

Acidochromicity of a Low-Molecular-Weight Pyrimidine-Based Copolymer

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ABSTRACT: A new type of low-molecular-weight polypyrimidine in a π -conjugated main chain was prepared by a Grignard reaction between 2-amino-4,6-dichloropyrimidine and 1,4-dibromo-2,5-didodecyloxybenzene in the presence of [1,2-Bis(diphenylphosphino)ethane]dichloronickel(II). The structure of the copolymer was fully elucidated by Fourier transform infrared spectroscopy, $^1\text{H-NMR}$, and elemental analysis. The copolymer had good solubility in common organic solvents. The copolymer displayed a bathochromic shift when protonated with an organic or inorganic acid in chloroform or tetrahydrofuran. The copolymer depicted facile p -doping and good electron-transporting electrochemical properties in a 1M H_2SO_4 aqueous solution. The copolymer showed a narrow polydispersity of 1.04. Thermogravimetric analysis showed that the copolymer had a certain thermal stability with no decomposition at a temperature of 250°C under N_2 . © 2014 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2014**, *131*, 41174.

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INTRODUCTION

During the past decade, conjugated polymers are an interesting class of organic materials for different (nonlinear) optical and electrical applications.^{1–5} The main classes of conjugated polymers are polyarylenes, poly(arylene vinylene)s, and poly(arylene ethynylene)s.^{3–6} In addition to polymers, oligomers have received attention as model systems, optical brighteners, and electronic or scintillating materials.^{3–9} The prototype of the arylene subunit is a benzene ring, but a large variety of fluorophores contain aromatic heterocycles, such as furans or oxadiazoles.^{2–7} Benzene can be replaced by heteros such as furane, oxadiazoles, or pyridine.^{5–9} Oligo(phenylenevinylene)s with terminal pyridines has been investigated.² However, π -conjugated polymers having pyrimidine rings have received less attention, despite the presence of a certain number of reports about their analogs, such as polymers containing pyridine rings.^{3,9} Because of their electron-deficient nature, pyrimidines could theoretically establish a charge-transfer (CT) structure with phenyl units, which have a π -excessive nature.^{1–9} During our work on conjugated systems, we became interested in oligomers containing electron-deficient heterocycles such as 2-amino-4,6-dichloropyrimidine. Although several articles from our previous study have been published on the preparation and properties of π -conjugated polymers [poly(2,5-dido-

decyloxy-1,4-diethynyl phenylene-*alt*-2-amino-4,6-pyrimidine) (PI)] containing pyrimidine rings (Scheme 1) with the CT structure^{7–9} which showed interesting acidochromism in solution with a high acid sensitivity, more detailed studies seem to be required to reveal their basic properties. These acidochromic polymers are also attractive because of their applications in the sensor industry. In a suitably designed donor–acceptor polymer, modulation of the donor or acceptor properties by the interaction of analytes leads to signal amplification.

In this study, we chose 2-*N,N*-dimethylamino-4,6-dichloropyrimidine to act on the electron density of the conjugated chain by means of an electroactive functional group and an electron acceptor attached on a phenyl-moiety-synthesized copolymer [poly(2,5-didodecyloxy-1,4-phenylene-*alt*-2-*N,N*-dimethylamino-4,6-pyrimidine)] (PII, Scheme 1) to study the relationship between the structure and optical properties of a pyrimidine CT-type polymer, especially to further investigate their acidochromism.

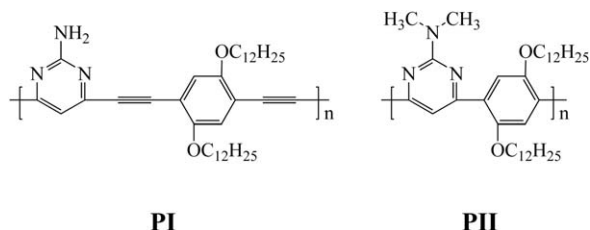
EXPERIMENTAL

Materials

All of the chemicals used were analytical grade. 2-Amino-4,6-dichloropyrimidine, [1,2-Bis(diphenylphosphino)ethane]dichloronickel(II), bromine, 1-bromododecane, tetrahydrofuran (THF),

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Scheme 1. Structures of the discussed copolymers.

hydroquinone, dimethylformamide (DMF), and magnesium were purchased from Acros Chemical Co. (China) and were used as received. 2-*N,N*-Dimethylamino-4,6-dichloropyrimidine,⁹ 1,4-didodecyloxybenzene,¹⁰ and 1,4-dibromo-2,5-didodecyloxybenzene¹¹ were synthesized according to the literature. Other reagents, such as methanol and chloroform, were commercially available and were used as received unless otherwise stated.

