Effects of Acidity and Hydrolysis on the UV and Fluorescence Spectra of Poly(2-acetylamino-1, 4-phenylene) and its Hydrolyzed Derivatives in Acidic Solutions

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ABSTRACT: Poly(2-acetylamino-1,4-phenylene) was synthesized by coupling polymerization of 2,5-dibromoacetanilide. Partially hydrolyzed poly(2-acetylamino-1, 4-phenylene)s were prepared with a degree of hydrolysis per repeating unit from 15 to 98%. UV spectra and fluorescence were studied in DMSO and DMF/water solutions with varied acidity. The UV absorption maximum of poly(2-acetylamino 1,4-phenylene) is red-shifted from 255 to 285 nm with increasing degree of hydrolysis. At low degree of hydrolysis the emission spectra of polymers in DMSO/water solutions at different concentrations show that the emission λ_{max} is almost independent of concentration. However, when the extent of hydrolysis increased, the emission bands were red-shifted and became dependent of concentration. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 106: 1806–1812, 2007

Key words: poly(2-acetylamino-1,4-phenylene); hydrolysis; acidity

INTRODUCTION

Opto-electronic devices, e.g. light-emitting diodes (LEDs),[1] thin film transistors (TFTs),[2] and photovoltaic cells (PVCs),[3] utilizing π-conjugated polymers as active layers have become a subject of great interest since the first report of electroluminescence devices (LEDs) based on poly(phenylenevinylene) in 1990.[1] The application of conjugated polymers as active materials in Opto-electronic devices offers several advantages such as easy processing by solvent or spin castings from solution, and the chemical tailoring of the absorption and emitting wavelength.[4,5]

Blue light emitting diodes using N-containing rigid polymers as the emitting layer have been reported in the literature.[6–9] These polymers are soluble in acidic solvents such as formic and dichloroacetic or sulfuric acids. The excitation and emission wavelengths were varied by the protonation and methylation of the nitrogen atom.[10–15] We have studied the fluorescence of poly(pyridine),[10] poly(2,6-[4-phenyl quinoline]),[11] PPQ, and poly (2,6-[p-phenylene]-4-phenyl quinoline),[11] PPPQ, and the dependence of the emission spectra on the acid strength of the solvent. However, the effects of the protonation and methylation of the aforementioned three polymers on photoluminescent properties can only be studied in protic acidic solvents in which the polymers are soluble but not in common organic solvents due to poor solubility. To alleviate this shortcoming we have now synthesized a new polymer; poly(2-acetylamino-1,4-phenylene), PAAP. The acetylamino groups in PAAP were then converted to amino groups by hydrolysis to obtain a series of polymers HPAAP with conversions up to 98%. The resulting polymers were soluble in organic and in mixed organic/aqueous solvents. The facile solubility in mixed solvents offers the advantage of being able to vary the pH of the medium without grossly changing the nature of the solvent. The results are reported below.

EXPERIMENTAL SECTION

Materials

1,4-dibromoaniline, acetic anhydride, anhydrous dimethylformamide (DMF), anhydrous toluene, 2,2′-bipyri-

dine, zinc, nickel chloride, 1,5-cyclooctadiene, and 1M solutions of HCl and NaOH were purchased from Aldrich and used without further purification. Zinc was activated by treated with 1% hydrochloric acid and diethyl ether before use.

Characterization

^{1}H and ^{13}C NMR spectra were recorded by using a Brucker 300 MHz spectrometer. Elemental analysis (C, H, N) was measured by Elementar Vario EL III. GC-MS analysis of the monomers was performed on a VG Quattro instrument, EI 70 eV, to check their purity and mass. The fluorescence spectra were collected by using Perkin-Elmer LS-50B Luminescence spectrophotometer and UV spectra were recorded by Varian Cary 2300 spectrophotometer. The pH measurement was performed at 25°C calibrated with standard buffer solutions at pH 4.00 and 7.01. Gel permeation chromatography (GPC) analysis was conducted with a Polymer Laboratories HPLC system equipped with Polymer Laboratories ultra Styragel Mixed-D 3, 2, and 1 (7.8 mm i.d. × 300 mm) columns in series using polystyrene as the standard and tetrahydrofuran as the eluent. The inherent viscosity was measured in DMF at 25 ± 0.05°C by an Ubbelohde viscometer.

