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A novel fluorescent poly(pyridine-imide) acid chemosensor

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Abstract

A novel diamine containing heterocyclic pyridine and triphenylamine substituents namely, 4-(4-triphenylamine)-2,6-bis(4-aminophenyl)-pyridine was synthesized via the Chichibabin reaction and reduction. The diamine was used in the preparation of poly(pyridine-imide) by direct polycondensation with 4,4′-hexafluoroisopropylidenediphthalic anhydride in *N*-methyl-2-pyrrolidinone. The poly(pyridine-imide) was converted into a lightly colored, optically transparent, flexible and tough polyimide film which exhibited good thermal stability ($T_d^{10} = 538$ °C) in air and high glass transition temperature ($T_g = 342$ °C); the polyimide film had a tensile strength of 71 MPa and a tensile modulus of 2.2 Gpa. The UV–vis absorption cutoff at 400 nm and fluorescence at ∼600 nm following protonation with acid, enable the film to act as an "off–on" fluorescent switcher for acids.

Keywords: Pyridine; Protonation; Triphenylamine; Chemosensor; Polyimide

1. Introduction

Aromatic polyimides are a very important class of high performance polymers in various applications requiring high thermal, mechanical and electrical characteristics because of their unique mechanical properties, and thermal stability [1–4]. However, the application is frequently limited by their infusible and insoluble nature. To overcome this drawback, a large number of structural modifications have been attempted such as introduction of bulky lateral substituents, flexible alkyl side chains, unsymmetric, alicyclic or kinked structures in the polymer [5–10]. In recent years, the interest has been focused on the synthesis of rigid-rod polyimides by incorporating aromatic rings into the polymer backbone, while the polyimides still possessed highly organosoluble nature [4,5].

Polymers with basic nitrogen atoms offer the possibility of protonation or alkylation of the lone pair as a way of modifying their properties [11]. Recently, some reports have concerned the incorporation of pyridine and its derivatives into polymeric frameworks [12–17]. Compared to benzene ring, pyridine is an electron-efficient aromatic heterocyclic substituent with a localized lone pair electrons in sp^2 orbital on the nitrogen atom; consequently, the derived polymers have increased electron affinity [15] and improved electron-transporting properties [16]. On the other hand, triarylamine-based polymers and dendrimers have been intensively studied because of their potential applications in the field of organic light-emitting diode, information storage devices, hole-transporting materials, field-effect transistors, and photovoltaic cells [18–21]. Recently, there is an intense activity in the design, synthesis and characterization of molecular switches and logic gates. Molecular systems in which fluorescence switches between "on" and "off" states are currently of great interest as these can be modulated, or tuned, by employing external sources such as chemical, ions, molecules and light [22–25]. Controllable fluorescence signals mostly resulting from photoinduced electron transfer activity are at the center of current activity in the field [24,25].

The present investigation deals with the synthesis and characterization of a new poly(pyridine-imide) derived from a new

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monomer, 4-(4-triphenylamine)-2,6-bis(4-aminophenyl)pyridine (TBAP), containing a pyridine heterocyclic group and a triphenylamine pendant group. The solubility, mechanical, thermal, and optical properties of the poly(pyridine-imide) as well as the fluorescence switch phenomena by protonation of this polymer are investigated in this study.

2. Experimental

2.1. Materials

4′-Nitroacetophenone, ammonium acetate, hydrazine monohydrate and 10% palladium on activated carbon were purchased from Merck and used as received. Glacial acetic acid and 4-(diphenylamino)benzaldehyde were purchased from Aldrich Chemical Co. and used as received. 4,4′-Hexafluoroisopropylidenediphthalic anhydride (6FDA, Chriskev, USA) was purified by sublimation and dried at 120 °C under vacuum before use. *N*-methyl-2-pyrrolidinone (NMP), acetic anhydride and pyridine were purchased from Merck and purified by distillation under reduced pressure over calcium hydride and store over 4 Å molecular sieves.

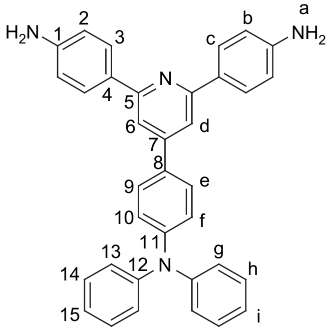
2.2. Synthesis of monomer (Scheme 1)

2.2.1. 4-(4-Triphenylamine)-2,6-bis(4-nitrophenyl) pyridine (TBNP)

In a 500 ml, round-bottomed flask, a mixture of 12.30 g (45 mmol) of 4-(diphenylamino)benzaldehyde, 14.86 g (90 mmol) of 4′-nitroacetophenone, 50 g of ammonium acetate and 250 ml of glacial acetic acid was refluxed for 16 h. Upon cooling, the precipitated light yellow solid was collected by filtration and washed with cold *N*,*N*-dimethylacetamide (DMAc). The crude product (yield 63%) was recrystallized from DMF thrice to afford 5.0 g of fluffy, yellowish, fine needles; m.p. 325 °C (by DSC); FTIR (KBr): 1519 and 1346 cm^{-1} (NO_2), 1541 and 1591 cm^{-1} (pyridine); elemental analysis: calcd. for $C_{35}H_{24}N_4O_4$: C, 74.46; H, 4.28; N, 9.92, found C, 74.13; H, 4.57; N, 9.95.