Characterization

¹H-NMR spectra were taken with a Varian Inova-400 spectrometer (400 MHz). Photoluminescence and excitation spectra were recorded on a Hitachi F-4500 spectrophotometer. Elemental analyses were made on a Flash EA series 1112 NCHS-O analyzer. Fourier transform infrared (FTIR) spectra of the copolymer were obtained with a Bruker Equinox-55 FTIR spectrometer (frequency range = 4000–500 cm⁻¹). Ultraviolet-visible (UV-vis) spectra of the copolymer were recorded with a Shimadzu UV-2450 spectrophotometer. The X-ray diffraction (XRD) studies were performed on a D/Max 2400 X-ray diffractometer with a Cu K α radiation source ($\lambda = 0.15418$ nm, λ is X-ray wavelengths). The scan range (2θ) was 5–60°. Molecular weight determinations were made with a Waters Alliance GPC V 2000 at a flow rate of 0.94 mL/min in DMF Waters Styragel columns (HT 3, HT 4, and HT 6) at a temperature of 65°C. The thermal stability of the dry sample was determined with a Netzsch STA 449C instrument with a temperature range of 26–900°C at a heating rate of 10°C/min with a nitrogen purge at a flow rate of 30 mL/min.

Synthesis of PII

A suspension of magnesium powder (0.091 g, 3.8 mmol) in dry THF (30 mL) was activated with a drop of 1,2-dibromoethane and heated to reflux. A solution of 1,4-dibromo-2,5-didodecyloxy (1 g, 1.65 mmol) in 10 mL of anhydrous THF was added dropwise and refluxed for 4 h. The THF solution (5 mL) of 2-

N,N-dimethylamino-4,6-dichloropyrimidine (0.317 g, 1.65 mmol) and [1,2-Bis(diphenylphosphino)ethane]dichloronickel(II) (0.08 mmol) were added dropwise to the Grignard reagent. The mixture was heated under reflux for 24 h, and the reaction was quenched with a saturated solution of NH₄Cl. The aqueous phase was extracted with chloroform. The solution was then dried over anhydrous Na₂SO₄, and the solvent was evaporated at reduced pressure. The residue was dissolved in chloroform and recrystallized twice from ethanol/CHCl₃. After it was dried overnight at 40°C, it was isolated in a 75% yield. Gel permeation chromatography analysis (in DMF) gave a number-average molecular weight of 3910 and a weight-average molecular weight of 4073.

¹H-NMR (CDCl₃): 7.089–7.113 (broad, 2H, Ar-H), 6.791–6.838 (broad, 1H, Ar-H), 3.866–3.965 (broad, 4H, -OCH₂-), 3.241–3.398 [broad, 6H, -N-(CH₃)₂], 1.20–1.85 (m, 40H), 0.86(t, 6H). ANAL. Calcd for (C₃₆H₅₉N₃O₂): C, 76.41; H, 10.51; N, 7.53. Found: C, 76.66; H, 10.32; N, 7.91. UV-vis (CHCl₃) λ_{\max} : 397 nm (λ is maximum absorption peak wavelength).

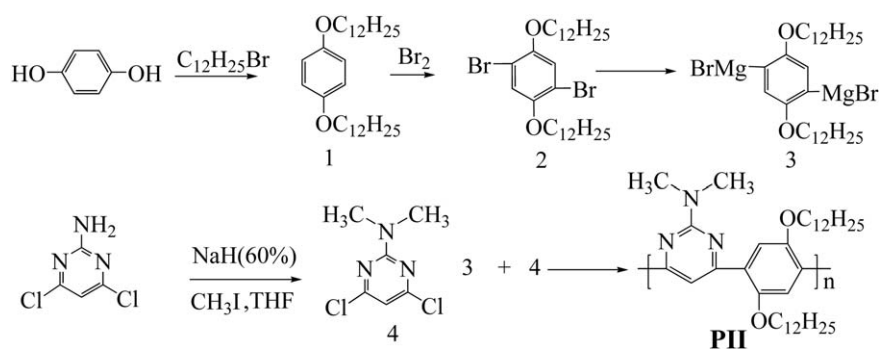
RESULTS AND DISCUSSION

The synthetic procedure of the monomers and copolymers is described in Scheme 2. The precursor of 2-*N,N*-dimethylamino-4,6-dichloropyrimidine was prepared via a route from the reported route,⁹ and the low-molecular-weight copolymer was obtained by a Grignard reaction with a good yield. The chemical structure of the copolymer was characterized by ¹H-NMR (Figure S1, Supporting Information), ¹³C-NMR (Figure S2, Supporting Information), and FTIR (Figure S3, Supporting Information) spectroscopy and elemental analyses, and these indicated that the copolymer structure contained a pyrimidine ring and benzene.