Sample preparation

Filtered solutions were employed to minimize light scattering in UV and fluorescence measurements. The solutions were aerated. Purging with nitrogen did not significantly alter the emission intensity.

Monomer synthesis

2,5-Dibromoacetanilide (1)

A mixture containing 1, 4-dibromoaniline (20.0 g, 80 mmol) and acetic anhydride (100 mL) was stirred for 3 h at room temperature (Scheme 1). The mixture was then washed with water (200 mL) three times. The precipitate was recrystallized in methanol to give 22.0 g of a white product. The product yield was 93%. m/z: 293. Anal. Calcd for $C_8H_7Br_2NO$ (292.955 g/mol): C, 32.80; H, 2.41; N, 4.78. Found: C, 32.77; H, 2.43; N, 4.75. ^{1}H NMR. (300 MHz, CDCl$_3$, ppm) : δ 8.51 (s, 1 H, NHAc), δ 7.57 (s, 1H, Ph H), δ 7.32 (d, 1H, Ph H), δ 7.01 (d, 1H, Ph H), δ 2.19 (s, 3H, NCOCH$_3$) (Scheme 1).

Polymerization

Poly(2-acetylamino-1,4-phenylene)

An anhydrous dimethylformamide (100 mL) solution containing the monomer 2,5-dibromoacetanilide (7.00 g,
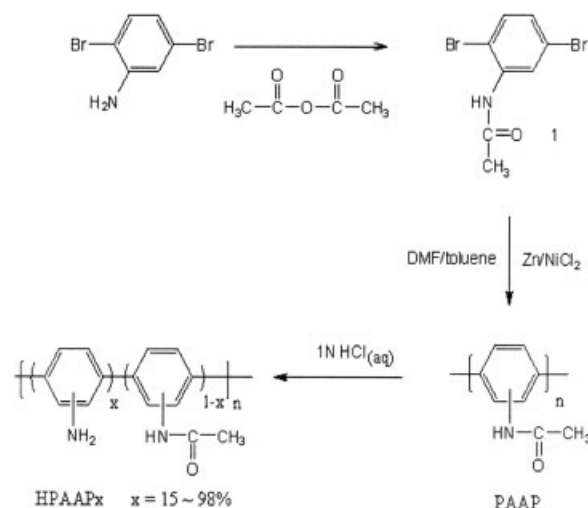
Scheme 1 Synthesis of poly(2-acetylamino-1, 4-phenylene) and its hydrolyzed derivatives.

0.02 mol), NiCl$_2$ (0.26 g, 0.002 mol), bipyridine (4.1 g, 0.15 mol), Zn (5.28 g, 0.08 mol), and 1,5-cyclooctadiene (5 mL, 0.04 mol) in a 250 mL round bottom flask equipped with a condenser was stirred at 75°C for 48 h. The reaction mixture was poured into 0.01M HCl (200 mL) and stirred for 30 min to remove the Zn residue. The precipitate was filtered and washed with 0.01M HCl (200 mL) three times. It was then redissolved in DMF, precipitated in water and dried under vacuum. (M_w/M_n = 1.4, M_n = 6300). Anal. Calcd for $(C_8H_7NO)_n$, $(133.15)_n$ g/mol: C, 72.16; H, 5.30; N, 10.52. Found: C, 71.31; H, 5.24; N, 10.39. ^{1}H NMR. (300 MHz, d$_7$-DMF, ppm) δ 8.05 (s, 1 H, NHAc), δ 7.72 (s, 1H, Ph H), δ 7.22–7.49 (m, 2H, Ph H), δ 2.19 (s, 3H, NCOCH$_3$).

Hydrolysis of poly(2-acetylamino 1, 4-phenylene) (HPAAP)(Scheme 1)

A solution containing hydrochloric acid (1M, 180 mL) and poly(2-acetylamino-1,4-phenylene) (0.69 g, 5.61 mmol) in a 250 mL round bottom flask was stirred at reflux for various time periods. The reaction mixture was poured into 200 mL of water and neutralized with sodium carbonate aqueous solutions. The precipitates were filtered and washed with water (200 mL) three times. The polymers were then redissolved in DMF, precipitated in water, and dried under vacuum.