2.2.2. 4-(4-Triphenylamine)-2,6-bis(4-aminophenyl) pyridine (TBAP)

A mixture of 4.56 g (8.1 mmol) TBNP, 0.25 g 10% Pd/C, 5.9 ml hydrazine monohydrate and 200 ml ethanol was placed in a three-necked flask. The reaction was heated at 85 °C for 24 h. The mixture was then filtered to remove Pd/C. After cooling, the precipitated crystal was isolated by filtration, recrystallized from ethanol twice, ground into powder and dried under vacuum. The yield was 65%; m.p. 237 °C (by DSC); FTIR (KBr): 3351 and 3427 cm^{-1} (NH_2), 1538 and 1592 cm^{-1} (pyridine); 35 ^{1}H NMR (500 MHz, DMSO-d_6, ppm): δ 7.99–8.01 (d, 4H; c), 7.84–7.85 (d, 2H; e), 7.75 (s, 2H; d), 7.31–7.34 (t, 4H; h), 7.05–7.10 (m, 8H; i, g, f), 6.68–6.70 (d, 4H; b), 5.43 (s, 4H; a); ^{13}C NMR (125 MHz, DMSO-d_6, ppm): δ 156.5 (C_5), 149.8 (C_7), 148.0 (C_{11}, C_{12}), 146.9 (C_1), 132.0 (C_8), 129.7 (C_{14}), 128.1 (C_9), 127.7 (C_3), 126.7 (C_4), 124.5 (C_{13}), 123.6 (C_{15}), 122.6 (C_{10}), 113.7 (C_2), 112.1(C_6); elemental analysis: calcd. for $C_{35}H_{28}N_4$: C, 83.30; H, 5.59; N, 11.11, found C, 83.20; H, 5.64; N, 11.16.



2.3. Synthesis of poly(pyridine-imide) (Scheme 2)

To a stirred solution of 0.8167 g (1.62 mmol) of TBAP in 5 ml of NMP, 0.7190 g (1.62 mmol) of 4,4′-hexafluoroisopropylidenediphthalic anhydride was gradually added. The mixture was stirred at ambient temperature overnight (ca. 12 h) to form viscous poly(amic acid). Chemical imidization was carried out by addition of 1 ml of acetic anhydride and 0.5 ml of pyridine into the above-mentioned poly(amic acid) solution with stirring at room temperature for 1 h, and then heating at 110 °C for another 4 h. The polymer solution was poured into methanol. The precipitate was filtered, washed with methanol, and dried at 100 °C under vacuum (1.302 g). The inherent viscosity of the poly(pyridine-imide) in *N*,*N*-dimethylacetamide was 0.53 dl/g, measured at a concentration 0.5 g/dl at 30 °C; FTIR (film): 1783, 1726, 1371 (imide), 1425, 1540, 1591 (pyridine) cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6): δ 8.44–8.46 (d, 4H), 8.12–8.15 (m, 4H), 8.03–8.04 (d, 2H), 7.94 (s, 2H), 7.83–7.85 (d, 2H), 7.66–7.67 (d, 4H), 7.27–7.30 (t, 4H), 7.18–7.20 (d, 2H), 7.13–7.15 (d, 4H), 7.03–7.06 (t, 2H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 166.7, 157.4, 150.8, 150.1, 148.6, 140.0, 139.5, 136.8, 134.4, 134.0, 133.9, 133.0, 130.4, 129.1, 128.4, 127.4, 126.1, 125.8, 125.5, 124.8, 124.5, 124.2, 123.8, 117.3; elemental analysis: calcd. for 25 $C_{54}H_{30}F_6N_4O_4$: C, 71.19; H, 3.58; N, 6.04, found C, 70.00; H, 3.62; N, 5.63.