Optical Properties

The chloroform solution of the copolymer was light yellow and fluoresced blue under irradiation with UV light (365 nm).

Figures 1 and 2 show the UV-vis and photoluminescent (PL) spectra, respectively, of the copolymer. The copolymer showed λ_{\max} (maximum absorption peak wavelength) at a longer wavelength (397 nm, log $\epsilon = 1.61$; the molarity was based on the repeating unit) than the corresponding monomers (5 $\lambda_{\max} = 316$ nm and 2 $\lambda_{\max} = 302$ nm); this indicated that the copolymer had an expanded π -conjugated system. The film of the copolymer showed the UV-vis peak at a somewhat longer



Scheme 2. Procedure for the preparation of PII.

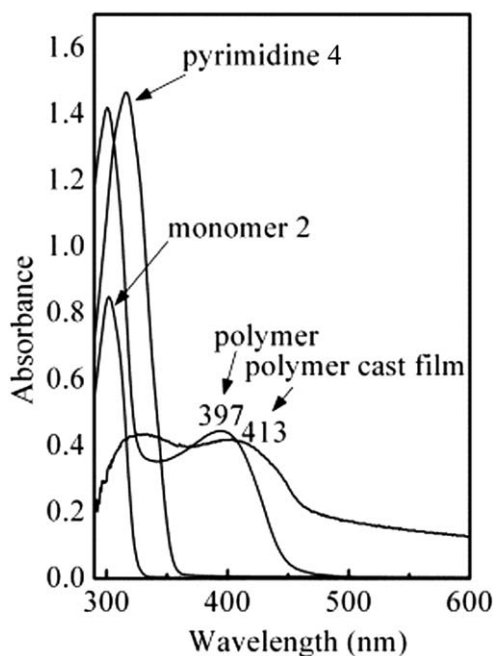


Figure 1. UV-vis spectra of the monomers and PII (2.12×10^{-3} mol/L CHCl_3).

wavelength (413 nm); this suggested the presence of certain intermolecular electronic interactions. However, the degree of the redshift was considerably smaller than those observed with copolymers that took face-to-face stacking.^{11–13} This suggested that the UV-vis spectra of the film may have resulted from the longer distance between the interlayers of the copolymer (Figure

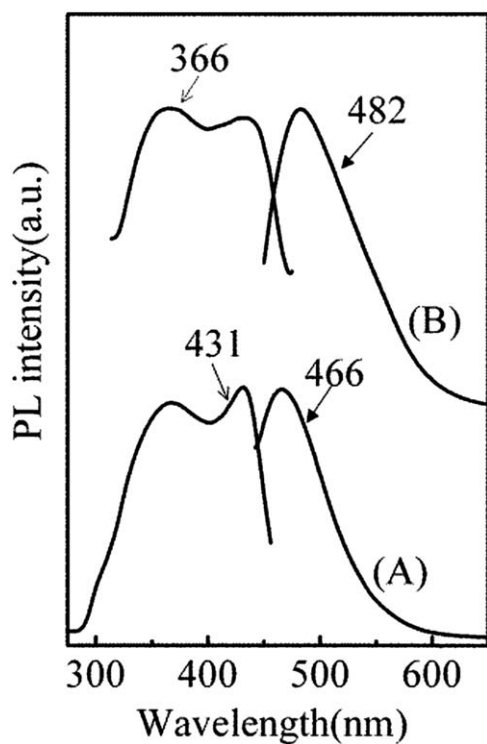


Figure 2. Excitation spectra (light arrows) and emission spectra (dark arrows) of PII (A) in CHCl_3 (2.12×10^{-3} mol/L) and (B) in the solid state.

1), which decreased the degree of face-to-face stacking. The PL spectrum of the chloroform solution showed a peak at 466 nm. The PL peak appeared near the onset position of the UV-vis absorption band of the copolymer. The photoluminescence peak in the solid state was observed at 482 nm and shifted by 16 nm to a longer wavelength than that of the chloroform solution, and the PL peak appeared near the onset position of the UV-vis absorption band of the copolymer.

