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Synthesis and Characterization of Poly(amide-imide-imide)s Containing 1,1-Bis(4-amino 3,5-dimethyl)-1-(4-methoxy phenyl) Methane

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A series of new organosoluble poly(amide-imide-imide)s were synthesized by direct polycondensation of tetrimer dicarboxylic acid containing 1,1-bis (4-amino 3,5-dimethyl)-1-(4-methoxy phenyl) methane and various aromatic diamines using pyridine and TPP as condensing agents. The structure of the prepared tetrimer dicarboxylic acids and the poly(amide-imide-imide)s were confirmed using elemental analysis, FTIR and proton NMR. All the polymers were readily soluble in a variety of organic solvents including NMP, DMAc, DMF etc., The thin films cast from DMAc exhibited tensile strengths of 96–101 Mpa, elongation at break at 9–14% and initial moduli of 1.9–2.3 Gpa. The glass transition temperature of the polymers was found to be in the range of 282–332°C. They had 10% weight losses at temperatures beyond 530°C and left more than 50% char yield even at 800°C in nitrogen atmosphere.

Keywords poly amide-imides, FT-IR, thermal properties, UV visible spectroscopy

Introduction

Aromatic polyimides are well known as high performance polymer materials for their excellent thermal stabilities and balanced mechanical and electrical properties (1–5). However, these polymers are normally insoluble and inflexible in their fully imidized form. An approach to avoid this processing problem of polyimides is to make a poly(amide-imide). Such a structure allows the complete development of the imide function in the monomer (or) reactive oligomer followed by polymerization via amide formation. Furthermore, the inclusion of an amide group into the polyimide backbone increases its processibility, solubility and moldability. However, the thermal stability of the copolymer is typically intermediate between the thermal stabilities of the two homopolymers.

Polyamide-imides are usually prepared by low temperature polycondensation of an aromatic diamine with the acyl chloride of trimellitic anhydride (6–8) or with acyl chloride of aromatic diacids containing preformed imide rings (9, 10).

The direct polycondensation route, in which pyridine and TPP are used as condensing agent, avoids the usage of moisture-sensitive acid chlorides and provides significant

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advantages in manufacturing operations compared with conventional methods. In addition, this synthetic route to polyamide-imides can offer the option of introducing specific functionality between amide or imide groups. Several attempts have been made to modify the polyamide-imides structure by introducing different functional groups (or) substituents capable of reducing the chain rigidity and further increasing their tractability (11–15).

In this article, we report the novel soluble poly(amide-imide-imide)s based on the new diacid bearing bulky pendent group and methyl substituted phenylene units. It was expected that the presence of bulky methoxy group in the polymer backbone may reduce the crystallinity and enhance the solubility and melt moldability of the polymers. In addition, the methyl substitution on phenylene may also effectively enhance the solubility without affecting the thermo oxidative stability of polymers.

Experimental

Materials

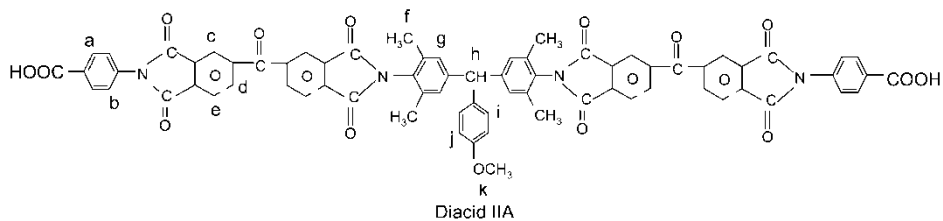
p-amino benzoic acid (p-ABA, SRL) and m-Phenylene diamine (SRL) were vacuum distilled before use. Other diamines, 2,6-Pyridine diamine, 4,4'-oxydianiline, p-Phenylene diamine, 4,4'-diamino 3,3'-dichloro Diphenyl methane (E. Merck) were used as received. BTDA (3,3', 4,4'-benzophenone tetra carboxylic acid dianhydride) and PMDA (Pyromellitic dianhydride) were recrystallized from acetic anhydride before use. N-methyl 2 pyrrolidone (NMP, Spectrochem) and Pyridine (Spectrochem) were purified by distillation under reduced pressure and stored over 4 Å molecular sieves. Triphenyl phosphite (TPP, E. Merck) was used as received. Anhydrous calcium chloride was dried under reduced pressure at 150°C for 6 h prior to use.