RESULTS AND DISCUSSION

Polymerization and molecular weight

The coupling polymerization of 2,5-dibromoacetanilide yielded chain molecules of poly(acetylamino-1,4-phenylene), PAAP, (Scheme 1) with head-to-head
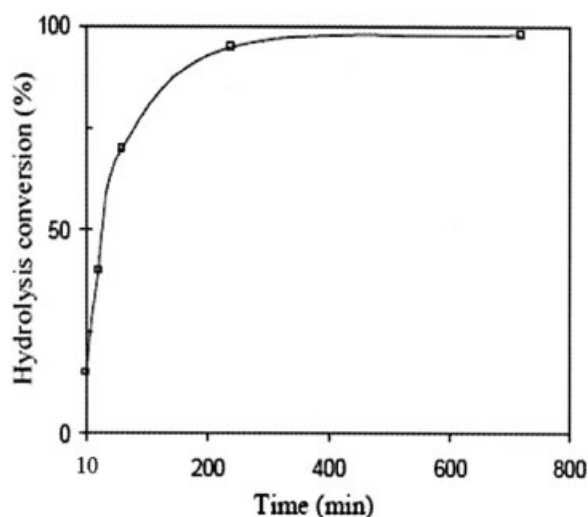
Figure 1 Hydrolysis of PAAP as a function of time.

(HH), head-to-tail (HT), and tail-to-tail units, the HT unit being much more frequent[7,8] although it can not be totally identified by our NMR data. Henceforth, the resulted polymers were named poly(2-acetyl-amino-1,4-phenylene), PAAP, throughout this study. The molecular weight of PAAP was estimated from GPC using polystyrene as standard, to be 6300 (M_n) which corresponds to about 48 repeating units. The polydispersity index (M_w/M_n) of both polymers is about 1.4. Although the molecular weight obtained from GPC does not represent the true value, the information about the molecular weight distribution is useful in the assessment of the synthetic procedure used for the coupling reaction. The intrinsic viscosity of PAAP was 0.15 dL/g measured in dimethylfor-mamide at 25°C.

Hydrolysis

Hydrolysis of poly(2-acetylamino-1,4-phenylene) was done in an aqueous solution of $1M$ hydrochloric acid at reflux for various time periods. The degree of the hydrolysis was determined by ^{1}H NMR spectros-copy. The peak ratios, 1.9 ppm for the acetyl group and 7.2–8.0 ppm for the aromatic protons, were determined, and the degrees of hydrolysis were cal-culated at different reaction times (Fig. 1): 15% at 10 min, 40% at 20 min, 70% at 1 h, 95% at 4 h, and 98% at 12 h. Although the degrees of hydrolysis obtained here could be deviated from the true value owing to instrument's ineffective accuracy, the infor-mation about the relative degrees of hydrolysis is meaningful for studies in later sections about under-standing the relationship between the UV-absorption or fluorescence emission spectra and the variation of structural conformations due to hydrolysis. Each polymer was identified in the ensuing code by the

percent conversion, for example, HPAAP40 for the sample with 40% amino group conversion.

Solubility

The PAAP and HPAAP polymers are soluble in dimethylformamide (DMF), dimethyl sulfoxide (DMSO), and in mixed organic/aqueous solvents. For the purpose of varying the pH of the solvent media we found it convenient to use DMSO/water mixtures to which 2 mL of $NaOH_{(aq)}$, H_2O or $HCl_{(aq)}$ were mixed with 8 mL of DMSO to form 10 mL sol-utions. The 80/20 (v/v) DMSO/water mixture was used throughout this investigation to study the effect of pH on fluorescence.