The excitation spectrum in Figure 2 is not similar to the absorption spectra in Figure 1. This may have been caused by the low molecular weight of copolymer; this was also the proof of the copolymer, which was a mixture comprised of small molecules. The copolymer was soluble in common organic solvents, such as chloroform, THF, toluene, DMF, and dimethyl sulfoxide; partially soluble in hexane and acetone; and insoluble in alcohol and methanol.

Previously, we reported the acidochromism of PI, and we have a great interest in the influence of the acidochromism caused by differences in the molecular structure. Consequently, we investigated the effect of protonation on the PII through the measurement of the UV-vis spectra of the copolymer in CHCl_3 -organic acid mixtures or in THF-inorganic acid mixtures.

Figure 3(1–6) depicts the changes in the absorption spectra of the copolymer with increasing concentrations of acids. As shown in Figure 3, the addition of acids to a CHCl_3 /THF solution of copolymer (2.12×10^{-3} mol/L) led to a quick decrease in the intensity of the absorption at 397 nm and, at the same time, an appearance of a new band at the interval of 420–433 nm. These results suggest that the absorption at a longer wavelength in CHCl_3 or THF solution originated from a π - π^* transition along the main copolymer chain. Moreover, this π - π^* transition was strongly affected by the protonation of nitrogen in the copolymer, which caused a distortion of the interring bonds ascribable to an increase in steric repulsion.^{14,15} The CHCl_3 - H_2SO_4 mixture and THF- H_2SO_4 mixture [Figure 3(5,6)] showed the same results; this indicated that the protonation was not strongly affected by the CHCl_3 or THF moiety. From a comparison with PI, which was comprised of ethynyl and active amino groups, the acid sensitivity of PII was about 10 times lower than that of PI. We inferred that the differences were caused by the molecular structures, especially by the changing of the amino group into a dimethylamino group.⁸ According to the previous discussions, the $-\text{NH}_2$ group would not be protonated but would be affected by the intermolecular hydrogen bond, which was supposed to influence the acidochromism sensitivity.⁸ Meanwhile, some linear relations between the absorptions of the polymer-acid mixtures and the concentrations of acids were observed. A sharp decrease in the emission intensity (Figure S4, Supporting Information) was noticed with the addition of trifluoroacetic acid (TFA) to a CHCl_3 solution of the copolymer. It appeared that the protonated species, because of the presence of strong donor-acceptor interactions, experienced a more pronounced dipolar relaxation from the excited state.

On the basis of the previous observations, we believe that PII exhibited the same proteolytic equilibria as PI.⁸ We became interested in the identification of acids from each other,

