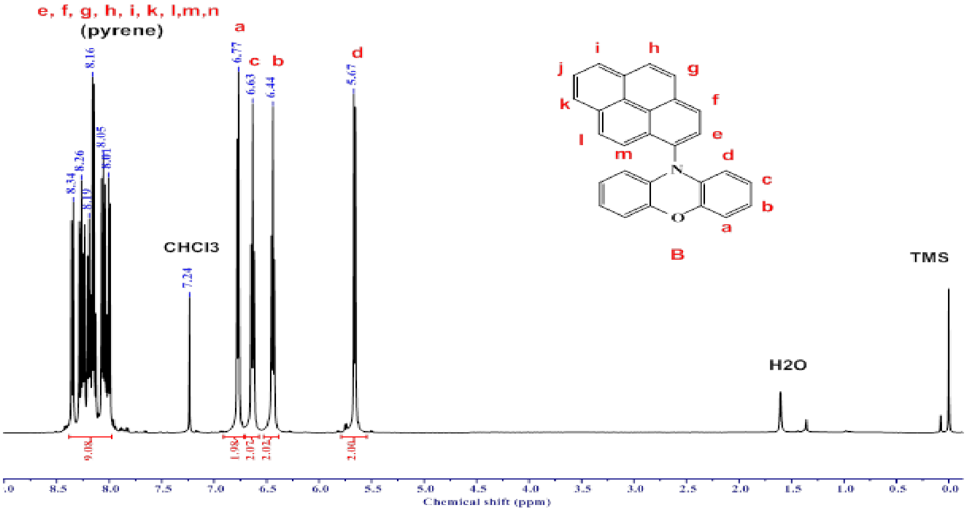


Figure 1. *^{1}H NMR of 10-(pyren-1-yl)-10H-phenothiazine (PyP)*

The 10-(pyren-1-yl)-10H-phenothiazine (PyP) monomer underwent direct arylation polycondensation with 3-(9-heptyl-9-octyl-9H-fluoren-2-yl (DIF) to have the Poly(3-(9-heptyl-9-octyl-9H-fluoren-2-yl)-alt-10-(pyren-1-yl)-10H-phenothiazine) (PFPyP) as Scheme 1. The polycondensation of PFPyP polymer was carried out in DMAc solvent at 100°C using $Pd(OAc)_2$ and $PCy_3.HBF_4$ as the catalyst and ligand, respectively. The reaction solution became green after 3h and gradually turned into deep green accompanying the appearance of a solvent-insoluble part. After 24 h, the conjugated polymer was obtained by purification via extraction, filtration via the Celite layer to remove the Pd catalyst, subsequently precipitated in cold *n*-heptane. The yield of polymerization reactions was in the range of 70-75 %. It should be mentioned that solvent insoluble part (about 5%) and soluble oligomer fraction were removed via filtration through Celite layer and via washing with *n*-hexane and acetone, respectively. The number average molecular weights (M_n) were characterized via GPC relative to polystyrene standards, the PFPyP exhibited the M_n of 25.200 $g.mol^{-1}$ with polydispersities (Đ) of 2.56.

In the ^{1}H NMR of conjugated polymer PFPyP (Fig. 2), a signal was observed at 8.64 ppm and 8.95 ppm which corresponding to the protons of aromatic moieties. The peaks from 0.88 ppm to 1.9 ppm are attributed to the aliphatic protons of alkyl side chains of DIF units. The peaks from 6.99 ppm to 8.10 ppm assigned to the aromatic protons of 10-(pyren-1-yl)-10H-phenothiazine (PyP) moieties. These results indicate that direct arylation coupling polymerization successfully took place to form the alternating conjugated polymers. Table 1 presents the characteristics of the obtained conjugated polymers of PFPyP.