UV-absorption spectra

The UV absorption spectra of the polymers in mixed DMSO/water solution are sensitive to solvent com-position. In 20/80 DMSO/water solution the spectra are broad and featureless, but in an 80/20 mixture, distinct absorption bands can be identified. Hence-forth, all measurements were carried out in 80/20 (v/v) DMSO/water mixture. The concentration 0.005 g/dL was chosen throughout the UV measurements because fluorescence emission intensity, which will be discussed later, was highest in the concentration range between 0.005 and 0.01 g/dL. The UV spec-trum of PAAP is similar to that of HPAAP15 in Fig-ure 2(a), therefore, not shown. In Figure 2, two absorption bands, at 255 and 285 nm respectively, are seen for each hydrolyzed polymer (HPAAP). The intensity of the band at 255 nm decreases steadily with increasing degree of hydrolysis but the band at
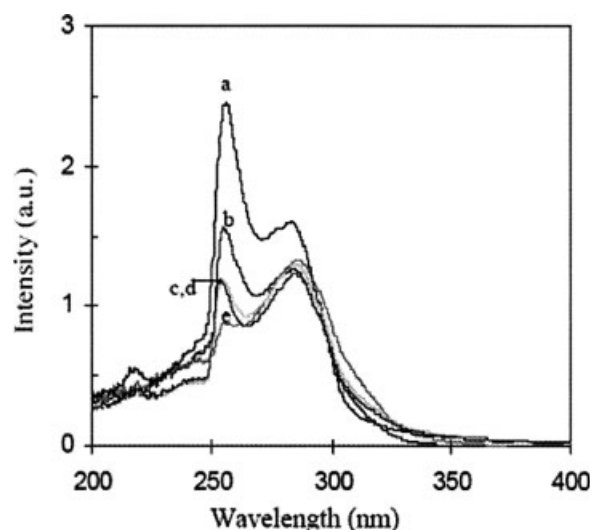

Figure 2 UV spectra of HPAAPs in 80/20 DMSO/H$_2$O at 0.005 g/dL as a function of degree of hydrolysis: (a) 15%, (b) 40%, (c) 70%, (d) 95%, and (e) 98%.

TABLE I
pH Values of Polymer Solutions

DMSO/ H$_2$O	1N NaOH	H$_2$O	0.1N HCl	0.5N HCl	1N HCl	10N HCl
pH	14	4.6	2.4	1.6	1.4	0.5

285 nm changes in a different way. After an initial decrease in intensity from HPAAP15 to HPAAP40, there are only minor changes with additional conversion. Since the 255 nm band decreases continuously with an increasing hydrolysis, the intensity ratio of 285–255 nm peaks increases monotonically with the amino content. This red shift could arise in part because the amino group has a stronger electron donating power than the acetylamino group, and thus has a favorable effect on conjugation. A second possible contribution to the shift towards 285 nm is the smaller steric interaction between adjacent phenyl rings because of the smaller size of the amino group, thereby reducing ring twisting and increasing coplanarity.

The effect of acidity of the medium on fluorescence was studied with two polymers, HPAAP40 and HPAAP98. Each 10 mL of the solution consists of 8 mL of DMSO and 2 mL of aqueous NaOH or HCl. The pH values of the solutions are listed in Table I.

The UV spectra of HPAAP40 in these solutions are shown in Figure 3. In a basic solution (curve a), the intensity of the 255 nm band is weaker than that of the 285 nm band. Since the unprotonated —NH$_2$ group is the dominant species at pH 14, the result is consistent with the earlier explanation of the 285 nm
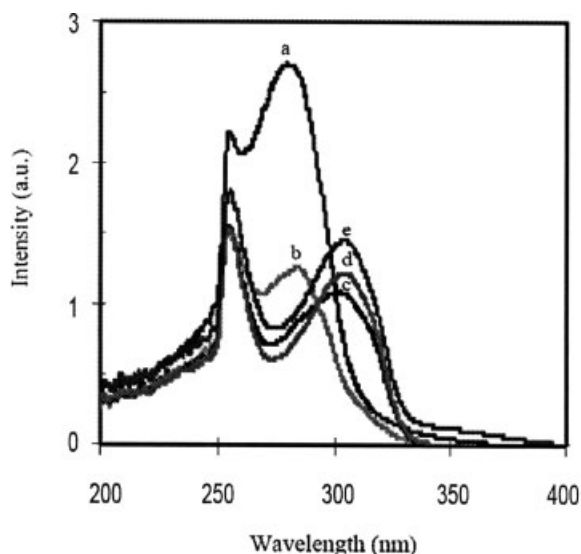
band being favored by —NH$_2$ groups. In the mixed solvent without added NaOH or HCl, at pH value of 4.6, protonation is expected to occur, though not to a large extent. This is accompanied by a diminution of the intensity of the 285 nm peak (curve b). In more acidic media with pH of 2.4 or less, the 285 nm band is red shifted to 305 nm (curves c, d, e). The intensity of the shifted band increases with increasing acidity or decreasing pH. The red shift implies a smaller energy of the π-π* transition of the protonated structure.

