



## Synthesis and Properties of New Heat-resistant Pyridine-based Polyimides Containing Preformed Amide Units

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### ABSTRACT

A new diamine monomer containing pyridine and amide units was prepared via two step reactions. Nucleophilic chloro-displacement reaction of 2,6-diaminopyridine with 4-nitrobenzoyl chloride in the presence of propylene oxide afforded 2,6-bis(4-nitrobenzamido)pyridine and subsequent reduction of the nitro groups led to 2,6-bis(4-aminobenzamido)pyridine. The diamine was reacted with aromatic dianhydrides including pyromellitic dianhydride, benzophenone tetracarboxylic dianhydride, and hexafluoroisopropylidene diphthalic anhydride to form polyimides via two-step polycondensation method, and formation of poly(amic acid) followed by chemical imidization. All the prepared materials and polyimides were fully characterized by common spectroscopic methods and elemental analysis. Physical and thermal properties of the polymers including solubility, inherent viscosity, thermal behaviour and thermal stability were studied and structure-property relations in these polyimides were investigated. The inherent viscosity of the polyimides in NMP at a concentration of 0.5 g.dL<sup>-1</sup> at 30°C was between 0.47-0.51 dL/g. The polymers showed high thermal stability while their solubilities were improved. The glass transition temperatures ( $T_g$ ) of the polymers were about 244-283°C. The 10% weight loss of polymers as a main criterion for their thermal stability was about 420-453°C. Char yield of the polymers at 600°C was in the range of 49-71%. High thermal stability resulted from incorporation of imide backbone, phenylation of chain, symmetry of structures to avoid the weak linkages. The polymers revealed good solubility in dipolar aprotic solvents such as *N*-methyl-2-pyrrolidone, *N,N*-dimethylacetamide, dimethylformamide, dimethylsulphoxide and also *m*-cresol. Improved solubility was attributed to the presence of pyridine polar unit and amide structures in the polyimide backbones.

### Key Words:

synthesis;  
polyimides;  
polycondensation;  
high performance polymers;  
thermal properties.

### INTRODUCTION

Aromatic polyimides are heat resistant polymers commonly exhibit unique physical and mechanical properties. The applications of these aromatic polymers are rising progressively due to the increased performance characteristic required on polymers in different fields including the aerospace, automobile and micro-

electronic industries [1-5].

However, one of the disadvantages to the employment of these high performance polymers is difficulty in processing due to their high melting temperature or high glass transition temperature and also their limited solubility [6].

Strong interaction between polyimide chains and their rigid

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structure are the main reasons for these behaviours. Therefore, some significant synthetic efforts in the field of heat-resistant polymers have been focused on improving their processability and solubility. In this way, various approaches have been devoted to improve the processability and solubility without sacrificing other excellent properties [7,8].

The introduction of flexible units such as alkylene, ether and sulphone units and also incorporation of bulky pendant groups such as phenyl, anisyl and naphthyl groups in the polymer backbone has been found to be effective methods to improve the solubility of polyimides. Another efficient way for improving the solubility and processability of the polyimides is copolymerization. Thus, several copolymers have been developed, such as poly(amide imide)s (PAIs), which are known to have high thermal stability and good solubility in polar amide-type solvents [9-11].

Also, with introduction of heterocyclic ring such as pyridine in the main chain of a polymer impart certain properties to the polymer. The selection of pyridine nucleus is based on two factors: first, high thermal stability derived from its molecular symmetry and aromaticity and second, improved solubility of the pyridine containing polymers (in comparison to phenyl group) because of increased dipole-dipole interaction of polymer-solvent system [12-15]. Recently, more attention has been paid to polyimides containing pyridine unit in their backbones. For example, a new series of polyimides from 2,6-diaminopyridine and different dianhydrides have been reported. It is found that incorporation of the pyridine group in the main chain increased the chemical, mechanical, and thermal stability of polymers [16]. New soluble and thermally stable poly(ester imide)s from a diimide-dicarboxylic acid pyridine-biphenylene pendant groups have been reported. These polymers have an amorphous nature and almost all polymers are soluble in both polar aprotic solvents and some less polar solvents [17]. Also, different derivatives of 4-aryl-2,6-bis-(4-amino phenyl)pyridine have been prepared and used for the preparation of related polyimides and polyamides. They showed enhanced thermal stability and high solubility [18-20]. Other pyridine containing structures such as 2,6-bis(4(-fluoro-3(-trifluoromethyl

benzyl) pyridine and 2,6-diphenyl pyridine moiety have been investigated in polyimides and similar properties are reported [21,22].