Monomer Synthesis

Diamine Synthesis. The diamine containing methoxy pendant group and tetramethyl substituent was prepared by the optimized procedure previously reported (16, 17). The prepared diamine was recrystallized using ethanol.

Synthesis of Imide Containing Dicarboxylic Acid. A three-necked 150 ml RB flask equipped with nitrogen inlet and reflux condenser was charged with a solution of 2.880 g (8 mmol) of diamine 1,1-bis(4-amino 3,5-dimethyl)-1-(4-methoxy phenyl) methane and 2.192 g (16 mmol) of p-ABA in 20 mL of NMP. To this, 3.488 g (16 mmol) of BTDA was added in one portion. The mixture was stirred at room temperature for 2 h. About 25 mL of toluene was then added and the mixture was refluxed for about 3 h. The water formed in the reaction was distilled off azeotropically via a Dean-stark trap. At the end of the reaction, the residual toluene was distilled off under reduced pressure. After cooling, the obtained solution was trickled into water and the precipitated product was collected by filtration and dried in vacuum at 100°C for 12 h to get diacid II. Melting point-34°C (by DSC) IR (KBr, cm^{-1}); 3447 (acid -OH), 1778 (imide, symmetric C=O stretching), 1721 (acid C=O stretching and asymmetric imide C=O stretching), 1371 (imide-imide ring vibration, axial) 2925 (-CH stretching of -CH₃ group), 1109 (imide-imide ring vibration, transverse) and 726 cm^{-1} imide ring deformation. ¹H-NMR (400 MHz, CDCl₃) 8.19δ (H_a), 8.15δ, 8.24δ, 8.08δ (H_b, H_c, H_d), 8.22δ (H_e), 2.43δ (H_f), 7.05δ (H_g), 7.14δ, 6.9δ (H_i, H_j),

5.53 δ (H_k). The elemental analysis results also confirm the formation of diacid. The found values are very close to the calculated ones.



Polymer Synthesis

Synthesis of Poly(amide-imide-imide)s. A two-necked 100 ml RB flask equipped with reflux condenser and additional funnel was charged with a solution of 0.998 g (1 mmole) of diacid and 0.108 g (1 mmole) of *p*-phenylene diamine in 5 ml of NMP. About 0.30 g of anhydrous calcium chloride, 0.6 ml of TPP and 1 ml of pyridine were added to this and refluxed for 3 h. As the reaction proceeds, the viscosity of the reaction increases and an additional 3 ml NMP was added. The obtained viscous solution was trickled into excess methanol with vigorous stirring. The polymer thus obtained was filtered, washed with methanol, and dried under vacuum at 100°C for 12 h.

