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Syntheses and Characterization of Model Imide Compounds and Chemical Imidization Study

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ABSTRACT: In order to characterize various depolymerization and imidization reactions involved during thermal and chemical imidazation of poly(amic acid) by UV-visible and fluorescence spectroscopies, several model compounds have been synthesized and characterized from 1,5-diaminonaphthalene (DAN) and phthalic anhydride. The model compounds synthesized are the derivatives of DAN such as amic acid-amine, diamic acid, amine-imide, amic acid-imide, diimide, and diisoimide. Only DAN is found to be strongly fluorescent while amic acid-amine, amine-imide, and diisoimide are very weakly fluorescent. The others have negligible fluorescence. During imidization, therefore, the fluorescence intensity can be used to quantify the amount of depolymerized DAN. Due to strong substituent effects on the UV-visible spectra, the model compounds exhibit characteristic absorption maxima and extinction coefficients. Proton NMR, IR, and differential scanning calorimetry have been used also to confirm the chemical structures and the purity of the model compounds. Chemical imidization using acetic anhydride and pyridine for polyamic acid made from DAN and a partially fluorinated dianhydride has been investigated. Deconvolution of UV-visible spectra on the basis of the model compounds provided the composition of the involved species such as diamic acid, diimide, and diisoimide. The rate constants for chemical imidization occurring in 1% solution in *N*-methyl-2-pyrrolidinone indicate a fast first step in which simultaneous conversion of diamic acid to diimide and diisoimide takes place, followed by slower conversion of diisoimide to diimide in a second step.

Introduction

Polyimides are important high-performance materials due to their high thermal stability, strength, desirable dielectric properties, and resistance to organic solvents. Polyimides are often made by a two-step process; first, the poly(amic acid)s are made by reaction of diamines with dianhydride at low temperatures in polar solvents, followed by a second step during which poly(amic acid)s are cured by either thermal or chemical treatment to obtain polyimides. During thermal imidization of poly(amic acid)s, some side reactions such as the reverse depolymerization from poly(amic acid)s are known to occur. For example, Kolegov studied the relative importance of some side reactions when 4,4′-diaminodiphenyl ether was reacted with pyromellitic dianhydride in dimethylformamide at ambient temperature.[1] It was found that the imidization was 2-3 orders of magnitude slower than that for the reverse depolymerization at room temperature.[1] More recently, Young et al. synthesized polyimides which are soluble in the same solvent at all stages of imidization. By careful investigation using viscosity, low-angle laser light scattering combined with GPC, they observed an initial reduction in molecular weight during cure, followed by increase in molecular weight upon extensive imidization.[2] There was a concurrent appearance and eventual disappearance of anhydride peak at 1860 cm^{-1} in the IR spectra. They concluded that the poly(amic acid) depolymerized during initial cure to generate anhydride and presumably amine, which eventually reacted at higher temperature. Thus, the depolymerization due to the reverse reaction from poly(amic acid) to amine and anhydride seems to play an important role in the synthesis of high molecular weight polyimide. The relative rates of involved reactions will be decided by the equilibrium constant and its temperature dependence as well as the temperature dependence of the imidization rate. Kamzolkina et al. found that the equilibrium constant is very temperature dependent.[3] For example, it changed from 1970 L/mol at 40 °C to 4 L/mol at 200 °C in acetonitrile. This means that at the high temperature, depolymerization is particularly important at early stages of cure.

While the IR peak due to anhydride seems well identified for a quantitative analysis, such an analysis for free amine end groups is very difficult due to the close overlap of amine and acid hydroxyl bands. As a sensitive technique to monitor amine groups, which were formed due to the depolymerization, fluorescence spectroscopy in combination with UV-visible spectroscopy would be desirable since aromatic amines are known to be fluorescent.[4] In our recent work to understand the kinetics and the mechanisms of thermal imidization of poly(amic acid)s, we used *p,p*′-diaminoazobenzene as a diamine to synthesize several model compounds involved during imidization and side reactions.[5] Due to the strong substituent effects as the diamine changes to diamic acid, amic acid-imide, diimide, and amine-imide, it was possible to distinguish these products in UV-visible spectra and to quantify their formation and disappearance. Unfortunately, none of these products exhibited significant fluorescence. However, we have demonstrated how UV-visible and fluorescence spectroscopic analyses can be used to study cure reactions in other polymers such as epoxies,[6] polyurethanes,[7] and polyamides[8] as well as for in-situ cure monitoring in epoxy composites.[9a]

Since fluorescence can be a much more sensitive method to monitor even minute amounts of depolymerized diamine or amine-amic acid, we decided to investigate 1,5-diaminonaphthalene (DAN) as a model diamine. The reasons for choosing DAN are its potential as an extrinsic sensor to monitor cure for in-situ processing since its reactivity can be correlated to other aromatic diamines and its fluorescence spectra appearing in longer wavelengths than those of the matrix polyimides. Chart I shows the chemical structures for the model compounds involved in both chemical and thermal imidization. We first synthesized various model compounds from DAN and phthalic anhydride and characterized their UV-visible and fluorescence spectra. Their spectra will be representative of the poly(amic acid)s, polyimides, and their intermediates as long as the dianhydride has a conjugation blocker