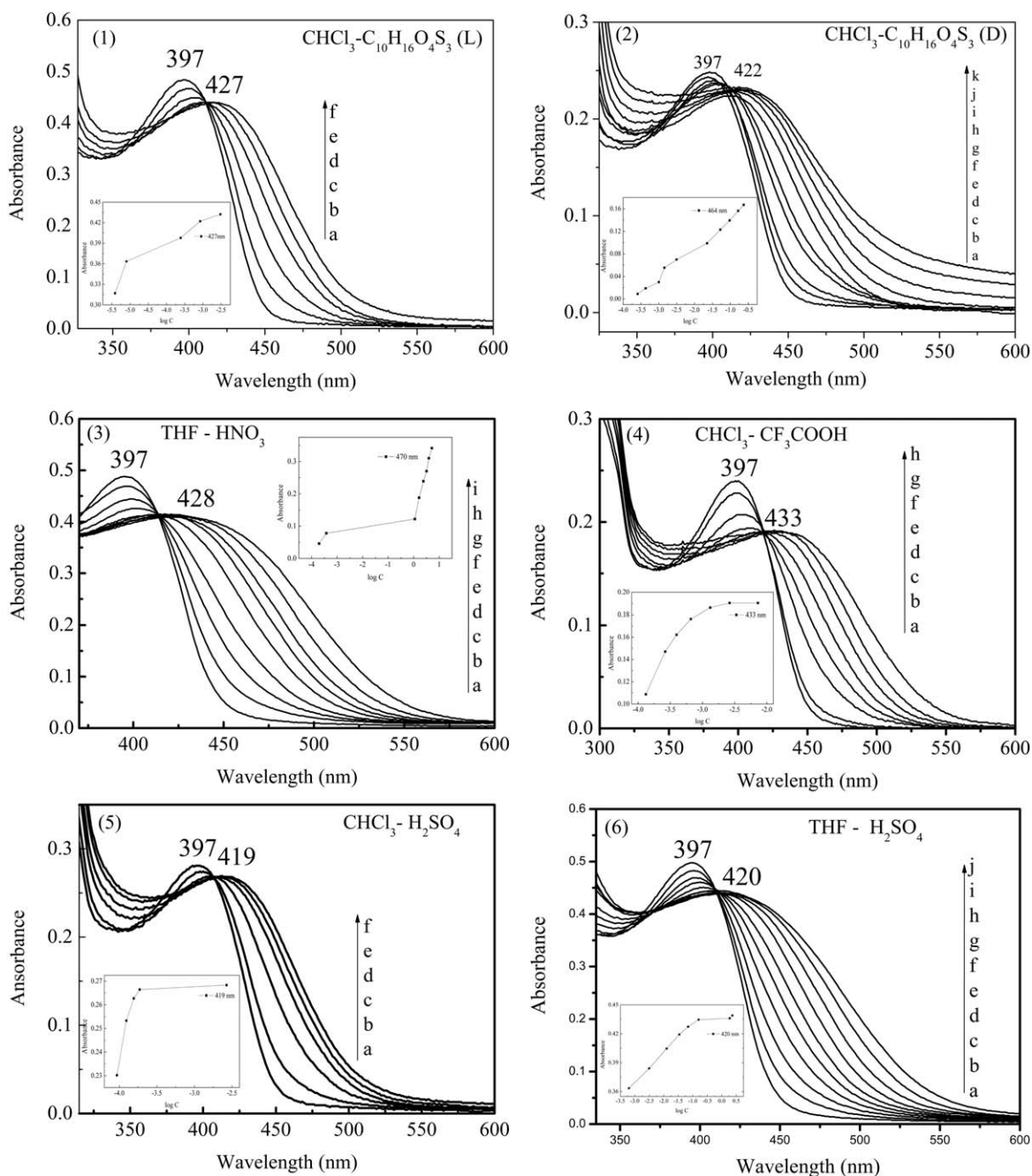


Figure 3. Changes in the absorption spectra of PII (2.12×10^{-3} mol/L) in various polymer–acid mixtures: (1) $\text{CHCl}_3\text{-C}_{10}\text{H}_{16}\text{O}_4\text{S}_3$ mixtures at different concentrations of (L)-camphor-10-sulfonic acid (mol/L) [(a) 0, (b) 3.87×10^{-6} , (c) 7.94×10^{-6} , (d) 2.46×10^{-4} , (e) 8.41×10^{-4} , and (f) 3.02×10^{-3}]; (2) $\text{CHCl}_3\text{-C}_{10}\text{H}_{16}\text{O}_4\text{S}_3$ mixtures at different concentrations of (D)-camphor-10-sulfonic acid (mol/L) [(a) 0, (b) 2.57×10^{-4} , (c) 4.28×10^{-4} , (d) 10.00×10^{-4} , (e) 14.28×10^{-4} , (f) 3.143×10^{-3} , (j) 2.24×10^{-2} , (h) 5.24×10^{-2} , (i) 9.57×10^{-2} , (j) 16.53×10^{-2} , and (k) 23.67×10^{-2}]; (3) THF– HNO_3 mixtures at different concentrations of HNO_3 (mol/L) [(a) 0, (b) 1.96×10^{-4} , (c) 3.77×10^{-4} , (d) 1.11, (e) 1.63, (f) 2.41, (j) 3.20, (h) 3.94, and (i) 5.15]; (4) $\text{CHCl}_3\text{-CF}_3\text{COOH}$ mixtures at different concentrations of CF_3COOH (mol/L) [(a) 0, (b) 1.32×10^{-4} , (c) 2.64×10^{-4} , (d) 3.96×10^{-4} , (e) 6.60×10^{-4} , (f) 13.20×10^{-4} , (g) 26.40×10^{-4} , and (h) 72.60×10^{-4}]; (5) $\text{CHCl}_3\text{-H}_2\text{SO}_4$ mixtures at different concentrations of H_2SO_4 (mol/L) [(a) 0, (b) 6.26×10^{-4} , (c) 31.30×10^{-4} , (d) 12.52×10^{-3} , (e) 34.43×10^{-3} , (f) 68.87×10^{-3} , (g) 159.65×10^{-3} , (h) 1.88, (i) 2.32, and (j) 2.89]; and (6) THF– H_2SO_4 mixtures at different concentrations of H_2SO_4 (mol/L) [(a) 0, (b) 0.93×10^{-4} , (c) 1.24×10^{-4} , (d) 1.56×10^{-4} , (e) 1.87×10^{-4} , (f) 26.77×10^{-4} , (g) 89.05×10^{-4} , (h) 1.045×10^{-2} , and (i) 1.20×10^{-2}]. The insets show the relation between the absorption peak data and the concentrations of the acids.