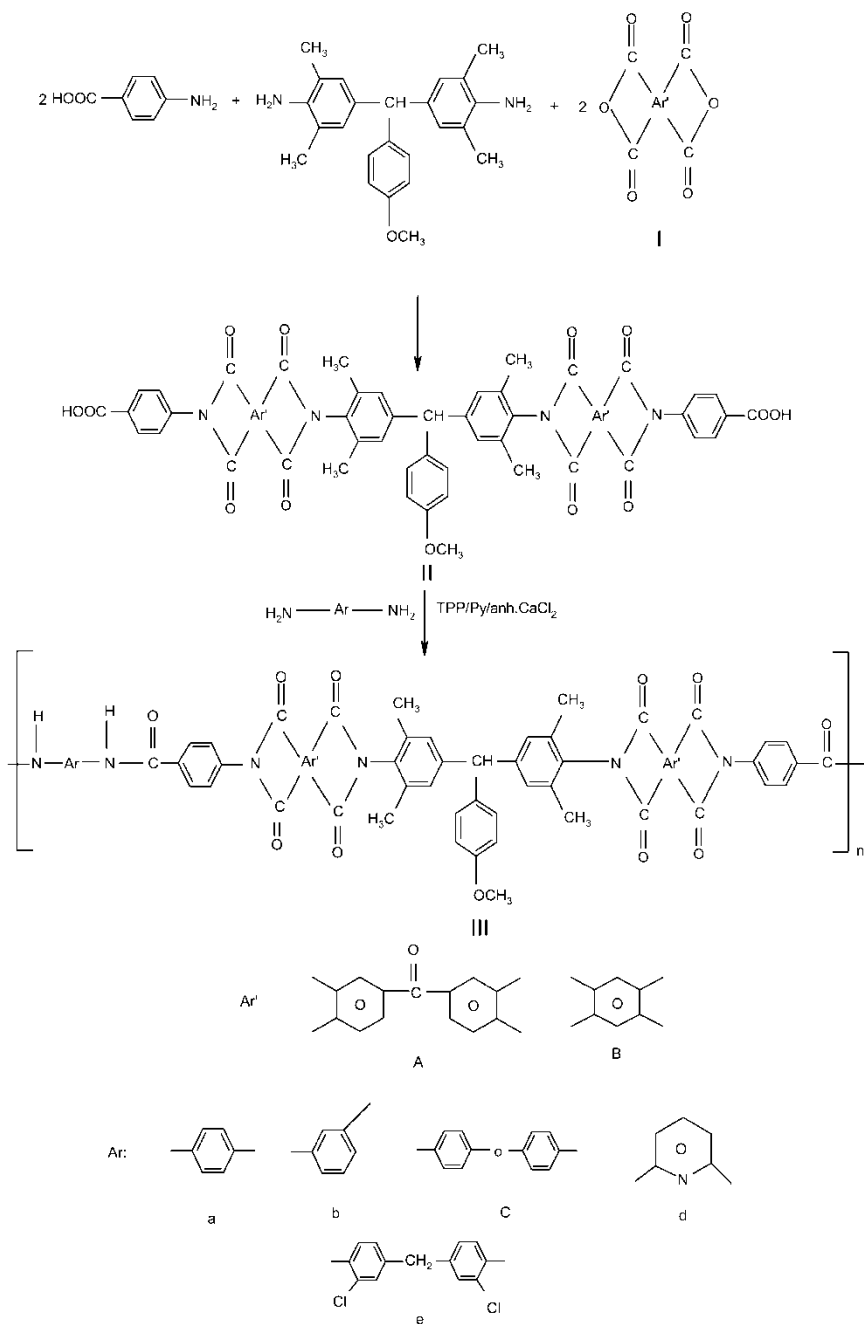
Preparation of the Poly(amide-imide-imide) Films. The polymer solution (10%) was made by dissolving the polymer in DMAc. The solution was poured into a glass culture dish and placed in an oven at 90°C overnight for the removal of solvent. Then, the obtained semi-dried polymer film was stripped out from the dish and dried in vacuum oven at 160°C for 12 h.

Measurements

Infrared spectra were recorded on a Nicolet Fourier transform infrared spectrometer. ¹H-NMR spectra were obtained on a Jeol Ex-400 spectrometer. Elemental analysis were carried out with a Perkin-Elmer model 2400. The inherent viscosities of all polymers were measured at 0.5 g dL⁻¹ in NMP using Ubbelohde viscometer at 30°C. Thermogravimetric data were obtained on a Dupont 2100 in flowing nitrogen at a heating rate of 20°C/min. Differential scanning calorimetry (DSC) analysis was performed on a Dupont 2100 Differential scanning calorimeter. Mechanical properties of the films were measured with an Instron model 1130 tensile tester with 5 Kg load cell at a crosshead speed of 5 cm/min on strips approximately 30–40 μm thick and 0.5 cm wide with a 2 cm gauge length. Dielectric properties were studied on polymer films by the parallel plate capacitor method by using HP-4194A Impedance/Gain phase analyzer at a frequency of 10³ Hz.