2.4. Measurements

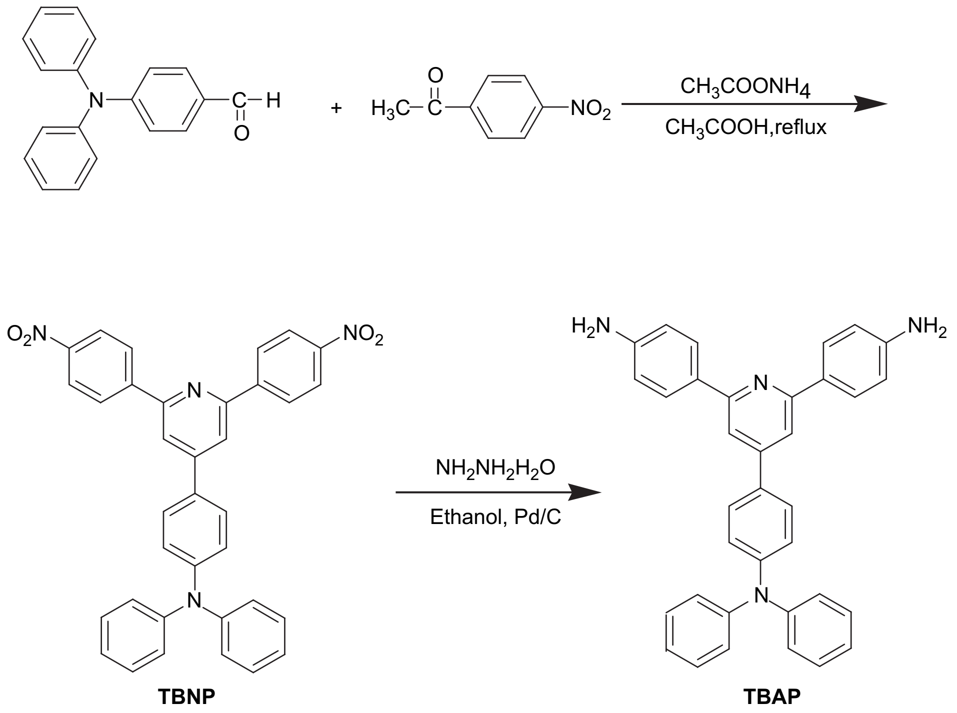
IR spectra were recorded in the range 400–4000 cm^{-1} for the synthesized monomers and polymers in a KBr disk or polymer film (Bio-Rad Digilab FTS-3500). Elemental analysis was made on a Perkin–Elmer 2400 instrument. The inherent viscosity of polyimide was measured using Ubbelohde viscometer. NMR spectra were recorded using a Bruker Avance 500 NMR. Weight-average (M_w) and number-average (M_n) molecular weights were determined by gel permeation chromatography (GPC). Four Waters (Ultrastyragel) columns (300 × 7.7 mm, guard, 10^5, 10^4, 10^3, and 500 Å in a series) were used for GPC analysis with tetrahydrofuran (THF; 1 ml/min) as the eluent. The eluent was monitored with a UV detector (JMST Systems, VUV-24, USA) at 254 nm.

Polystyrene was used as the standard. Thermogravimetric data were obtained on a TA instrument Dynamic TGA 2950 under nitrogen flowing condition at a rate of 50 cm^3/min and a heating rate of 10 °C/min. Differential scanning calorimetric analysis was performed on a differential scanning calorimeter (TA instrument TA 910) under nitrogen flowing condition at a rate of 50 cm^3/min and a heating rate of 10 °C/min. Tensile properties were determined from stress–strain curves obtained from an Orientec Tensilon with a load cell of 10 kg. A gauge of 2 cm and a strain rate of 2 cm/min were used for this study. UV–vis spectra of the polymer films were recorded on a V-550 spectrophotometer at room temperature in air. The fluorescence spectra were recorded by a Shimadzu RF-5031 spectrophotometer. Morphology observations were performed with a Hitachi S-2400 scanning electron microscope. The specimens for SEM observations were prepared by depositing a drop of the solutions onto a glass slide.