The changes in UV spectra with increasing acidity for the HPAAP98 polymer (Fig. 4) follow a trend similar to that observed for HPAAP40. The λ_{max} values are almost the same in Figures 3 and 4, but the relative intensities are different. For HPAAP98, the intensity of the 255 nm band decreases more quickly with increasing acidity. The 305 nm bands which are red shifted from 285 nm have comparable intensities with their counterparts in HPAAP40, but the intensity ratios of the 305–255 nm bands are much higher for HPAAP98. Apparently, the 255 nm transition, probably n-π*, is quenched by —NH$_3^+$ formation. Although the protonated amino group has a stronger electron withdrawing effect than —NH$_2$, the red shift from 285 to 305 nm suggests that the coplanar conformation of the molecule as affected by protonation outweighs the unfavorable electron withdrawing effect in its contribution to the transition energy.

The UV spectra of HPAAP40 and 98 in DMF or DMSO are similar to those obtained in 80/20 DMSO/H$_2$O and therefore not reproduced. However, the fluorescence emission spectra are different, as will be seen in the next section.
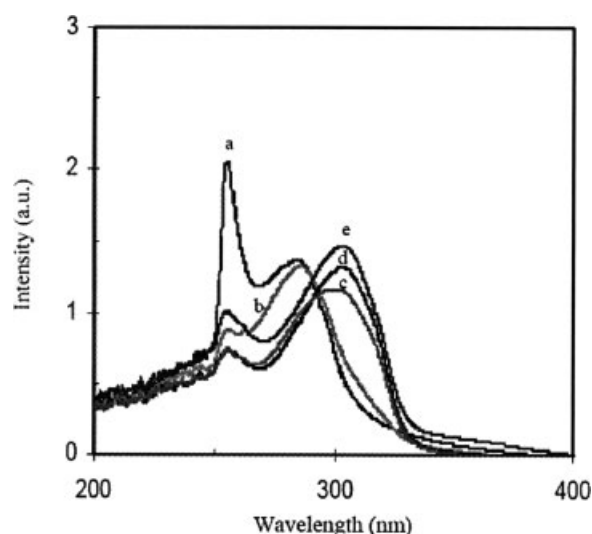

Figure 3 UV spectra of HPAAP40 in 80/20 DMSO/H$_2$O at 0.005 g/dL: (a) 1M NaOH, (b) H$_2$O, (c) 0.1M HCl, (d) 0.5M HCl, and (e) 1M HCl.

Figure 4 UV spectra of HPAAP98 in 80% DMSO/H$_2$O at 0.005 g/dL: (a) 1M NaOH, (b) H$_2$O, (c) 0.1M HCl, (d) 0.5M HCl, and (e) 1M HCl.

Fluorescence emission spectra

Acidity effect

Before we engaged in the study of the variation of the fluorescence behavior as a function of acidity, it was necessary to select a suitable concentration at which the emission intensities of different solutions could be compared. In Figure 5, the emission spectra of PAAP in DMSO/water solutions are shown for solution concentrations ranging from 1×10^{-5} to 0.5 g/dL when excited at 280 nm. The intensity is highest at 5×10^{-3} g/dL, with smaller values at both lower and higher concentrations. The results obtained with excitation at 330 nm are similar and are not shown. In the series of experiments in which the pH values of the solutions were varied, the emission intensity at 5×10^{-3} g/dL was always at or near the highest value observed. Therefore, a single concentration was used to compare the intensities in different acidic solutions. The results are shown in Figures 6 and 7 for HPAAP40 and 98, respectively. The λ_{max} was located at 405 nm in both figures but interestingly the highest emission intensity was observed in each case when no base or acid was added.