This paper describes preparation of a series of pyridine-based aromatic polyimides with preformed amide units. The main objective of this study was enhancing the solubility and maintaining the thermal stability of the polymers. This has been achieved by introduction of pyridine polar aromatic group and amide units into the backbone of aromatic polyimides. The superiority of this pyridine-based system over previously reported systems was the incorporation of amide bond in the main chain of rigid groups, whose rigidity was not far lower than that of the imide group and led to preparation of polyimides with improved processability and balanced thermal resistance.

## EXPERIMENTAL

### Materials

All chemicals were purchased either from Merck or Aldrich Chemical Co. 2,6-Diamino pyridine was recrystallized from benzene. *N*-Methyl-2-pyrrolidone (NMP), *N,N*-dimethylacetamide (DMAc), *N,N*-dimethylformamide (DMF) and toluene were purified by distillation over calcium hydride under reduced pressure. Dianhydrides were dried in a vacuum oven at 120°C for 5 h.

### Instruments

Infrared measurements were performed on a Bruker-IFS 48 FTIR spectrometer (Ettlingen, Germany). The <sup>1</sup>H NMR spectra were recorded in dimethyl sulphoxide (DMSO-d<sub>6</sub>) solution using a Bruker Avance DPX 400-MHz (GmbH, Germany). Elemental analyses were performed by CHN-O-Rapid Heraeus Elemental Analyzer (Wellesley, MA). Differential scanning calorimetry (DSC) from 30-300°C in air at a heating rate of 10°C/min, thermogravimetric analysis (TGA) from 30-600°C in air at a heating rate of 10°C/min, and differential thermogravimetric (DTG) were recorded on Stanton Redcraft STA-780 (London, UK). The dynamic mechanical measurements were performed on a polymer laboratories dynamic mechanical thermal

analyzer (DMTA) in the bending mode at 1 Hz and a heating rate of 5°C/min (Surrey, UK). Inherent viscosities were measured with an Ubbelohde viscometer.

### Monomer Synthesis

#### *Synthesis of 2,6-Bis(4-nitrobenzamido)pyridine (BNBP)*

A 250 mL two-necked round bottomed flask equipped with a magnetic stirrer, nitrogen gas inlet tube and calcium chloride drying tube was charged with 2,6-diaminopyridine (0.03 mol, 3.375 g) and NMP (90 mL). The mixture was stirred at 0°C for 0.5 h. Then, propylene oxide (about 43 mL) and 4-nitrobenzoyl chloride (0.07 mol, 13.635 g) were added and the mixture was stirred at 0°C for 30 min. The temperature was raised to room temperature and the solution was stirred for 20 h. 2,6-Bis(4-nitrobenzamido)pyridine was precipitated by pouring the flask content into water. Then, it was filtered, washed with hot water and methanol successively and dried overnight under vacuum at 50°C (yield = 88.6%).

#### *Synthesis of 2,6-Bis(4-aminobenzamido)pyridine (BABP)*

2,6-Bis(4-nitrobenzamido)pyridine (BNBP) (0.0123 mmol, 5.0 g), Pd/C 10% (0.32 g), and ethanol (63 mL) were introduced into a two-necked flask to which a mixture of hydrazine monohydrate (25 mL) and ethanol (20 mL) was added dropwise over a period of 2 h at 85°C. After the complete addition, the reaction was continued at reflux temperature for another 18 h. Then, tetrahydrofuran (25 mL) was added to the suspension to re-dissolve the precipitated product and refluxing was continued for 3 h. The mixture was filtered to remove the Pd/C and the filtrate was poured into water. The product was filtered off, washed with hot water and dried (yield = 87.6%).

#### *Polyimide Synthesis*

A two-step method was used for the preparation of polyimides as follows: a 100 mL two-necked round-bottomed flask equipped with a magnetic stirrer, a nitrogen gas inlet tube and a calcium chloride drying tube was charged with the prepared diamine (BABP) (2 mmol, 0.694 g) and dry NMP (15 mL). The mixture was stirred at 0°C for 0.5 h. Then, dianhydride