Results and Discussion

Tetrimide-dicarboxylic acids containing bulky methoxy pendant group and methyl substituted phenylene moiety was synthesized according to the route illustrated in Scheme 1. The reaction proceeds through the ring opening addition of diamine, BTDA and *p*-ABA in 1:2:2 molar ratio at room temperature in NMP followed by intramolecular



Scheme 1.

cyclodehydration of the intermediate tetramic acid. The structure of the monomer was characterized by elemental analysis, FTIR, and $^1\text{H-NMR}$ spectroscopy.

The FTIR spectrum of diacid (Figure 1) showed absorption bands appearing at 3447 cm^{-1} for acid $-\text{OH}$ stretching vibration, 1778 cm^{-1} and 1721 cm^{-1} for

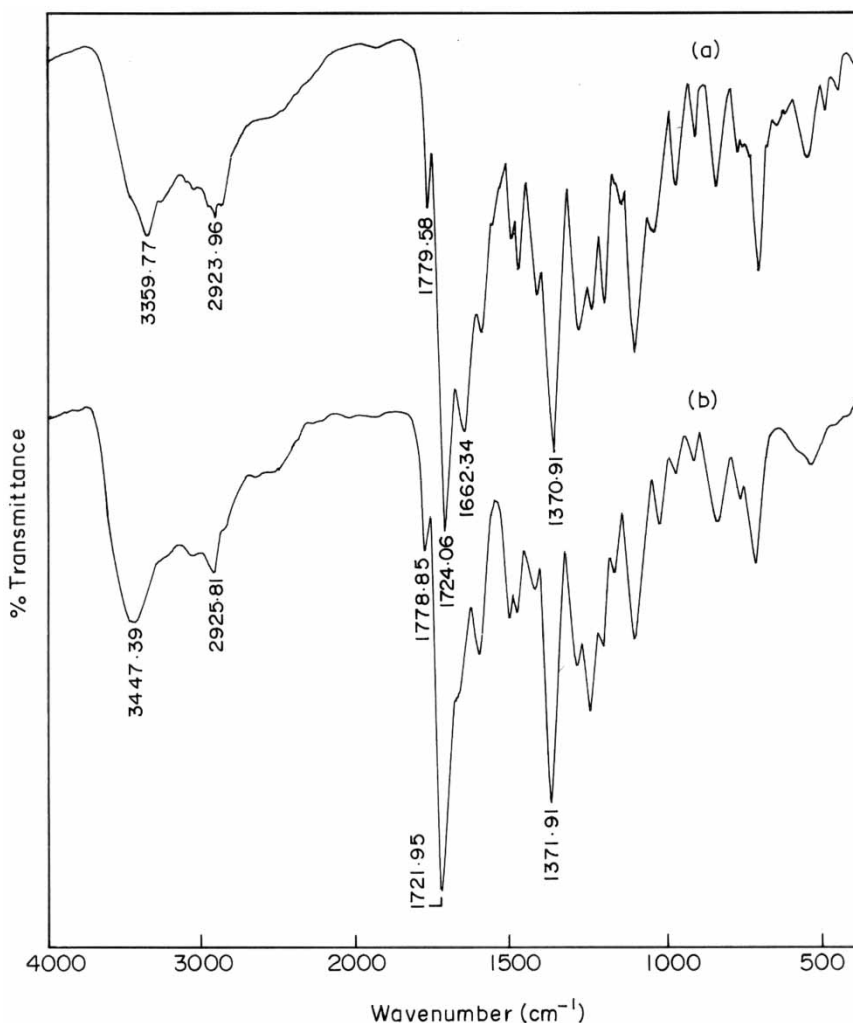


Figure 1. FTIR spectra of (a) polyamideimide, (b) tetrimidediacid.

symmetric and asymmetric C=O stretching of the imide carbonyl group, 1371 cm^{-1} for imide ring axial vibration. The absorption at 2925 cm^{-1} corresponds to C–H stretching of CH_3 and the band at 726 cm^{-1} is for out of plane imide ring vibration. The diacid (IIB) was prepared using a similar procedure also shows similar absorption patterns.