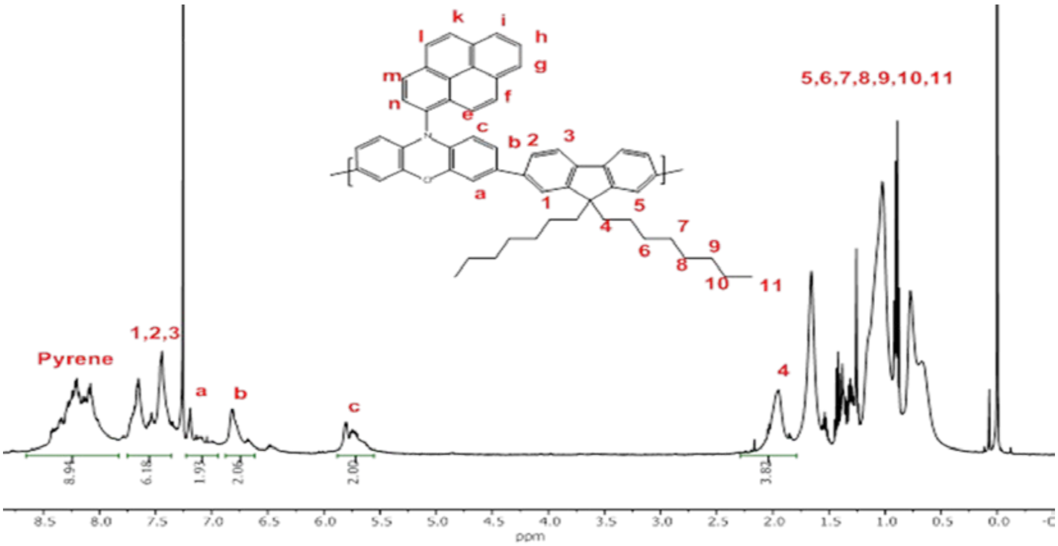


Figure 2. *^{1}H NMR of Poly(3-(9-heptyl-9-octyl-9H-fluoren-2-yl)-alt-10-(pyren-1-yl)-10H-phenothiazine) (PFPyP)*

Figure 3 present the UV–vis spectra of PFPyP measured in the dilute solution of different solvent (~10^{-6} M). PFPyP showed an absorption maximum at 350 nm, and 335 nm in $CHCl_3$, THF, CH_2Cl_2 and toluene solvents. The λ_{onset} of PFPyP is 440 nm, which corresponds to the optical band gaps ($_{Egopt)}$ of 2.85 eV for conjugated polymers.

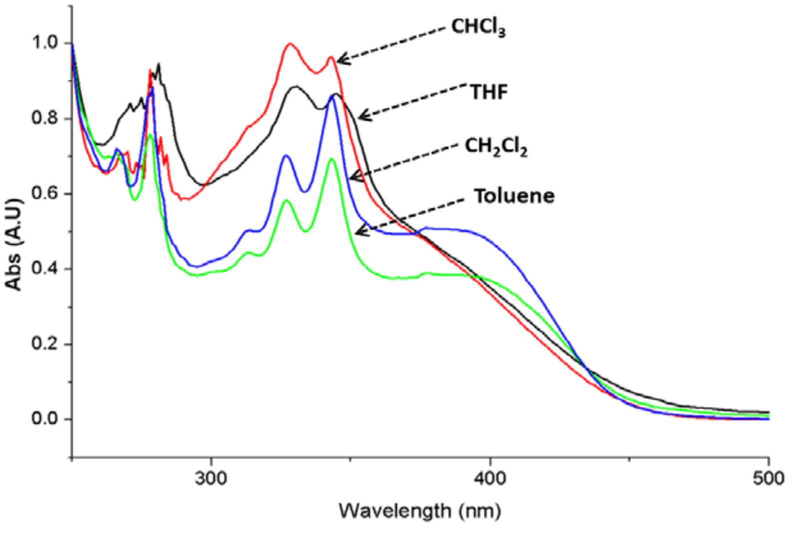


Figure 3. *Absorption spectra of PFPyP in dilute different solvents*

In addition, the photoluminescence (PL) spectra of the conjugated polymers in dilute solution ($CHCl_3$) excited at the absorption maxima are presented in Figure 4. The PFPyP displayed double emission peaks at 240 nm and 310 nm upon excitation at 350 nm in $CHCl_3$. The PyPBPP also exhibited a single emission peak at 310 nm upon excitation at 340 nm for THF and Toluene. Clearly, the fluorescence properties of conjugated polymers PFPyP in $CHCl_3$ is efficient in a diluted solution, promising for the application of conjugated polymers as electrochromic device, organic light-emitting diode, fluorescent sensors…etc devices.

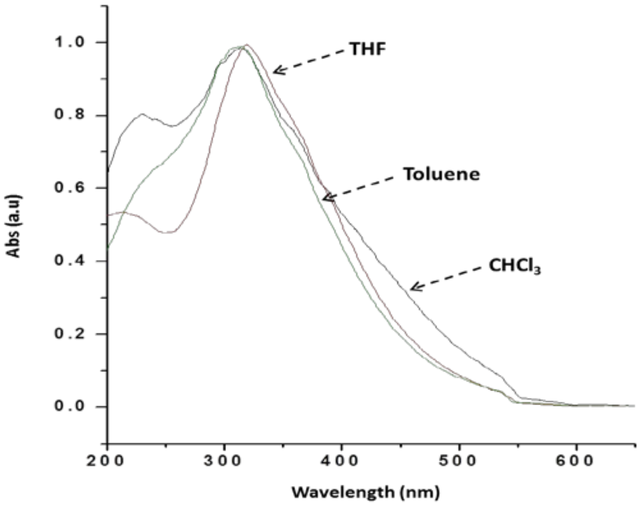


Figure 4. *Fluorescence spectra of PFPyP in different solvents (concentrations of 0.05 g L^{-1})*