3. Results and discussion

3.1. Monomer synthesis

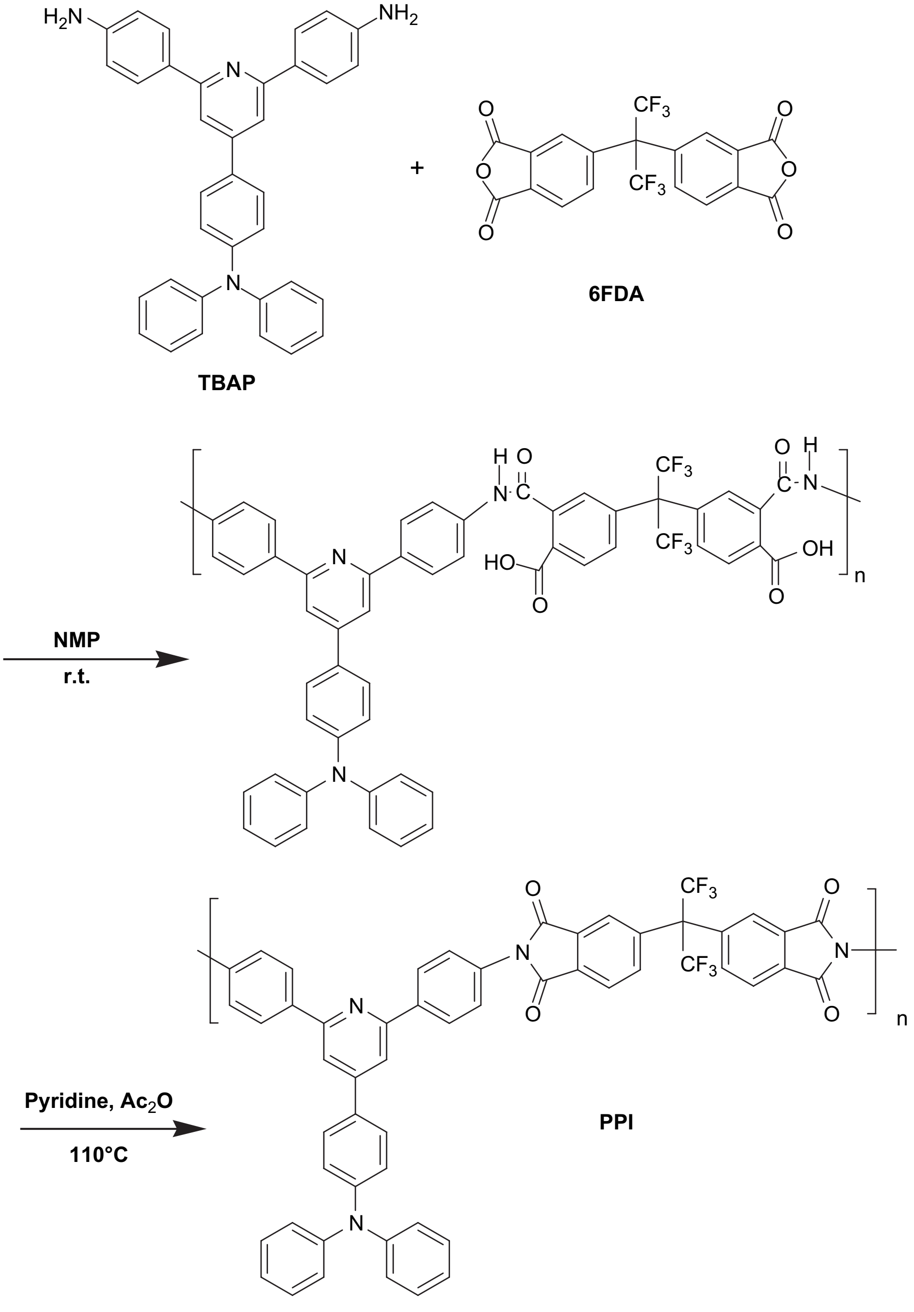
Scheme 1 shows the synthesis of the novel diamine containing a heterocyclic pyridine substitute and a triphenyl pendant group, 4-(4-triphenylamine)-2,6-bis(4-aminophenyl)pyridine (TBAP). The dinitro compound, 4-(4-triphenylamine)-2,6-bis(4-nitrophenyl)pyridine (TBNP), was synthesized with a modified Chichibabin reaction, which is a facile way for the preparation of substituted pyridine [26,27]. The major disadvantage of Chichibabin reaction is that the reaction usually yields tarry crude by-product that requires special efforts to isolate and purify the desired pyridine. Herein, Chichibabin reaction of 4-(diphenylamino)benzaldehyde and 4′-nitroacetophenone in the presence of ammonium acetate was carried out and afforded dinitro compound (TBNP) in one step. Unfortunately, the solubility of this nitro compound is so poor (the solubility in DMSO is less than 0.3 wt% at room temperature) that ^{1}H NMR could hardly be detected. Although the dinitro compound was recrystallized from DMF thrice, impurity still existed in the product from TLC testing. FTIR spectrum of TBNP shows characteristic bands of nitro groups at 1346 and 1519 cm^{-1}. In addition, characteristic C=C and C=N bands of pyridine ring at 1430, 1541, and 1591 cm^{-1} were also observed in FTIR spectrum. However, the elemental result shows that the impurity is very few. The reduction of the dinitro compound (TBNP) in ethanol with hydrazine monohydrate in the presence of catalytic amount of palladium on activated carbon at 85 °C produced a new diamine compound (TBAP). The structure of this diamine was confirmed by elemental analysis, IR, and NMR spectroscopies. The characteristic absorptions at 1519 and 1346 cm^{-1} for the nitro groups were absent, and new absorptions at 3351 and 3427 (N–H stretching) appeared. In ^{1}H NMR spectrum, diamine (TBAP) has a new singlet peak (5.43 ppm) due to the amino proton. There are 15 resonance signals in the ^{13}C NMR spectrum for the diamine and three ^{13}C NMR signals (156.5, 149.8, and 112.1 ppm) confirmed the formation of pyridine ring. In ^{1}H NMR spectra, there are eight protons overlapped at the range of 7.05–7.10 ppm. The full assignment of NMR peaks and the structure of the diamine were also confirmed by two-dimensional H–H COSY and HMQC spectra. FTIR, NMR, and elementary analysis results have clearly confirmed that the diamine (TBAP) prepared herein is consistent with the proposed structure.