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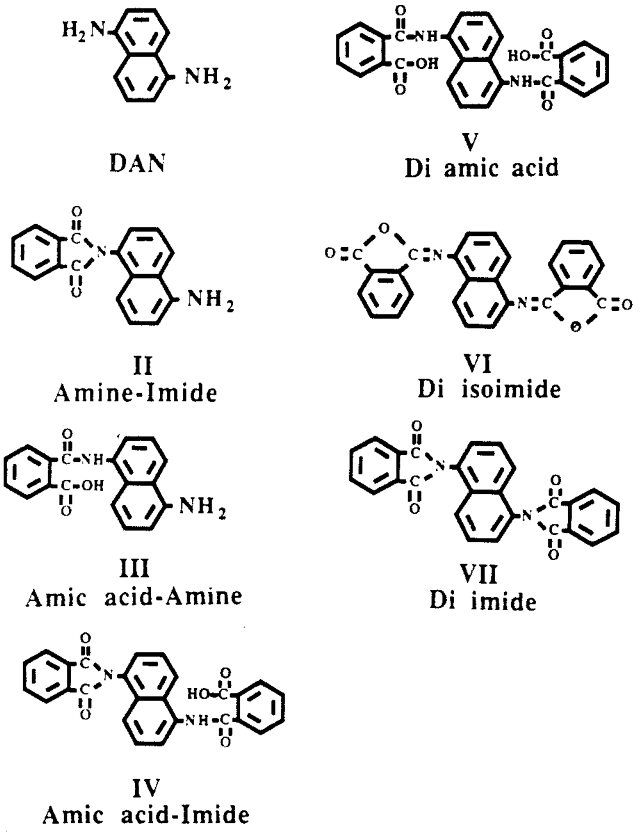
Chart I
Chemical Structure of Model Compounds



DAN

V
Di amic acid

II
Amine-Imide

VI
Di isoimide

III
Amic acid-Amine

VII
Di imide

IV
Amic acid-Imide

between two aromatic rings such as 2,2-bis(3,4-dicarboxyphenyl)hexafluoropropane dianhydride (6FDA). While some recently developed polyimides are soluble, most polyimides are insoluble in many solvents, making their thorough characterization difficult. However, UV–visible and fluorescence techniques can be applied to insoluble polymers, making them powerful tools for polymer characterization.

In order to demonstrate how the spectra of various model imide compounds can be used to analyze the imidization processes, we report some results of the chemical imidization study using an acetic anhydride–pyridine mixture as dehydrating agent, starting from polyamic acid made with DAN and 6FDA. In the following paper,[9b] we describe a more extensive example of the analyses of thermal imidazation using the UV–visible and fluorescence spectra of model imide compounds in combination with size-exclusion chromatography and FTIR.

Experimental Section

Solvents and Reagents. Spectrophotometric grade *N*-methyl-2-pyrrolidinone (NMP) from Aldrich Chemical was dried over molecular sieves (4 Å), followed by vacuum distillation over P_2O_5. 1,5-Diaminonaphthalene (DAN) with purity greater than 98% was purchased from Fluka Chemical, recrystallized from an acetone–petroleum ether (bp 30–60 °C) mixture, rinsed with petroleum ether, and vacuum dried before sublimation. The melting point of sublimed DAN was 190.3 °C, which is very close to the reported melting point of 190 °C.[10] Other reagents such as phthalic anhydride and solvents of highest purity were purchased from Aldrich Chemical or Fischer Chemical and used without further purification. Electronic grade 6FDA was a gift from Hoechst Celanese, Coventry, RI, and was used without further purification.

Synthesis of Model Compounds. (1) Amic Acid–Amine. The synthesis of amic acid–amine followed generally reaction 1 in Scheme I. A solution of 7.80 g (52.7 mmol) of phthalic anhydride and 5.20 g (32.9 mmol) of DAN in 450 mL of acetone was stirred for 24 h at room temperature in an airtight, stoppered 500-mL three-neck flask. After the precipitate, which is diamic acid, was filtered, a crude amic acid–amine was recovered after removing the solvent by use of a rotary evaporator.

Proton NMR data on this crude product indicated that it contains 80% amic acid amine, 20% diamic acid, and 0.02% DAN. From this crude product, diamic acid was removed since it is not soluble in acetone, which dissolves amic acid–amine and DAN. However, DAN is more soluble in acetone than amic acid–amine. Therefore, amic acid–amine was purified by repetitious fractional precipitation after adding petroleum ether into an acetone solution of the crude product. NMR was used to confirm the purity.

(2) Diamic Acid. The white precipitate from reaction 1 in Scheme I was found to be 97% diamic acid and 3% amic acid–amine, judging by proton NMR. It was purified by dissolving 0.3 g in 10 mL of dimethyl sulfoxide by using an ultrasonic bath to ensure dissolution, followed by precipitation of diamic acid in acetone, since amic acid–amine is soluble in acetone. The fluffy precipitate from acetone was washed six times with a large excess of fresh acetone to remove any amic acid–amine. The purity of diamic acid was confirmed by proton NMR.