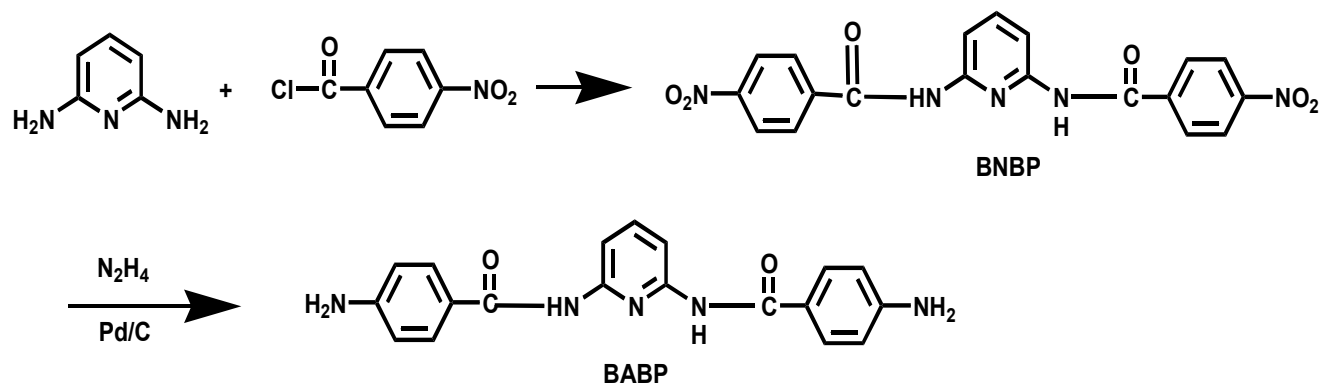
(2 mmol) was added and the mixture was stirred at 0°C for 1 h. The temperature was raised to room temperature, and the solution was stirred for 24 h. Poly(amic acid) was precipitated by pouring the flask content into 3:1 (v/v) mixture of water and methanol (200 mL). It was then filtered and dried overnight in vacuum oven at 40°C.

Chemical cyclization for the conversion of poly(amic acid) to polyimide was applied: into a 100 mL two-necked round bottomed flask equipped with a magnetic stirrer, N<sub>2</sub> gas inlet tube, and a reflux condenser were placed poly(amic acid) (1.0 g), DMAc (5 mL), acetic anhydride (5 mL) and pyridine (2.5 mL). The mixture was stirred for 0.5 h and then it was slowly heated to 140°C and held for 6 h at the same temperature. Afterwards, the mixture was cooled and poured into water. It was filtered, washed with hot water and methanol, and dried overnight in vacuum at 140°C. Yields were greater than 83.5%.

## RESULTS AND DISCUSSION

Diamines are important monomers for the preparation of variety of polymers including polyimides, polyamides and polyureas. They can confer special properties to the final polymers through their structural features [23-26]. In this way, a novel pyridine-based diamine containing amide groups was prepared in two steps. Nucleophilic substitution reaction of 2,6-diaminopyridine with 4-nitrobenzoyl chloride in the presence of propylene oxide (as an acid scavenger) led to preparation of 2,6-bis(4-nitrobenzamido)pyridine (BNBP). The related diamino compound named 2,6-bis(4-aminobenzamido)pyridine (BABP) was readily obtained in high yields by the catalytical reduction of dinitro groups of BNBP with hydrazine mono-hydrate and Pd/C catalyst in refluxing ethanol (Scheme I).

The structures of BNBP and BABP compounds were confirmed by FTIR and <sup>1</sup>H NMR spectroscopies and also elemental analysis. The FTIR spectrum of BNBP showed characteristic bands at 1522 and 1351 cm<sup>-1</sup> for NO<sub>2</sub> asymmetric and symmetric stretching, respectively. Reduction of the nitro groups and conversion to amino groups was confirmed by disappearance of these absorption bands and presence



Scheme I. Synthesis of diamine.