In a weak acid solution at pH = 4.6 the rod-like molecules are partially protonated and the electrostatic repulsion between chains reduces the probability of concentration quenching.[10,11] In basic solutions the —NH$_2$ groups are in the unprotonated form whereas in acidic media the amino groups are substantially in the protonated form. Yet the intensities in these two acidity regimes are both lower than the value at pH = 4.6. The decrease in emission inten-
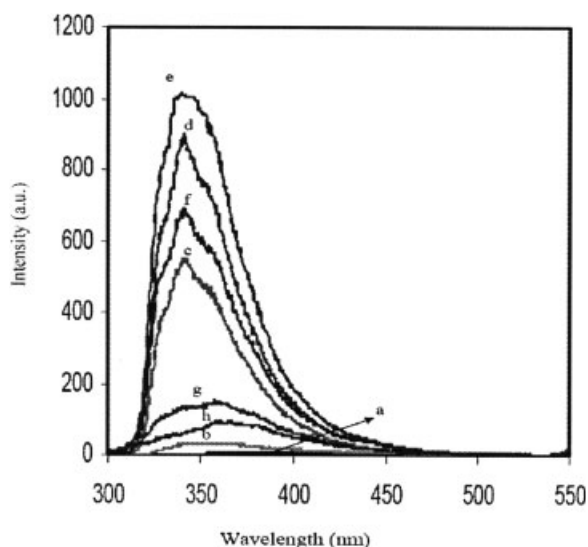

Figure 6 Emission spectra of HPAAP40 (0.005 g/dL) in 80/20 DMSO/H$_2$O excited at 330 nm: (a) 1M NaOH, (b) H$_2$O, (c) 0.1M HCl, (d) 0.5M HCl, (e) 1M HCl, and (f) 10M HCl.

sity with increasing acidity observed in this system is opposite to the trend found for poly(2,5-pyridine)[10] and poly(3,5-pyridine).[10] Since protonation affects both the ground and the excited states, there is a complex interplay of many conflicting factors: the electron withdrawing power of the —NH$_3^+$ moiety, the effect of electrostatic repulsion on the coplanarity of adjacent phenyl groups, and the possible formation of charge transfer complexes which was discussed by Turro and Coworkers[12,13] in their studies of fluorescence poly(4-vinylpyridine) in aqueous solutions.

Figure 5 Emission spectra of PAAP excited at 280 nm in DMSO/H$_2$O solutions: (a) 0.5 g/dL, (b) 0.1 g/dL, (c) 0.02 g/dL, (d) 0.01 g/dL, (e) 0.005 g/dL, (f) 0.001 g/dL, (g) 0.0001 g/dL and (h) 0.00001 g/dL.
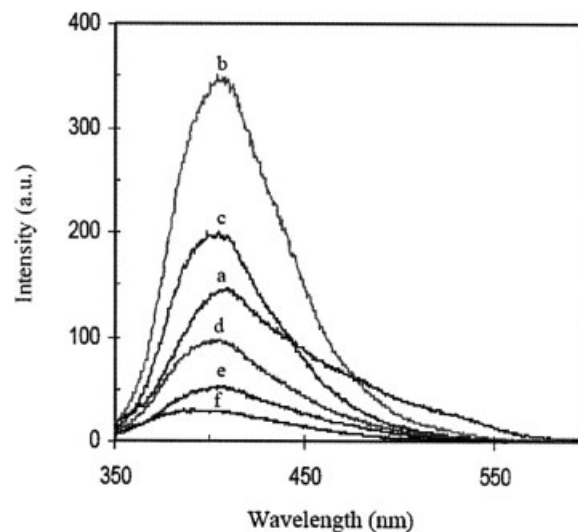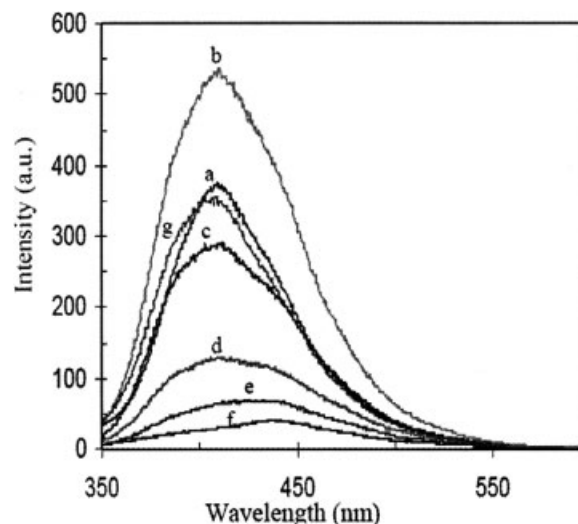