Scheme 1. Synthesis of new diamine, TBAP, 4-(4-triphenylamine)-2,6-bis(4-amionophenyl) pyridine.

3.2. Preparation of poly(pyridine-imide) (PPI)

Poly(pyridine-imide) was prepared by the conventional two-step polymerization method, as shown in Scheme 2, involving ring-opening polyaddition to form poly(amic acid) and subsequent chemical imidization. Polyaddition of dianhydride and diamine at ambient temperature gave a viscous poly(amic acid) solution. The chemical imidization of poly(amic acid) by dehydrating agents such as a mixture of acetic anhydride and pyridine was effective in obtaining polyimides. Poly(pyridine-imide) obtained by chemical imidization had inherent viscosity of 0.53 dl/g in DMAc. The poly(pyridine-imide) exhibits number-average molecular weights (M_n) and weight-average molecular weights (M_w) of 3.0×10^4 and 8.8×10^4, respectively, as measured by GPC, relative to polystyrene standards. The molecular weight of poly(pyridine-imide) was sufficient to cast into a tough and flexible film. The polymer structure was confirmed by elemental, IR, and NMR analyses. The elemental analysis value of the poly(pyridine-imide) is in agreement with the value of the proposed structure. FTIR spectrum exhibits that the characteristic absorption bands are around 1783 and 1726 cm^{-1} due to the asymmetric and symmetric stretches of carbonyl group of imide. The C–N bond exhibits a band around 1371 cm^{-1} due to the formation of the imidization. The ^{1}H NMR spectrum also confirmed the chemical structure of the poly(pyridine-imide).



Scheme 2. Preparation of novel poly(pyridine-imide).

3.3. Polymer properties

The poly(pyridine-imide) exhibited excellent solubility in various solvents such as tetrahydrofuran, *N*-methyl-2-pyrrolidinone, *N*,*N*-dimethylacetamide, *N*,*N*-dimethylformamide, and dimethyl sulfoxide at room temperature. The excellent solubility of this polyimide results from both the bulky pendant triphenylamine group and 6FDA structures. The bulky pendant triphenylamine group in the pyridine unit for poly(pyridine-imide) derived from diamine (TBAP) reduces packing force and increases the free volume of the poly(pyridine-imide), thus resulting in good polymer solubility.

The thermal properties of the poly(pyridine-imide) were evaluated by differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). The poly(pyridine-imide) shows glass transition temperature at 342 °C. The thermal stability of the poly(pyridine-imide) was evaluated by TGA measurements in both air and nitrogen atmospheres at a heating rate of 10 °C/min. The temperature for 10% weight loss of poly(pyridine-imide) in nitrogen and air is 538 °C and 526 °C, respectively. The possible reason for the good thermal stability of poly(pyridine-imide) is attributed to the presence of rigid pyridine heterocyclic moiety in the polymer backbone [19] and triphenylamine group in the pedant.

The poly(pyridine-imide) could be cast into a film from DMAc solution, and the film was light-colored, optically transparent, flexible, and tough. The film exhibits ultimate tensile strength of 71 MPa, elongation to break of 11%, and tensile modulus of 2.2 GPa. The polymer film exhibits high mechanical properties; thus it can be considered as a strong material.

Fig. 1 illustrates the absorption spectra of poly(pyridine-imide) in THF as a function of HCl (a) and MSA (methanesulfonic acid) (b) concentrations with a polymer concentration of 1 mg/10 ml. The poly(pyridine-imide) exhibits absorption cutoff at 400 nm. The absorption maximum (λ_{max}) at 273 nm is due to the n–π* transition of pyridine group, and absorption maximum at 351 nm is due to π–π* transition. From protonation with HCl (Fig. 1a), the intensity of the two absorptions decreases and a new absorption band forms at 440 nm when HCl concentration is higher than 0.01 M. The absorption intensities at 273 nm (n–π*) and 351 nm (π–π*) decrease after protonation, because the lone pair electron of the nitrogen on pyridine is quaternated by protic acid. The intensity of new band around 440 nm depends on the HCl concentration. The polymer protonated by higher HCl concentration exhibits stronger absorption at 440 nm. The spectral behavior suggests that a lower π–π* transition occurs for the quaternated polymer. The absorption peaks at 273 and 351 nm decreased but do not disappeared at high acid concentrations. This indicates the coexistence of protonated and neutral segments in the poly(pyridine-imide). Ng et al. showed the same protonation phenomenon in a fluorescent polymer with pyridine repeat units [28]. The same phenomenon was also observed in the protonation with MSA (Fig. 1b). However, from a comparison with Fig. 1a, the absorption band of protonated polymer with MSA exhibits a slight red shift (from 440 to 450 nm), and its absorption intensity is a little bit stronger than that obtained with HCl for the same acid concentration. These observations reveal that the absorption characteristic of the quaternated form of the polymer is influenced by the conjugated base of the used acid. Fig. 2 shows the emission spectra of protonated poly(pyridine-imide) with HCl (a) and MSA (b). Calculated amount of HCl solution was added into the THF solution of the polyimide sample, and the solution was kept overnight before the fluorescence measurement. There was no fluorescence observed in neutral polymer solution excited at 440 nm. However, the strong fluorescence around 600 nm was observed after protonation with different HCl concentrations. Two interesting emission features caused by different acid concentrations are as follows. The first is that the emission intensity of protonated polymer exhibits an acid concentration dependent increase in the concentration range of 0.001–0.1 M and decreases at the acid concentrations beyond 0.1 M. The second is the spectral shift in the emission maxima to longer wavelength as the acid concentration increases from 0.001 to 0.1 M. The protonation of this poly(pyridine-imide) (PPI) is