The structure of diacid (IIA) was also confirmed by $^1\text{H-NMR}$ spectrum (Figure 2). The protons H_a and H_e adjacent to the acid carbonyl and the imide carbonyl resonate at down field region at a δ value of 8.19 and 8.22δ due to the inductive effect and resonance (18). H_c proton resonates at the farthest down field due to the resonance effect of both the imide carbonyl, as well as the anhydride carbonyl group. H_b and H_d protons, which are meta oriented to acid carbonyl group and imide carbonyl group resonate at up field region due to the shielding effect. The H_f , H_h and H_k protons resonate at a lower delta value. However, the protons for acid group of diacid were not observed clearly, which may be due to the fast exchange of proton with a trace amount of moisture associated with the solvent.

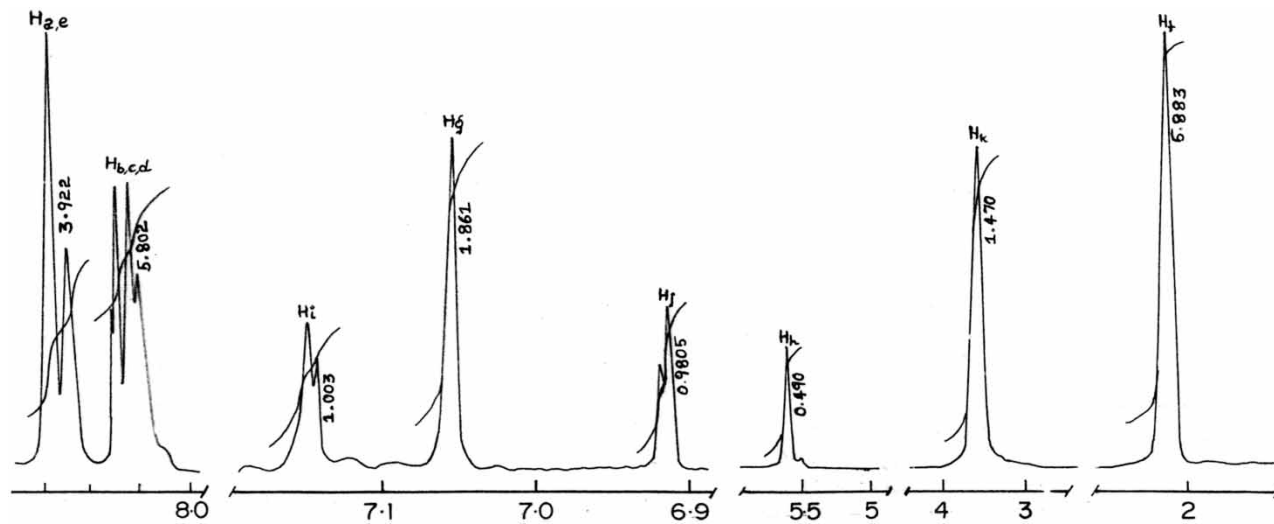


Figure 2. ¹H-NMR spectrum of diacid-IIA.

Polymer Synthesis

A series of PAIs was synthesized by a phosphorylation reaction using TPP as a promoter in NMP in the presence of pyridine and calcium chloride. The viscosity of reaction solution increased after about one hour and an additional volume of NMP was added for smooth stirring. The formation of poly (amide-imide-imide) was confirmed by IR and elemental analysis.

The FTIR spectrum of the polymer (Figure 1) displayed characteristic absorption bands for the imide ring at 1779 cm^{-1} and 1724 cm^{-1} because of asymmetric and symmetric C=O stretching vibration and at 1111 cm^{-1} and 714 cm^{-1} because of imide ring deformation. The absorption of -CH stretching of -CH₃ was observed at 2923 cm^{-1} . The amide groups showed characteristic absorption at 3359 cm^{-1} , 1662 cm^{-1} , and 1605 cm^{-1} . The results of elemental analysis of all the poly(amide-imide-imide)s are listed in Table 1. The results correlated sufficiently with the proposed structure.