(3) Amine–Imide. Amine–imide was synthesized by following reaction 2 in Scheme I, via the route of thermal imidization of amic acid–amine. A solution of 0.53 g of crude amic acid–amine dissolved in 8 mL of NMP in a 25-mL flask was heated at 170 °C for 24 h with the drying tube and under dry argon gas purge. The imidized product was precipitated by adding 50 mL of distilled water. Proton NMR of this precipitate showed it contained ~23% amine–imide and 77% diimide.

Since the amine–imide is soluble in acetone while the diimide is not, amine–imide was extracted several times with acetone. After the acetone solution was concentrated, it was added to petroleum ether to precipitate amine–imide. Proton NMR confirmed the precipitate to be pure amine–imide.

(4) Amic Acid–Imide. The synthesis of amic acid–imide followed reaction 3 in Scheme I, by reacting amine–imide with phthalic anhydride. About 30 mg (0.1 mmol) of pure amine–imide in 3 mL of acetone was reacted with 50 mg (0.34 mmol) of phthalic anhydride at room temperature for 28 h. A white precipitate which was formed at the bottom of the vial was rinsed several times with acetone before air and vacuum drying. Proton NMR showed this solid to be amic acid–imide.

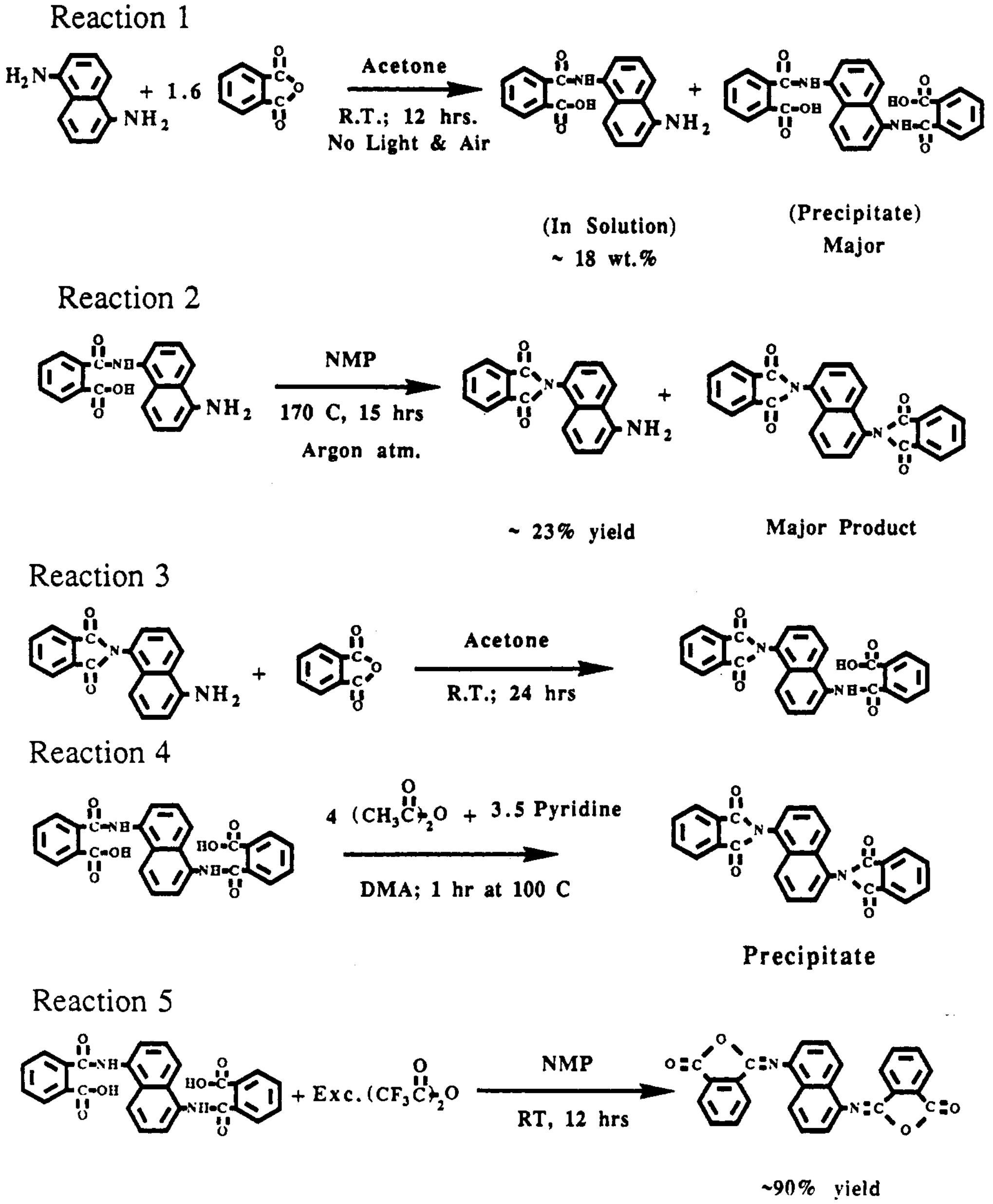
(5) Diimide. The synthesis of diimide was carried out by chemical imidization of diamic acid, as indicated by reaction 4 in Scheme I, according to the general method reported by Vinogradova et al.[13a] A NMP solution containing 0.965 g (0.12 mmol) of diamic acid, 1.6 mL (17 mmol) of acetic anhydride, and 1.2 mL (14.9 mmol) of pyridine was reacted at 100 °C for 2 h with stirring. During the course of reaction, the reaction mixture first became yellow white before the formation of a precipitate and the disappearance of the yellow color. The yellow color is due to formation of isoimide, which is an intermediate. The white precipitate after acetone washing and vacuum drying was found to be pure diimide, as judged by proton NMR.