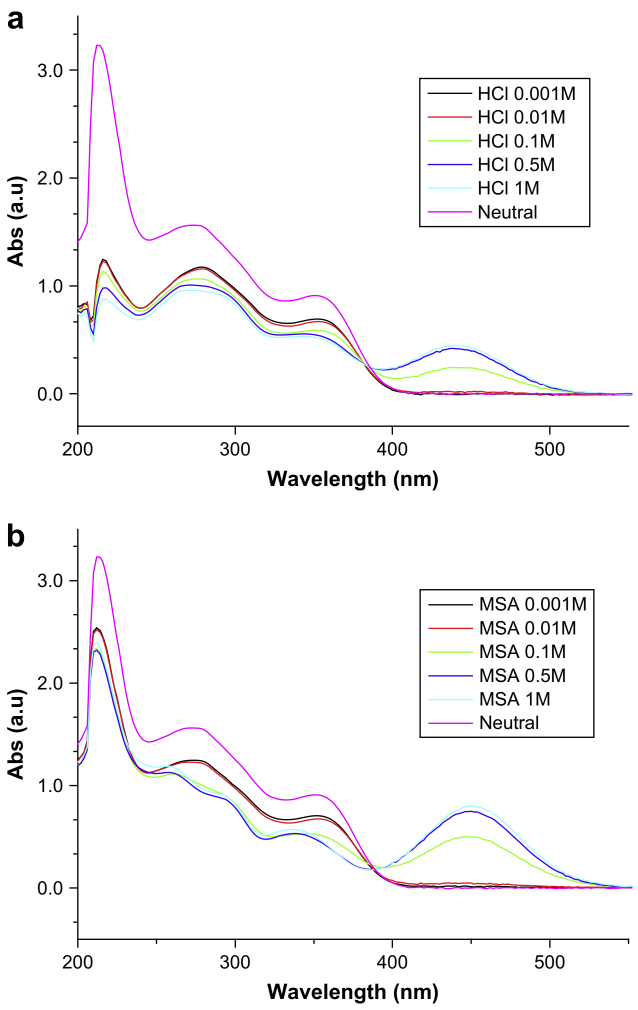


Fig. 1. UV–vis spectra of poly(pyridine-imide) in THF solution as a function of (a) HCl and (b) MSA concentrations with a polymer concentration of 1 mg/10 ml.