Figure 7 Emission spectra of HPAAP98 (0.005 g/dL) in 80/20 DMSO/H$_2$O excited at 330 nm: (a) 1M NaOH, (b) H$_2$O, (c) 0.1M HCl, (d) 0.5M HCl, (e) 1M HCl, (f) 10M HCl and (g) pH = 8 (buffer).

Concentration effect

The emission spectra of HPAAP15 in DMSO/water solutions at different concentrations are similar to that of PAAP in Figure 5, which show that the emission λ_{max} is almost independent of concentration. However, the emission spectrum was very different from that of HPAAP15 when the extent of hydrolysis increased. The fluorescence emission spectra of HPAAP98 DMSO/water solutions are shown in Figure 8. Compared to the spectra of HPAAP15, the emission bands of HPAAP98 were red-shifted; this indicates that HPAAP98 has a longer average conjugation length or a different species from that of HPAAP15. The exact origin for this is not clear. However, it may stem from: (1) hydrogen bond formation between adjacent carbonyl and amino groups,[14–16] which results a higher coplanarity between adjacent phenyl rings, or (2) complex formation that causes a different radiating species.[17–19] Figure 8 shows that the emission intensities at ~405 nm gradually increased as the concentration of HPAAP98 increased from 1×10^{-4} to 7.5×10^{-3} g/dL. At 0.01 g/dL, the emission peak shifted suddenly to ~530 nm as though a completely different mechanism becomes operative. Upon further increase in concentration, the emission intensity decreased to low levels until it is barely measurable at 0.3 g/dL. The large red shift from 405 to 530 nm and the attendant decrease in emission intensity with increasing concentration was typical of excimer/exciplex emission from rod like conjugated polymers.[20–23] Interestingly, we also found that the emission spectrum of HPAAP98 was strongly dependent on the nature of the solvent. For example,
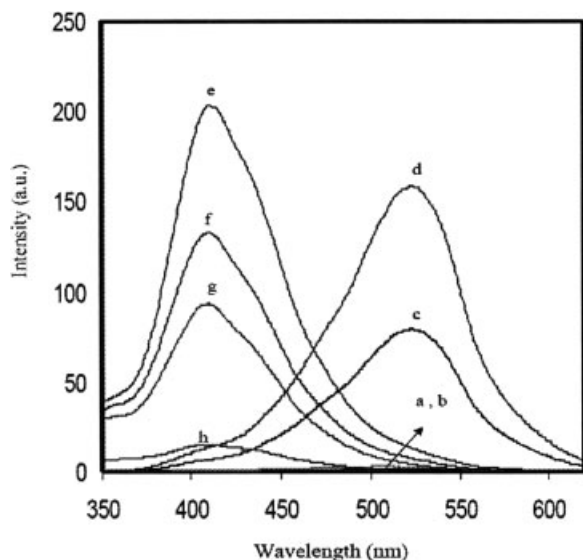

Figure 9 Normalized emission spectra of HPAAP98 excited at 330 nm in DMF/H$_2$O solutions: (a) 0.1 g/dL, (b) 0.01 g/dL, (c) 0.005 g/dL, and (d) 0.0025 g/dL.

the trend of concentration dependence of fluorescence of HPAAP98 in DMF/water solutions (Fig. 9) is similar to that in DMSO/water solutions. However, in DMF/water solutions, two peaks at ~375 and 480 nm, respectively, were observed at each concentration, while in DMSO/water solutions, only one emission peak was observed at low concentrations. To compare relative intensities of both peaks at ~375 and 480 nm, the normalized fluorescence emission spectra of HPAAP98 in DMF/water solutions are shown in Figure 9. Assuming that the two emission peaks observed in DMF/water solutions can be interpreted as the responses of two coexisting species, the results in DMSO/water solutions suggest that the two species equilibrate quickly at low concentrations, compared to the time scale of fluorescence, so that only one emission peak is seen. We also note that the emission intensity at ca 480 nm increases when the concentration of HPAAP98 increases. This may indicate the number of excimer/exciplex increases as the concentration of polymer increases.