(6) Diisoimide. Diisoimide was made according to the method reported by Kudryavstev et al.,[13b] as illustrated in reaction 5 in Scheme I. Trifluoroacetic anhydride (1.0 mL, 7.08 mmol) was added to the diamic acid (0.1128 g, 0.248 mmol) dispersed in 3 mL of NMP and allowed to react at room temperature for 48 h. The reaction mixture turned yellow immediately after adding the trifluoroacetic anhydride. The solid diisoimide was extracted seven times with 14 mL of anhydrous diethyl ether each, before vacuum drying. After drying under dry argon and vacuum drying, diisoimide was obtained. This compound was only slightly soluble in NMP, and no convenient solvent was found to run a NMR. FTIR of this solid in KBr pellet showed that it contains absorption bands due to the isoimide unit but none from the amic acid or from the imide unit.

Synthesis of Poly(amic acid). A NMP solution of poly(amic acid) (4 wt %) was made by reacting 0.5156 g (3.26 mmol) of DAN and 1.4478 g (3.26 mmol) of 6FDA in 50 mL of NMR overnight at room temperature. The poly(amic acid) was then stored in tightly closed 20-mL vials at 0 °C until used.

Chemical Imidization Procedure. Reactions were carried out in a three-neck flask by adding 10.0 mL of NMP solution

Scheme I
Synthetic Routes Used for Model Compounds



containing 1.60 mL of acetic anhydride and 1.20 mL of pyridine to a poly(amic acid) solution containing 20.0 ml of 4 wt % poly(amic acid) solution and 50.0 mL of NMP. Both solutions were thermostated at 38.0 °C before mixing. The total volume was thus 80.0 mL, and the poly(amic acid) concentration was 1 wt %. The concentrations of acetic anhydride and pyridine were 0.212 and 0.182 M, respectively. Aliquots of 4.0 μL were taken from the reaction flask at certain time intervals, diluted to 1.80 mL of NMP in a 1-cm path length cuvette, and stirred with a small stirring bar for 1 min before the UV-visible spectrum was taken.

Results and Discussion

Characterization of Model Compounds. Chart I shows the chemical structures of the model compounds from DAN and phthalic anhydride. We will describe how several characterization techniques such as proton NMR, FTIR, and differential thermal analysis were used to confirm the chemical structures before discussing UV-visible and fluorescence spectral data of these compounds.

NMR Results. This is the primary method used to identify and estimate the purity of the compounds. A summary of the proton NMR chemical shifts and their assignments are given in Table I. The positions of each type of proton as listed in Table I are expected from the chemical structure of each model compound. Furthermore, the integrated intensity of each proton type in Table I confirms the chemical structure of each model compound. For example, amic acid–amine shows a single amide proton at 10.17 ppm, an amine proton at 5.74 ppm, six naphthyl protons from 6.75 to 7.94 ppm, and four phenyl protons at 7.65 ppm. We also observe the effect of the substituent on the positions of the amine and amide protons on the naphthyl ring. As the substituent group becomes more electron withdrawing as in amic acid or imide, both amide and amine protons move downfield (higher ppm). When the two substituents on the naphthyl moiety are the same, as in DAN, diamic acid, and diimide, only three naphthyl protons are observed since both sides of the naphthyl are similar and each side contains three nonequivalent protons. However, when the two substituents are different, as in amic acid–amine, amine–imide, and amic acid–imine, all six of the naphthyl protons are nonequivalent, leading to six different naphthyl proton peaks.

FTIR Results. FTIR spectra for all of the model compounds show the presence of vibrational absorption

Table I
Proton NMR Chemical Shifts (in ppm) for Model Compounds[a]

	proton				
model compd	amide NH	amine NH_2	naphthyl	phenyl	acid OH
DAN		5.42 s	7.21 d, 7.05 t, 6.59 d		
diamic acid	10.41 s		8.13 d, 7.93 d, 7.75 m	7.70 m, 7.40 m	13.2 br
amic acid–amine	10.17 s	5.74 s	7.94 d, 7.87 d, 7.65 m, 7.37 t, 7.18 t, 6.75 d	7.65 m	
amine–imide		5.93 s	8.23 d, 7.51 p, 7.17 t, 6.85 d, 6.69 d	8.00 m, 7.98 m	
amic acid–imide	10.52 s		8.40 d, 8.37 d, 8.09 t, 8.00 m, 7.73 m	8.00 m, 7.67 m	13.2 br
diimide			7.75 d, 7.66 t, ~8.00 m	8.03 m, 7.98 m	
diisoimide			not soluble		

[a] Key: s, singlet; d, doublet; t, triplet; m, multiplet; br, broad peak. Solvent, DMSO-d_6; reference peak, DMSO-d_6 (2.49 ppm).