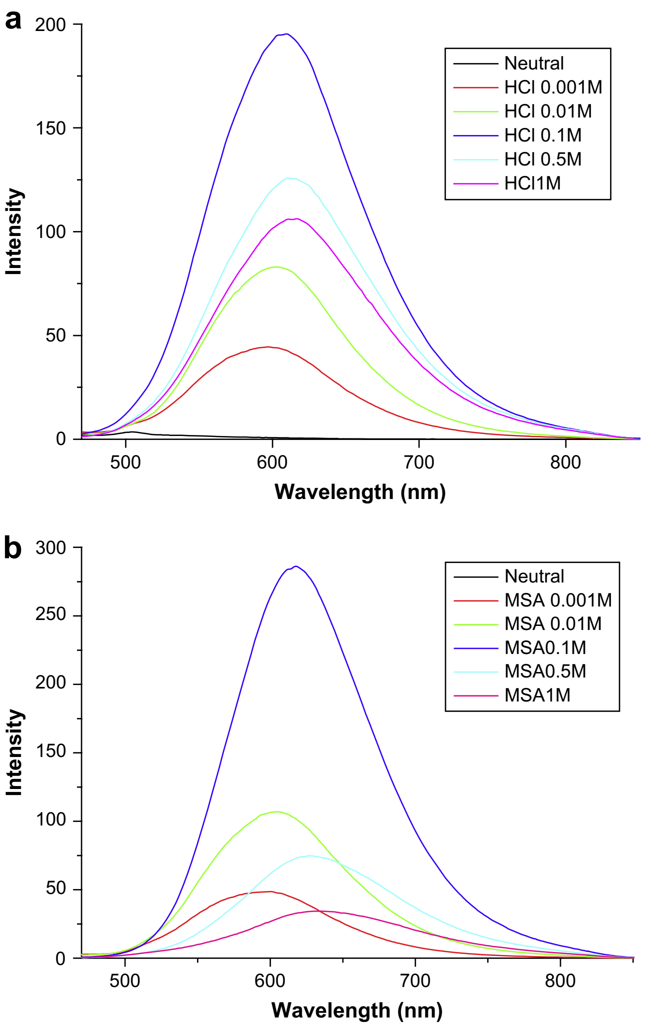
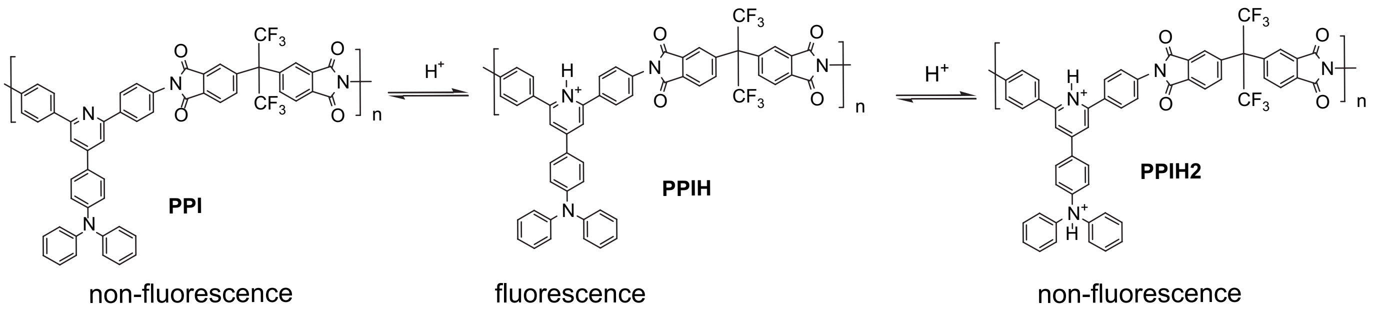


Fig. 2. Emission spectra of poly(pyridine-imide) in THF solution as a function of (a) HCl (excited at 440 nm) and (b) MSA (excited at 450 nm) concentrations with a polymer concentration of 1 mg/10 ml.

shown in Scheme 3. Because the pyridine group is more basic than triphenylamine group, the lone pair electron of the nitrogen on pyridine was quaternated by protic acid at first to form quaternated polymer (PPIH) and then the lone pair electron of the nitrogen on triphenylamine was further quaternated by protic acid to form PPIH2 (Scheme 3), as acid concentration increases. The triphenylamine is a well-known electron donor and the pyridine is a weak electron acceptor, therefore the neutral poly(pyridine-imide) (PPI) is nonfluorescence as it gets excited at 440 nm. However, the quaternated pyridine becomes a stronger acceptor as protic acid gets attached to the pyridine, and then a strong emission occurs between triphenylamine and pyridine group (PPIH). As described in the UV–vis spectral discussion, this protonation is an equilibrium transformation. Furthermore, the quaternated triphenylamine changes into a weak donor as protic acid attached to the nitrogen on triphenylamine (PPIH2). The emission does not disappear even at very high acid concentration (1 M). It means this protonation is also an equilibrium transformation. This statement coincides with the first feature of emission spectra which show a maximum intensity protonated by 0.1 M HCl. The two features were also observed when protonation was carried out by MSA (Fig. 2b). The features in MSA case are more obvious than in HCl case due to stronger acidity of MSA. In general, PL of polymers in the solid state is dominated by excited dimer emission, which is red shifted compared to that of a single chain exciton by an amount depending on the interchain coupling [29]. We suppose that the second feature regarding red shift is in relation to aggregation of the protonated polymer. The emission around 600 nm could be changed from ‘‘off’’ state to ‘‘on’’ state due to protonation. The principle of fluorescence switch between ‘‘on’’ and ‘‘off’’ states in this study is based on intramolecular charge transfer due to protonation rather than photoinduced electron transfer which was developed by de Silva et al. [23,30].