whereas the L or D type of camphor-10-sulfonic acid, HNO_3 , and H_2SO_4 showed different absorption peaks of the polymer–acid mixtures (the absorption difference was about 5 or 9 nm) in Figure 3(1–3,5).

CF_3COOH was easily recognized when a larger absorption peak difference was found by a comparison with other acids in Figure 3(4). Acidochromism was not observed in some organic or inorganic acid–polymer mixtures, and the properties of the

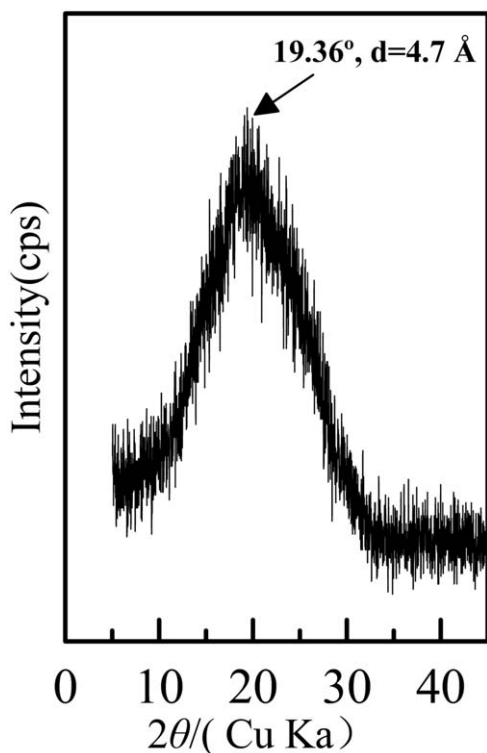


Figure 4. Powder XRD patterns of PII.

differentiation of these two groups of acids from each other are compiled in Table I. The mechanism of differentiating acids from each other could be explained by the proton-offering ability and pK_a of the acids because the observation of one or two isosbestic points suggested the presence of the neutral and pro-

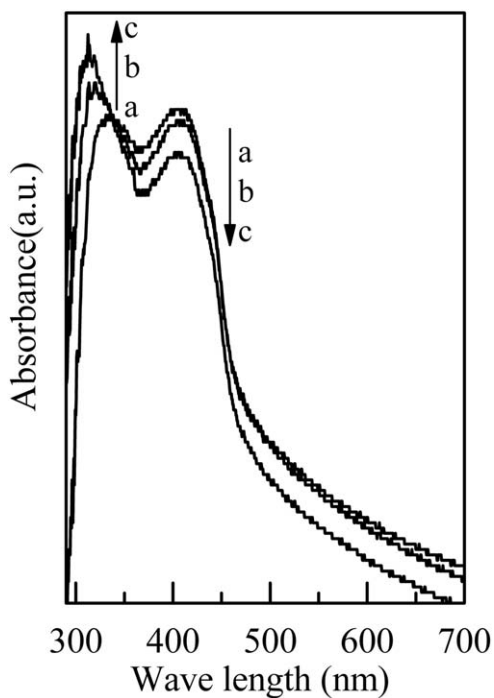


Figure 5. Changes in the UV-vis spectra of the PII film with increases in the temperature: (a) 25, (b) 50, and (c) 100°C.