Table II
Assignments of Thermal Transitions (°C) for Model Compounds

	transitions				
model compd	*a*	*b*	*c*	*d*	*e*
DAN	190				
diamic acid		240			478
amic acid–amine		127	246		
amine–imide	252				
amic acid–imide		244			477
diisoimide				341	471
diimide	482				

[a] Melting of pure nonreacting compound. [b] Melting with amic acid conversion to imide. [c] Melting of imide–amine. [d] Melting of diisoimide with conversion to diimide. [e] Melting of diimide.

Table III
UV–Visible Absorption Maxima, Spectral Shifts, and Extinction Coefficients for Model Compounds

model compound	abs max (nm)	spectral shift (nm)	ext coeff (L/mol·cm)
DAN	340 and 350[a]	0[b]	13 600
diamic acid	318	-27	12 600
amic acid–amine	342	-3	9 000
amine–imide	341	-4	7 600
amic acid–imide	304	-41	11 700
diimide	289	-56	13 800
diisoimide	385	+40	13 200

[a] Average, 345 nm. [b] Reference, 345 mm.

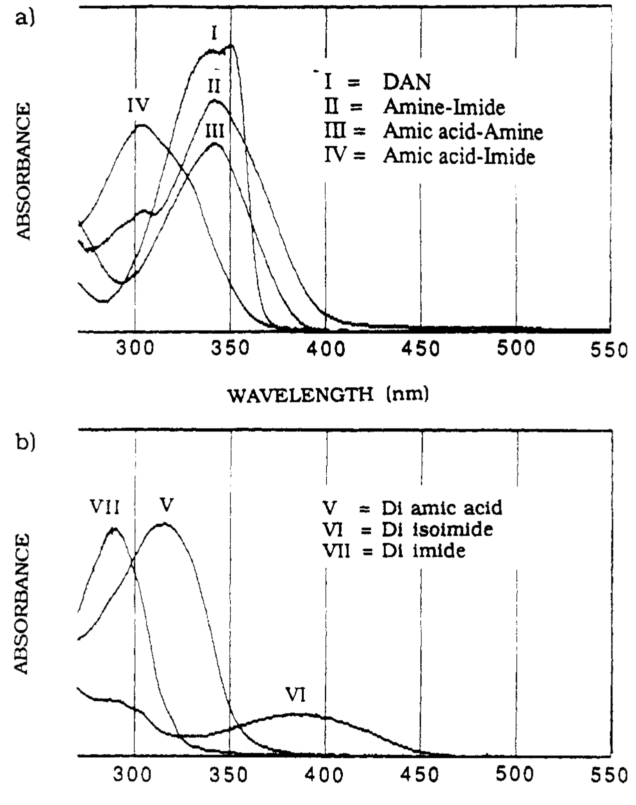


Figure 1. UV–visible spectra of model compounds in distilled NMP.

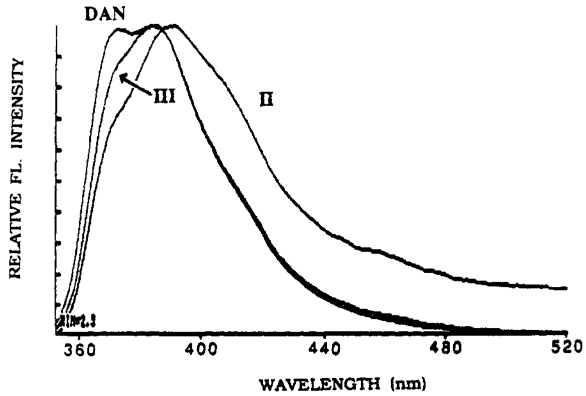


Figure 2. Fluorescence emission spectra for DAN (I), amine–imide (II), and amic acid–amine (III). Spectra plotted without regard to intensity.

peaks characteristic of the functional groups present in each model compound.

DSC Results. Table II summarizes the DSC resutls obtained for all the model compounds. As listed in Table II, each compound has its characteristic unique DSC peaks. DAN shows a melting peak at 190 °C, which is the same as the reported value.[10] The diamic acid shows a melting peak at 240 °C with simultaneous imidization to diimide with the latter showing a melting peak at 478 °C. A similar imidization of an amic acid compound was reported by Vogel.[11] The amic acid–imide shows similar melting points at 244 and 477 °C. The amic acid–amine melts at a lower temperature of 127 °C, but when the amic acid is converted to imide, the latter melts at 246 °C, which is close to the melting of pure amine–imide. Amine–imide shows a melting at 252 °C without any additional melting since it does not react further upon heating. Diimide shows only a single melting endotherm at 482 °C. Diisoimide showed a high melting endotherm at 341 °C, followed by simultaneous exothermic isomerization to the more stable diimide, which shows melting at 471 °C. This trend of isoimide is due to its thermal instability.[12]