CONCLUSION

Poly(2-acetylamino-1,4-phenylene) and its hydrolyzed derivatives, partially hydrolyzed poly(2-acetylamino-1,4-phenylene)s (15–98% of hydrolysis), were synthesized. The UV absorption spectra of the polymers in mixed DMSO/water solution are sensitive to solvent composition. In 20/80 DMSO/water solution the spectra are broad and featureless, but in an 80/20 mixture, two absorption bands, at 255 and 285 nm respectively, are seen for each polymer. Since the 255 nm band decreases continuously with

Figure 8 Emission spectra of HPAAP98 excited at 330 nm in DMSO/H$_2$O solutions: (a) 0.3 g/dL, (b) 0.1 g/dL, (c) 0.05 g/dL, (d) 0.01 g/dL (e) 0.0075 g/dL, (f) 0.0025 g/dL, (g) 0.001 g/dL, and (h) 0.0001 g/dL.
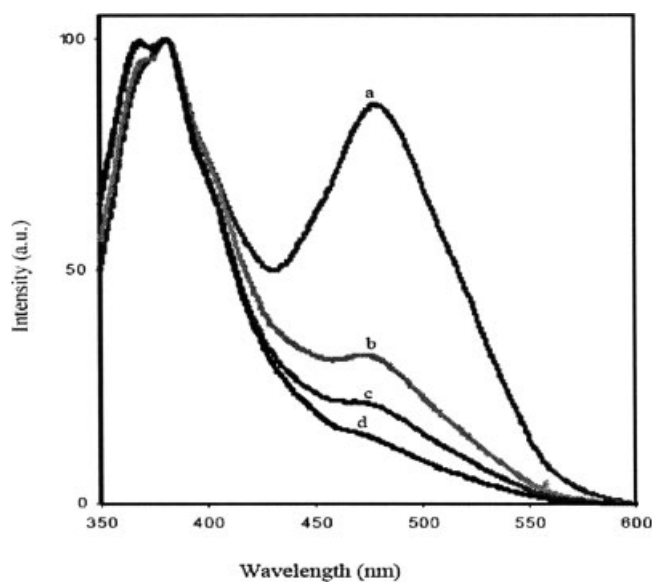
an increasing hydrolysis, the intensity ratio of 285–255 nm peaks increases monotonically with the amino content. Furthermore, in acidic media with pH of 2.4 or less, the 285 nm band is red shifted to 305 nm and the intensity of the shifted band increases with increasing acidity or decreasing pH. Apparently, the 255 nm transition, probably n-π*, is quenched by —NH$_3^+$ formation.

The fluorescence emission efficiencies of DMSO/water solutions of HPAAP are sensitive to pH changes. In a weak acid solution at pH = 4.6 the rod-like molecules are partially protonated and the electrostatic repulsion between chains reduces the probability of concentration quenching. In basic solutions the —NH$_2$ groups are in the unprotonated form whereas in acidic media the amino groups are substantially in the protonated form. Yet the intensities in these two acidity regimes are both lower than the value at pH = 4.6. The decrease in emission intensity with increasing acidity observed in this system is similar to the trend found in aqueous solutions of fluorescence poly(4-vinylpyridine) studied by Turro and coworkers. We also found that the emission spectrum of HPAAP was strongly dependent on the nature of the solvent. For example, the trend of concentration dependence of fluorescence of HPAAP in DMF/H$_2$O is similar to that in DMSO/H$_2$O. However, in DMF/water solutions, two peaks at ∼ 375 and 480 nm, respectively, were observed at each concentration, while in DMSO/water solutions, only one emission peak was observed at low concentrations. These results are explained in terms of structure features of the electronically excited polymer, in particular its conformation changes and acid–base properties.

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