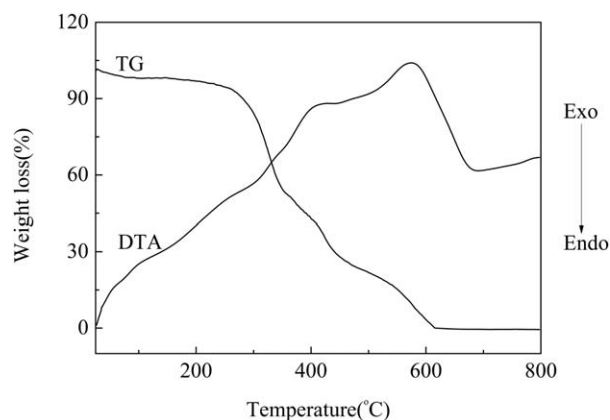


Figure 6. TG and DTA curves for PII at a heating rate of 10°C/min under dry nitrogen.

tonated forms at equilibrium, with the latter being predominant at higher concentrations of acids. It was clear that acidochromism was advanced by the high proton-offering ability or low pK_a of the acids. Detailed studies on the relations between pK_a and the recognition of acidochromism are currently in progress in our laboratory.

Structure of the Copolymer in the Solid State and Thermochromism

The XRD pattern of the PII powder is shown in Figure 4. The copolymer indicated some trends of crystallinity, as shown in Figure 4. π -Conjugated copolymers with long side chains often give an XRD peak in a low-angle region below $2\theta = 10^\circ$ (Cu $K\alpha$), and the d spacing calculated from the peak often corresponds to the distance between the π -conjugated main chains separated by the long side chains.^{11,13–21} When a flat plane of the π -conjugated copolymer forms a face-to-face stacked assembly, it usually gives another XRD peak corresponding to an interplane distance of about $d = 3.8 \text{ \AA}$.^{7–9} All-aromatic polyesters with long side chains also form a similar layered structure.¹² Thus, the XRD peaks in Figure 4 at $2\theta = 19.36^\circ$ ($d = 4.7 \text{ \AA}$) may have been due to a side-to-side distance between loosely packed alkyl chains, and the broad peak at about 20° , which is usually assigned to a side-to-side distance between the interlayer distance, which has a packing dimension of about 4.5 \AA , might have been overlapped with the peak at $2\theta = 19.36^\circ$.^{7–9,22}

The temperature-dependent UV-vis absorption spectrum of PII is depicted in Figure 5. With increasing temperature, the strength of the absorption peak at 397 nm decreased constantly, and in the meantime, we observed the appearance of a new and narrow band at 312 nm. Moreover, the strength of the absorption peak increased with increasing temperature. The thermochromism of the copolymer may have been caused by the film of the copolymer affected by the temperature. When the temperature increased, the movement of the copolymer in the film was enhanced, and according to the XRD analysis, the distance between interlayer was increased. This led to a decrease of the conjugating degree. The energy of the copolymer's conjugated system increased, and this led to a blueshift in the UV absorption peak of the copolymer.

Table I. Acidochromism of the Polymer–Acid Mixtures in the CHCl₃/THF Solutions

Polymer–acid mixture	Acidochromism	Polymer–acid mixture	Acidochromism
Trifluoroacetic acid	+	Acetic acid	–
Methane sulfonic acid	+	Formic acid	–
D-Camphor sulfonic acid	+	Salicylic acid	–
L-Camphor sulfonic acid	+	Citric acid	–
Sulfuric acid	+	Acrylic acid	–
Dodecyl benzene sulfonic acid	+	Boric acid	–
<i>p</i> -Toluene sulfonic acid	+	Benzoic acid	–
Naphthalene-2-sulfonic acid hydrate	+	Kwai acid	–
Naphthalene sulfonic acid	+	Succinic acid	–
<i>P</i> -Toluene sulfonic acid	+	Phthalic acid	–
Nitric acid	+	Malonic acid	–
Hydrochloric acid	+	Stearic acid	–
		Phosphoric acid	–
		Tungstenic acid	–

A plus sign indicates that acidochromism was observed, and a minus sign indicates that it was not observed. Organic acids were observed in CHCl₃ solutions, and inorganic acids were observed in THF solutions.

Thermal Properties

The copolymer's thermal stabilities were determined with thermogravimetry (TG)/differential thermal analysis (DTA) under nitrogen.

Figure 6 shows the thermograms for the neutral copolymer and exhibited an onset of degradation at a temperature greater than 250°C under nitrogen with no decomposition at lower temperatures. The copolymer was fully decomposed at 620°C.

DTA of the copolymer showed a shoulder exothermic peak in the temperature range 250–450°C. This was connected with an overall weight residue of 71.74% and was consistent with the gradual thermal breakdown of the alkyl side chains adjacent to the oxygen and pyrimidine ring.⁸ A very strong exothermic stage observed in the temperature range 451–687°C was due to the phase transition of the copolymer.