Inherent viscosities of the prepared PAIs were obtained using a Ubbelohde viscometer at 0.5 g/dL concentration in NMP at 30°C, which is the most commonly used technique for obtaining an indication of the molecular weight of polymers. The inherent viscosities of the PAIs are in the range 0.69–1.09 dL/g indicating the formation of high molecular weight polymers. This is because formation of poly amide-imides from diacids and aromatic diamine is promoted by TPP in NMP at 100°C and, the addition of inorganic salts (CaCl₂) determine an increase in the chain lengths of the copolymer probably due to an improved solubility of the copolymer in the reaction medium. However, the

Table 1
Elemental analysis of poly(amide-imide-imide)s

Polymer	Molecular formula and weight	Carbon (%)	Hydrogen (%)	Nitrogen (%)
IIIA-a	$(\text{C}_{78}\text{H}_{51}\text{N}_7\text{O}_{13})_n$	C = 72.39	C = 3.97	C = 7.58
		F = 71.06	F = 4.02	F = 7.24
IIIA-b	$(\text{C}_{78}\text{H}_{51}\text{N}_7\text{O}_{13})_n$	C = 72.39	C = 3.97	C = 7.58
		F = 70.76	F = 4.13	F = 7.39
IIIA-c	$(\text{C}_{84}\text{H}_{55}\text{N}_7\text{O}_{14})_n$	C = 72.78	C = 4.00	C = 7.07
		F = 71.20	F = 4.07	F = 6.89
IIIA-d	$(\text{C}_{77}\text{H}_{50}\text{N}_7\text{O}_{13})_n$	C = 72.19	C = 3.93	C = 7.65
		F = 71.13	F = 4.05	F = 7.59
IIIA-e	$(\text{C}_{85}\text{H}_{55}\text{N}_7\text{O}_{13}\text{Cl}_2)_n$	C = 70.30	C = 3.82	C = 6.75
		F = 68.96	F = 3.88	F = 6.56
IIIB-a	$(\text{C}_{64}\text{H}_{43}\text{N}_7\text{O}_{11})_n$	C = 70.78	C = 3.99	C = 9.03
		F = 71.32	F = 4.11	F = 8.92
IIIB-b	$(\text{C}_{64}\text{H}_{43}\text{N}_7\text{O}_{11})_n$	C = 70.78	C = 3.99	C = 9.03
		F = 69.15	F = 4.15	F = 8.84
IIIB-c	$(\text{C}_{70}\text{H}_{47}\text{N}_7\text{O}_{12})_n$	C = 71.37	C = 4.02	C = 8.32
		F = 69.79	F = 4.09	F = 8.14
IIIB-d	$(\text{C}_{63}\text{H}_{42}\text{N}_8\text{O}_{11})_n$	C = 69.61	C = 3.89	C = 10.3
		F = 69.43	F = 4.12	F = 8.99
IIIB-e	$(\text{C}_{65}\text{H}_{47}\text{N}_7\text{O}_{11}\text{Cl}_2)_n$	C = 66.56	C = 4.04	C = 8.36
		F = 65.22	F = 4.10	F = 8.17

PMDA series polymers have high viscosity than the BTDA series polymers which is due to the presence of highly rigid PMDA moiety in the polymer backbone, which leads to increased chain-chain interaction or in other words increased close packing of the polymer chains. Because of the high molecular weight, most of the poly(amide-imide)s could be solution cast into transparent and tough films.

Polymer Solubility

The solubility of PAIs was tested qualitatively in various solvents. Table 2 summarizes the solubility results of these PAIs. All the PAIs were easily soluble at room temperature in aprotic polar solvents such as NMP, DMAc, DMSO, as well as less polar solvents such as m-Cresol and CHCl_3 . The improved solubility of these PAIs compared with other related PAIs was apparently due to the presence of bulky pendant group and tetra methyl substituted phenylene unit. Due to these units, the packing of macromolecular chains in tight structures through hydrogen bonding between amide groups is probably disturbed and, consequently, the solvent molecules can penetrate easily to solubilize the chains. Furthermore, the teramethyl substituent on the phenylene unit makes the polymer less polar in nature and thus, effectively enhances the solubility of the polymer. However, the solubilities of the polymers slightly vary with the structure of diamines and dianhydrides used. Polymers IIIB-a, IIIB-b and IIIB-d are soluble on standing due to the presence of PMDA unit and rigid diamines like p-phenylene diamine, m-phenylene diamine and 2,6-diamino pyridine.