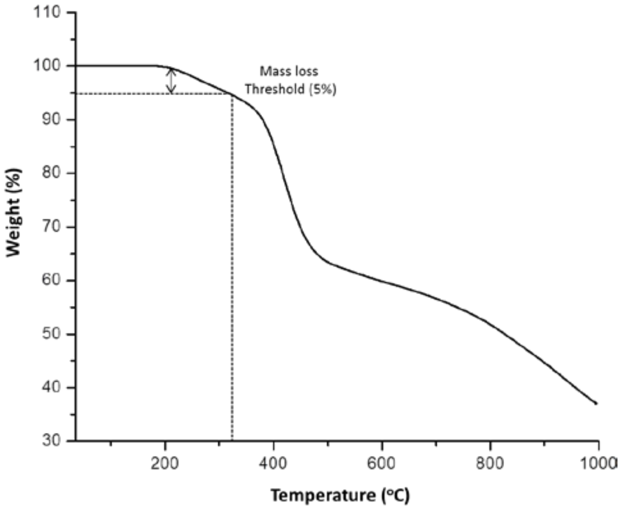


Figure 5. *TGA diagram of PFPyP polymer*

The thermogravimetric analysis (TGA) has been used to investigate the thermal properties of PFPyP. The D-A conjugated polymers were evaluated the thermal stability by TGA which operated under nitrogen flow in the range from room temperature to 1000 °C. The PFPyP polymer exhibited good thermal stability with mass loss threshold decomposition temperature around 310 ^{o}C.

4. Conclusion

In summary, we have synthesized successfully the novel fluorescent conjugated polymer PFPyP based on phenothiazines and 3-(9-heptyl-9-octyl-9*H*-fluoren-2-yl) as donor moieties. The novel conjugated polymer was obtained via direct(hetero)arylation polymerization in 70–74% yields where $Pd(OAc)_2$ and $PCy_3.HBF_4$ have been used as the catalyst/ligand system. The molecular weights of the obtained PFPyP conjugated polymers were 25.200g.mol^{-1}. The polymer exhibited the bandgap of 2.85 eV and the fluorescent properties which promising for electrochromic devices, organic light emitting diode, fluorescent sensors applications.

- ❖ ***Conflict of Interest:*** *Authors have no conflict of interest to declare.*

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TỔNG HỢP POLYMER DẪN PHÁT QUANG TRÊN CƠ SỞ PHENOOXAZINE BIẾN TÍNH VÀ 9,9-DIOCTYL-9H-FLUORENE

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TÓM TẮT

Trong nghiên cứu này, polymer dẫn điện mới Poly(3-(9-heptyl-9-octyl-9H-fluoren-2-yl)-alt-10-(pyren-1-yl)-10H-phenothiazine) (PFPyP) có tính chất phát huỳnh quang trên cơ sở 10-(Perylene-3-yl-10H-Phenoxazine (PyP) và 9,9-dioctyl-9h-fluorene (PF) đã được tổng hợp qua phản ứng polymer hóa ghép đôi trực tiếp sử dung hệ xúc tác Pd(OAc)$_2$, PivOH và PCy$_3$.HBF$_4$. Monomer trên cơ sở phenothiazine được tổng hợp qua phản ứng ghép đôi C-N để tạo ra hợp chất monomer 10-(Perylene-3-yl-10H-Phenoxazine (PyP). Cấu trúc và tính chất của polymer mới được phân tích các phương pháp phân tích phổ cấu trúc và quang học bao gồm ^{1}H NMR, GPC, FTIR, DSC, XRD, PL và UV-Vis. Polymer dẫn thu được thể hiện độ rộng vùng cấm hẹp khoảng 2,85 eV và polymer thể hiện tính phát huỳnh quang tại bước sóng 350 nm. Trên cơ sở các tính chất quang học của polymer PFPyP, polymer có khả năng ứng trọng trong việc chế tạo pin mặt trời hữu cơ và cảm biến hóa học huỳnh quang.

Từ khóa: polymer dẫn điện; quá trình polymer hóa ghép đôi trực tiếp; pin mặt trời nền hữu cơ