UV–Visible Results. Figure 1 shows UV–visible absorption spectra of the model compounds. The absorption maxima and the extinction coefficients are summarized in Table III. The trends in absorption spectral shifts are as expected in view of the electronic effects of the substituents and are similar to model compounds derived from another diamine, diaminoazobenzene.[5] As the amine is converted to amic acid, generally a blue shift is observed due to the fact that the amine is more electron donating than the amic acid. As amic acid is converted to imide, an additional blue shift is observed as an amic acid–imide and diimide. In the case of amine–imide, the

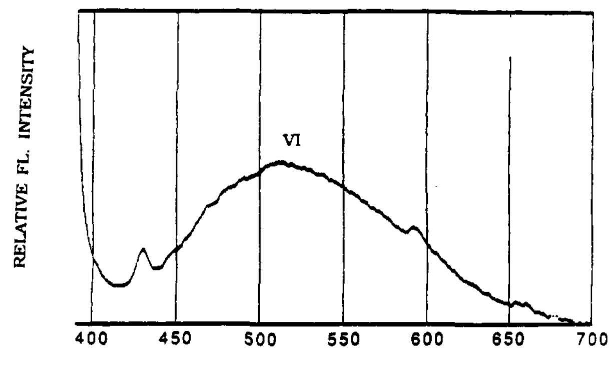


Figure 3. Fluorescence emission spectrum for diisoimide (VI) in distilled NMP. Excitation wavelength was 380 nm.

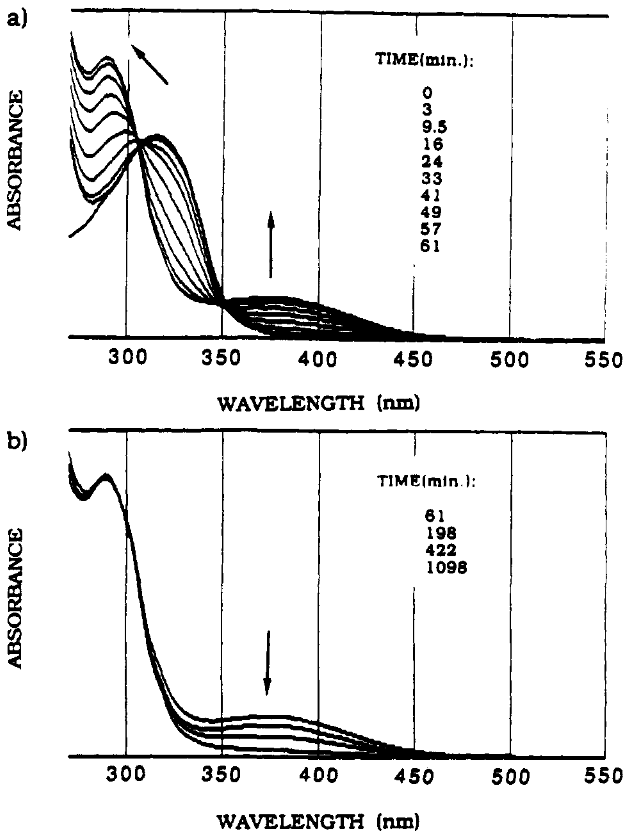


Figure 4. Changes in UV-visible spectra during chemical imidization of a poly(amic acid) in 1 wt % NMP solution, using acetic anhydride-pyridine (4:3.5 molar ratio) at 38 °C, as a function of time.

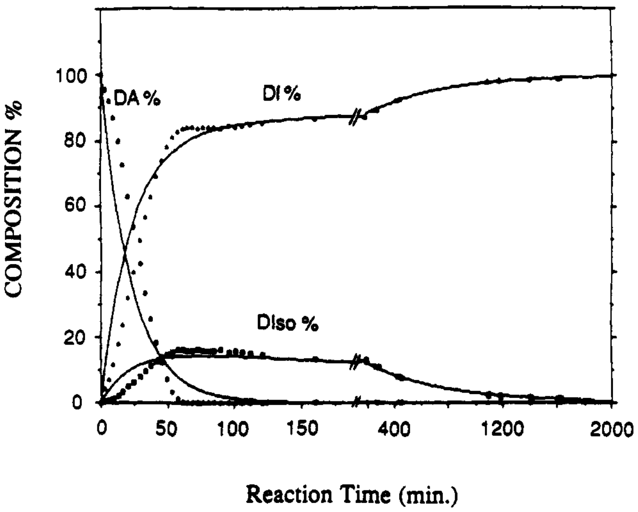


Figure 5. Composition profiles of diamic acid (DA), diimide (DI), and diisoimide (DIso) repeat units as a function of chemical imidization time. Continuous lines represent the fitting using k_1, k_2, and k_3 values of 0.037, 0.007, and 0.0018.