Electrochemical Redox Properties

To reveal the protonation nature of PII in the acidic moiety, the electrochemical properties of the copolymer were investigated in a 1M H₂SO₄ aqueous solution by cyclic voltammetry [Figure 7(a)]. The copolymer was electroactive in the –0.2 to 0.8 V region.

The electrochemical behavior of the copolymer depicted facile *p*-doping and good charge transporting properties attributable to the presence of the easily protonated pyrimidinyl unit.⁸ The first oxidation peak (protonation) was not clear at a scan rate if 50 mV/s, but at a scan rate of 25 mV/s, we observed that electrochemical oxidation (or *p*-doping) started at about –0.2 V and gave the first *p*-doping peak about at 0.02 V and a second *p*-doping peak about at 0.1 V. The corresponding *p*-undoping peak for the copolymer about at 0 V. These indicated that the redox of copolymer in the 1M H₂SO₄ solution may possibly have been correlated with the protonation of pyrimidine rings in polymer–acid mixtures. The film revealed stability upon repeated scanning of cyclic voltammetry at a certain low scan

rate, and the peak at 0.02 V decreased with increasing scanning rate. The electrode reaction exhibited quasi-reversibility at a low scan rate, and the nonlinear relation between the peak current and the square root of the scan rate indicated that this was not

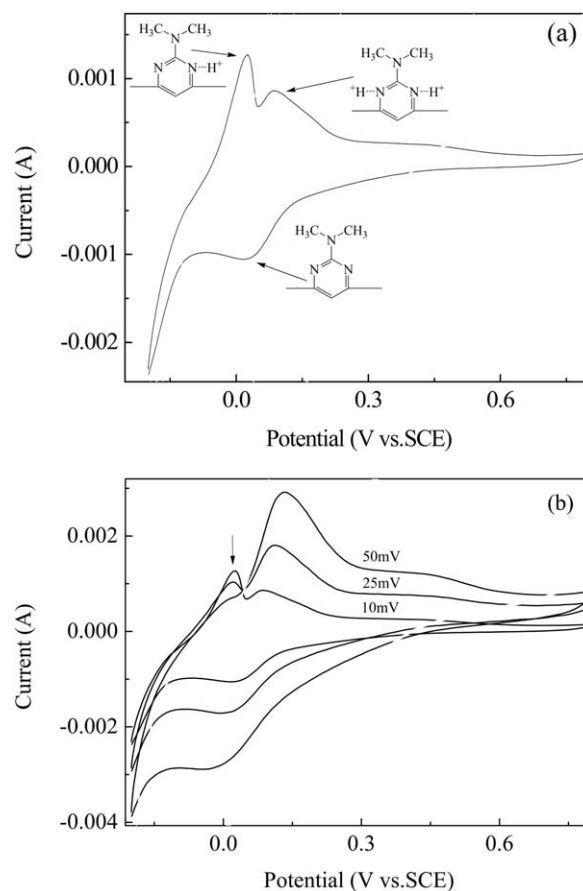


Figure 7. Cyclic voltammograms of the PII cast onto a Pt plate versus SCE in 1M sulfuric acid at scan rates of (a) 10 and (b) 10, 25, and 50 mV.

a diffusion-controlled mass-transport process [Figure 7(b) and Figure S5, Supporting Information].

More detailed studies on the relationship between the acidochromism of the copolymers and their electrochemical properties are currently in progress and will be reported later.

CONCLUSIONS

In summary, a novel, low-molecular-weight, conjugated copolymer composed of alternating electron-donating phenyl and electron-accepting pyrimidine units was synthesized by Grignard coupling reactions. The copolymer depicted good solubility in common organic solvents, such as chloroform, THF, and trifluoroacetic acid. This showed blue emissions in its solution and green emissions in the powder state. The copolymer-acid mixtures in CHCl_3 or THF depicted a bathochromic shift; this was identified to have originated from a π - π^* transition with a major contribution from the protonation of the pyrimidine segment and the minor deduction from the CT between the donor and acceptor fragments. The copolymer acid sensitivity could be influenced by the intermolecular hydrogen bonds, and the $-\text{NH}_2$ group could increase the copolymer's acid sensitivity more than the $-\text{N}(\text{CH}_3)_2$ group. Acidochromism was also used for acid recognition.

The electrochemical behaviors of the copolymer exhibited two oxidation peaks; these corresponded to the copolymer's protonation steps and depicted facile *p*-doping and certain electron-transporting properties in a 1M H_2SO_4 solution.

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