UV-Visible Spectroscopy

All the prepared PAIs are lightly colored and they were also subjected to UV-Visible spectrum. From the spectral results, it has been confirmed that the presence of alkyl groups in the polymer backbone has eliminated the electronic polarization by breaking up the extended conjugation along the chain, which results in color. Apart from this,

Table 2
Solubility of poly (amide-imide-imide)s

Polymer	DMSO	NMP	DMF	CHCl_3	THF	m-Cresol	DMAc
IIIA-a	+h	+ +	+ +	±	—	+h	+ +
IIIA-b	+ +	+ +	+ +	+h	—	+h	+ +
IIIA-c	+ +	+ +	+ +	+h	+h	+h	+ +
IIIA-d	+h	+ +	+ +	±	—	±	+ +
IIIA-e	+ +	+ +	+ +	+h	+h	+ +	+ +
IIIB-a	+h	+ +	+ +	—	—	±	+ +
IIIB-b	+ +	+ +	+ +	±	—	±	+ +
IIIB-c	+ +	+ +	+ +	+h	±	+h	+ +
IIIB-d	+h	+ +	+ +	—	—	±	+ +
IIIB-e	+ +	+ +	+ +	+h	±	+ +	+ +

+ +: Completely soluble.

±: Sparingly soluble.

—: Insoluble.

+h: Soluble in hot conditions.

the incorporation of a bulky methoxy unit has also inhibited the chain-chain packing via the increased entropy mechanism. However, the small variations observed in the cut-off wavelength may be due to the nature of diamine and dianhydride moieties present in the polymer backbone. Polymers IIIB-a to IIIB-e are more colored than that of other polymers due to the increase of CTC formation by PMDA. The polymers with *m*-Phenylene moiety and ether linkages in the polymer chain are less colored because of the decrease in extensive conjugation. The cut off wavelength of polymer IIIB-d has been shifted from 375 nm to 405 nm when the dianhydride moiety was changed from BTDA to PMDA, which is because of the combination of highly electron rich diamine with PMDA (Figure 3).

Moisture Absorption and Dielectric Properties

The moisture uptakes of the prepared polyamide-imides were in the range of 1.68 to 2.28%. These were calculated from the weight change of the vacuum-dried polymer samples after they were exposed to air at room temperature for 8–10 h.

The dielectric properties of the polymers IIIA-a to IIIA-e are in the 2.99 to 3.28 range. (Table 3) The variation in the dielectric constant values depend upon the polar groups and nature of diamine moieties present in the polymer backbone. Polymer IIIA-e has a lower dielectric constant value because of the presence of polar methylene group in diamine. The dielectric constant value of meta-linked diamine is lower than the corresponding *para* linked system. This may well be related to free volume in the polymer since the *m*-substituted systems have a higher degree of entropy. In general, with an increase in frequency, the orientation of the polar groups gradually decreases, and hence, the dielectric constant value also decreases. However, the dielectric values of the prepared PAIs are very low compared to that of commercial PAI Torlon (4.2 at 10^3 Hz).

Thermal Properties

The thermal stability of the copolymers was characterized by TGA carried out in N_2 at a heating rate of $20^\circ\text{C}/\text{min}$. The values are tabulated in Table 4. In all cases, the thermal

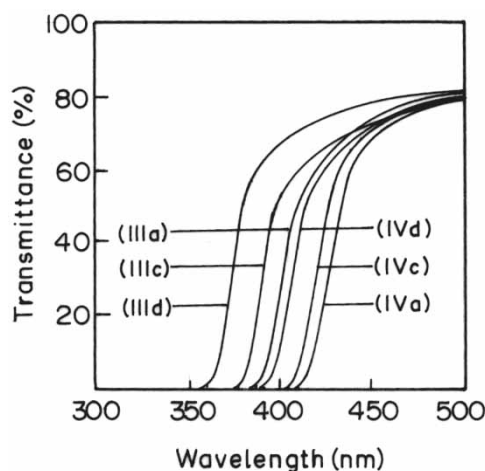


Figure 3. UV-spectrum of poly(amide-imides)