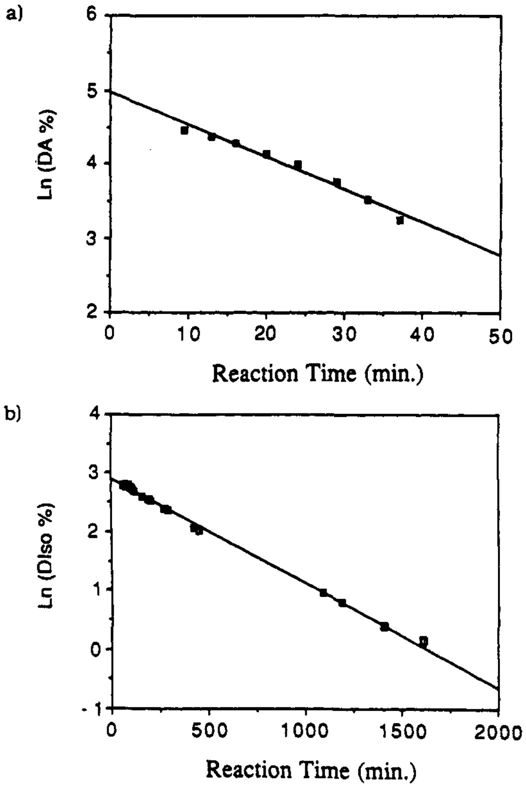


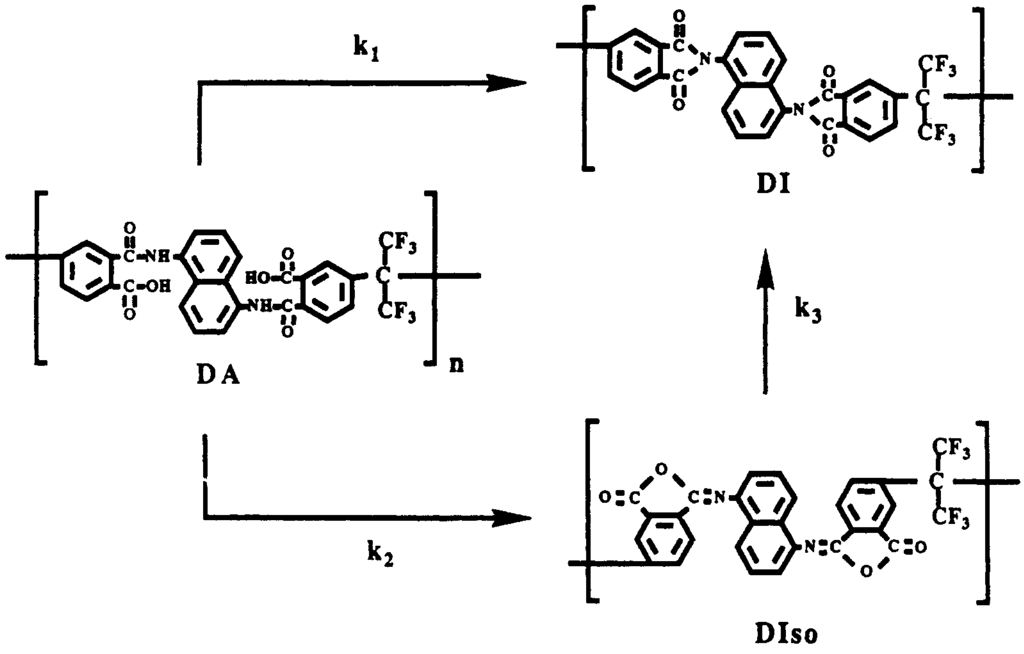
Figure 6. (a) ln [DA] vs time with least-squares fit. (b) ln [DIso] vs time (for second stage of the reaction) with least-squares fit.

absorption maximum is red shifted from amic acid-imide because of the pull-push effect of the electron-withdrawing imide and electron-donating amine. Diisoimide shows its absorption maxima at the longest wavelength (377 nm) due to the extended conjugation and the resulting planar conformation. Others have also confirmed the trend of isoimide to show its absorption at the longer wavelength.[12] As for the extinction coefficients, generally, an order of 10 000 L/mol·cm has been observed even though there are some differences among the model compounds.

Fluorescence Results. Of the seven model compounds, only DAN was found to be highly fluorescent, with the quantum yield close to 0.5. Two other amine-containing compounds, amic acid-amine and amine-imide, were found to be very weakly fluorescent. Figure 2 shows the fluorescence spectra of DAN and these amine-containing compounds. The spectra are plotted without regard to intensity to illustrate the shape and emission maxima. DAN shows a double peak as in the UV-visible spectra. Stokes shifts of ~20 nm were observed for DAN. The amic acid-amine shows an emission maximum at 383 nm, while that for the amine-imide is red shifted to 390 nm. This small red shift is in contrast to the very similar UV-visible absorption maxima for all three compounds. Diisoimide also shows a very weak fluorescence with an emission maximum near 515 nm, when excited at its absorption maximum, as shown in Figure 3.