The morphology of the MSA protonated poly(pyridine-imide) was observed at room temperature by scanning electron micrographs and is shown in Fig. 3. Thin films were obtained by depositing a drop of the polymer solutions (1 mg/10 ml in THF) onto a glass slide. Morphology of the film surface is generated by the aggregation of polymer chains during evaporating the solvent. The SEM micrograph of the protonated poly(pyridine-imide) with different MSA concentrations from THF solutions reveal that the surface of polymer is homogeneous with irregular shapes. It is apparent that MSA protonated sample with higher MSA concentration exhibits larger size of aggregates than the sample protonated with lower concentration. This result is consistent with the results of emission spectra (Fig. 2), wherein red shift exists at higher acid concentration. Although the SEM micrograph is not a direct evidence for the aggregation behavior of polymer chains in the dilute solution, it still can be considered



Scheme 3. Protonation of the poly(pyridine-imide).

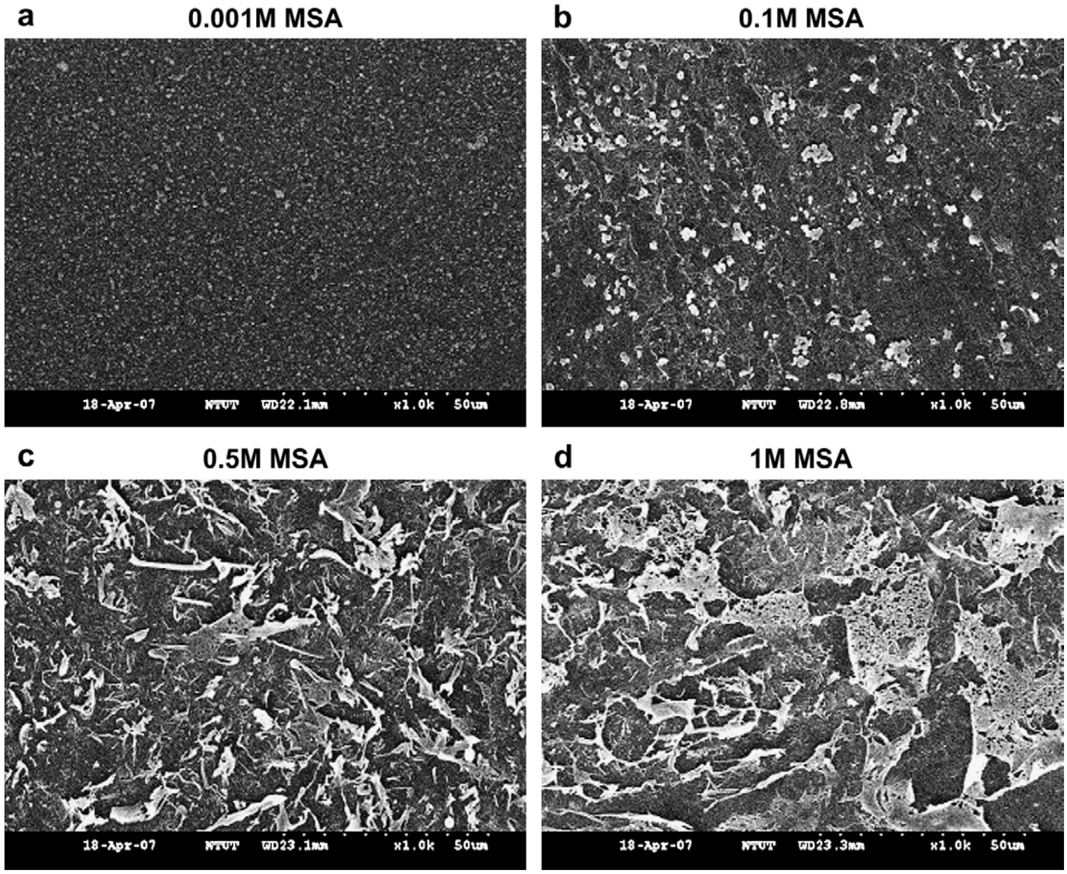


Fig. 3. SEM of protonated poly(pyridine-imide) depositing a drop of the polymer solutions using THF as solvent onto a glass slide: (a) protonated with 0.001 M MSA, (b) protonated with 0.1 M MSA, (c) protonated with 0.5 M MSA, and (d) protonated with 1 M MSA.

as an indirect evidence that influences the aggregation behavior of polymer chains in the solution.

4. Conclusion

In the present study, a novel poly(pyridine-imide) containing pyridine and triphenylamine groups derived from the synthesized new diamine, 4-(4-triphenylamione)-2,6-bis(4-aminophenyl) pyridine (TBAP), and a commercial dianhydride was successfully prepared. This poly(pyridine-imide) exhibits excellent solubility in organic solvents. The polymer shows good thermo-oxidative stability higher than 520 °C and excellent mechanical properties. After protonation with protic acid, a strong emission around 600 nm was observed. The polymer showed significant changes in the emission properties as a function of acids. The emission of the protonated ploy(pyridine-imide) presents bathochromic shift, and the intensity of emission depends on concentration and species of acids. The protonation phenomena based on intramolecular charge transfer indicates that the polymer is a promising material for the applications on molecular switches and gates.

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