Table 3
Inherent viscosity of the poly (amide-imide-imide)s

Polymer	Inherent viscosity (η_{DL}/g)	Moisture absorption %	Dielectric constant	Film quality
IIIA-a	0.93	2.23	3.22	Flexible
IIIA-b	0.69	2.06	3.07	Flexible
IIIA-c	0.74	2.28	3.15	Flexible
IIIA-d	0.84	2.16	3.28	Flexible
IIIA-e	1.03	1.83	2.99	Flexible
IIIB-a	0.98	—	—	—
IIIB-b	0.75	1.70	—	—
IIIB-c	0.79	1.79	—	—
IIIB-d	0.86	1.76	—	—
IIIB-e	1.09	1.68	—	—

stability is very well placed between those of polyimides and polyamides. The temperature at which 10% decomposition takes place are in the range of 532–575°C which is never under 500°C showing the very high thermal stability of the prepared polymers. The polymers of PMDA series have slightly higher T_{10} values than BTDA polymers, which is due to the presence of highly rigid PMDA moiety in the polymer structure. Among all the polymers, the PAI with methylene diamine and oxydianiline have lower T_{10} values due to lower thermo oxidative stability. The results also reveal that the polymers with p-oriented phenylene have higher thermal stability than the m-oriented ones due to catenation. Anaerobic char yield was in the range 57–68% showing a high intrinsic fire resistance.

DSC measurements were conducted at a heating rate of 20°C/min in nitrogen atmosphere. The DSC traces do not show any melting endotherm due to their

Table 4
Thermal properties of poly(amide-imide-imide)s

Polymer	(T _g in °C) ^a	(T ₁₀ °C) ^b	Char. yield (%) ^c
IIIA-a	318	558	62
IIIA-b	305	546	59
IIIA-c	282	540	67
IIIA-d	330	573	61
IIIA-e	275	532	68
IIIB-a	321	560	61
IIIB-b	309	550	57
IIIB-c	290	548	66
IIIB-d	332	575	62
IIIB-e	285	545	67

^aMid point temperature base line shift on the second DSC heating trace (scan rate = 20°C/min) of the sample after quenching from 400°C.

^bTemperature at which 10% weight loss was recorded by TGA at a heating rate of 20°C/min.

^cResidual weight % at 800°C in nitrogen.

Table 5
Mechanical properties of poly(amide-imide-imide)s films

Polymer	Tensile strength MPa	Tensile elongation %	Tensile modulus GPa
IIIA-a	97	9	2.3
IIIA-b	101	10	2.3
IIIA-c	96	12	2.0
IIIA-d	98	9	2.3
IIIA-e	84	14	1.9

amorphous nature. The T_g of the polymers are in the range 282–332°C, indicating high thermal stability of prepared polymers. However, when compared to commercial polymers, the prepared polymers have slightly lower T_g, which may be due to the incorporation of a bulky pendant group which could inhibit close chain packing and increases the polymer fractional free volume that increases polymer chain rotation and flexural mobility.

Mechanical Properties

With the exception of the brittle behavior of polymers of the PMDA series, other polymers were cast into flexible and tough films from polymer solutions of DMAc. The results are summarized in Table 5. These films had a tensile strength at break of 96–101 Mpa, elongation at break of 9–14% and initial tensile moduli of 1.9–2.3 Gpa. This series of poly(amide-imide-imide)s possessed good tensile properties suggesting that these polymers can be applied as new materials for engineering plastics.

Conclusions

A new tetrimide-dicarboxylic acid monomer containing bulky pendant methoxy group and methyl substituted arylene ether unit can be prepared. These polymers showed excellent solubilities and film forming ability. Besides, these polymers were characterized by excellent mechanical properties and thermal stability demonstrating a good combination of properties and processibility.

Acknowledgment

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