Chemical Imidization Studies. Studies of chemical imidization have been carried out on 1 wt % poly(amic acid) in NMP solutions made from DAN and 6FDA at 38 °C using the dehydrating agent of acetic anhydride-pyridine with a 4:3.5 molar ratio. This ratio of acetic anhydride to pyridine was found to result in the highest molecular weight polyimide, as measured by viscosity by

Scheme II
Chemical Imidization Scheme



Vinogradova et al.[13a] Figure 4 shows a series of UV–visible spectra as a function of reaction time in NMP. These spectra were obtained after 4-μL aliquots were diluted in 1.8 mL of NMP. As seen in Figure 4a, which represents the early reaction times, the UV–visible peak at 318 nm due to poly(amic acid) is simultaneously shifted to the diimide peak at 289 nm and the diisoimide peak at 377 nm. This trend suggests that poly(amic acid) is simultaneously converted to a copolymer containing the diimide and diisoimide units at early reaction times. Upon further reaction, the UV–visible peak for diisoimide repeat unit decreases while the UV–visible peak for diimide increases. On the basis of these spectral trends, we can use Scheme II to represent chemical imidization, which involves simultaneous conversion of poly(amic acid) (DA) to a poly(diimide-*co*-diisoimide), followed by slower conversion of diisoimide (DIso) units to diimide (DI), resulting in polyimide. This scheme does not assume reversible conversion between diisoimide and diimide units for the second stage of chemical imidization, which is the scheme used by Koton et al. to analyze chemical imidization for films of poly[4,4′-(oxydiphenylene)pyromellitamic acid] by IR analysis.[14] We tested their equilibrium hypothesis for our system by monitoring the UV–visible spectra for the diimide model compound under the same reaction conditions used for chemical imidization. No diisoimide was formed when chemical imidization was started from diimide, justifying the use of Scheme II.

In order to obtain the rate constants k_1, k_2, and k_3 as defined in Scheme II, we need to obtain the composition of three species experimentally as a function of reaction time. This was achieved by the deconvolution of UV–visible spectra based on the spectra of three involved species as reported in Figure 1b. Figure 5 gives the changes in composition from spectral deconvolution as a function of reaction time. The first stage is complete in 61 min when poly(amic acid) decreases to zero concentration with the consequent increase of diimide units up to 84% and diisoimide up to 16%. In the second stage, which is a much slower process than the first stage, diisoimide is converted to diimide over a period of ~4000 min. Small amounts of diisoimide was still present after ~10 000 min, as evidenced by the yellowish color in the solution.

Since the two stages are quite different in time scale, we can treat the reactions as two separate processes. Thus, the first process is a parallel reaction with only k_1 and k_2 involved. We obtained $(k_1 + k_2)$ values of 0.044 min^{-1} from the slope of ln [diamic acid] vs time, as illustrated in Figure 6a. In parallel reactions, two products (diimide and diisoimide) are produced in relative yields corresponding to the specific reaction rate. Therefore, the ratio of k_1/k_2 will be equal to the ratio of the concentrations of diimide/diisoimide during the first stage of the reaction.[15] From the ratio of k_1/k_2 which was determined to be 5.11, the values of k_1 and k_2 are estimated to be 0.037 and 0.007 min^{-1}, respectively.

The second-stage reaction can be treated as a first-order conversion of diisoimide units to diimide with a rate constant of k_3. The plot of ln [diisoimide] vs time gave a straight line with a slope of 0.0018 min^{-1}, as shown in Figure 6b. Using these values of k_1, k_2, and k_3, the predicted composition profiles were generated as solid lines in Figure 5. The equations used for the first stage are as follows:

$$[\mathrm{DA}] = e^{-(k_1+k_2)t}$$

$$[\mathrm{DIso}] = -\frac{k_2}{k_1 + k_2} e^{-(k_1+k_2)t}$$

$$[\mathrm{DI}] = -\frac{k_1}{k_1 + k_2} e^{-(k_1+k_2)t}$$

The equation used for the second stage is as follows:

$$-\ln\,[\mathrm{DIso}] = \ln\,[\mathrm{DI}] = k_3 t$$

In these equations, all the concentrations of DA, DIso, and DI represent fractional concentration. There is a good fit between experimental data and simulated data. These rate constants have trends similar to those reported by Koton et al. for other chemical imidization processes.[12]

Summary

By use of 1,5-diaminonaphthalene as a model diamine and phthalic anhydride as an anhydride, several model imide compounds were synthesized, purified, and characterized in this study. These model compounds include amic acid–amine, diamic acid, amine–imide, amic acid–imide, diimide, and diisoimide. They are characterized by UV–visible fluorescence, NMR, FTIR, and thermal analysis. Only DAN is found to be strongly fluorescent while amic acid–amine, amine–imide, and diisoimide are only very weakly fluorescent. Other compounds have only negligible fluorescence. Due to strong substituent effects on the UV–visible spectra, there is generally a blue shift when amine is converted to amic acid and to imide. For diisoimide, there is a strong red shift, which distinguishes it clearly from all the other species. The spectral changes occurring during chemical imidization of poly(amic acid) made from DAN and a partially fluorinated dianhydride (6FDA) were investigated by using the UV–visible spectra of the model imide species. The composition of diamic acid, diimide, or diisoimide was determined by spectral deconvolution. The analysis of the kinetics of chemical imidization indicates simultaneous conversion of diamic acid to diimide and diisoimide, followed by slower conversion of diisoimide to diimide. During the initial reaction stage, the conversion to diimide is faster than that for diisoimide.

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