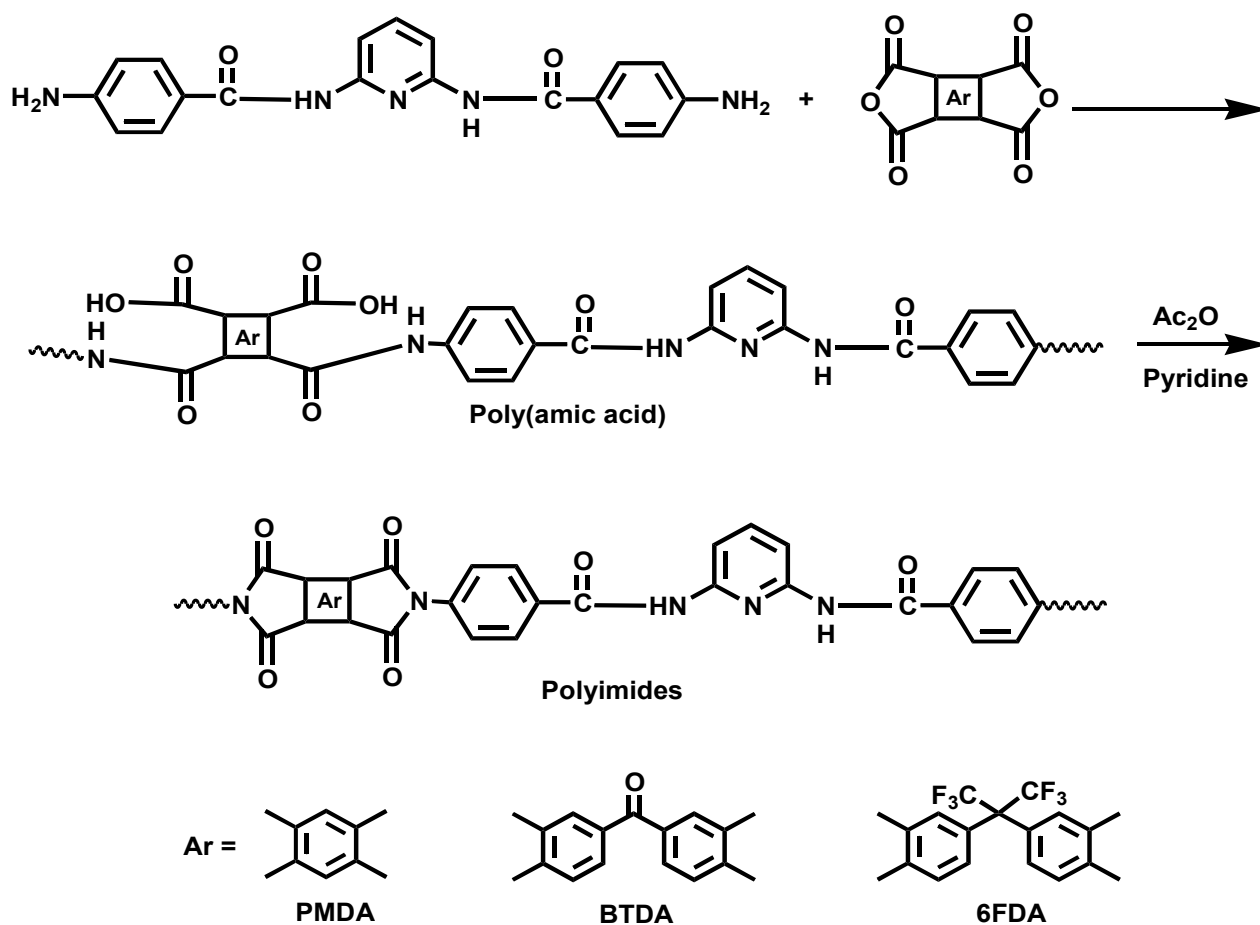
of amine bands at  $3370\text{--}3332\text{ cm}^{-1}$  (N-H stretching). Also the amide  $\text{C}=\text{O}$  stretching band at  $1694\text{ cm}^{-1}$  in BNP was converted to  $1673\text{ cm}^{-1}$  in BABP due to electron-donating character of  $\text{NH}_2$  groups. In the  $^1\text{H}$  NMR of BABP, with appearance of amine peaks at 5.80 ppm, the shift of other peaks to higher chemical fields was another criterion for amine formation. Also, the elemental analysis provided further evidence for the diamine monomer with the proposed structure. These results are collected in Table 1.

Generally, polyimides can be prepared via two different methods. One-step method consists of stirring of a dianhydride and a diamine in a high-boiling solvent at  $180\text{--}220^\circ\text{C}$ . Under these conditions chain growth and imidization occur spontaneously and this method leads to higher yields. Polyimides that are soluble in organic solvents are often prepared

by one-step method. In the two-step method of polyimide synthesis, a dianhydride is added to a solution of diamine in a polar aprotic solvent at ambient temperature. The generated poly(amic acid) is then separated and cyclodehydrated to the corresponding polyimide by extended heating at elevated temperatures or by treatment with chemical dehydrating agents. It is worth to mention that long-time maintaining of poly(amic acid) in solvents results in partial hydrolysis. Therefore, poly(amic acid) should be stored in dry condition and converted to related polyimide promptly. A two-step method always gives polyimides with lower yields in comparison to a one-step method. However, poly(amic acid) which is isolated in two-step method has higher solubility and processability than related polyimide. Since the polyimide is often insoluble and infusible, the

Table 1. Characterization of substrates.

Substrate	IR (KBr, $\text{cm}^{-1}$ )	NMR (DMSO- $d_6$ , $\delta$ , ppm)	Elemental analysis			Melting point ( $^\circ\text{C}$ )	Yield (%)			
			Calc. C	H	N			Found C	H	N
BNBP	3353-1694 1604-1586 1522-1351	10.97 s (2H, NH) 8.32 d (4H, phenyl) 8.15 d (4H, phenyl) 7.89-7.87 m (3H, pyridine)	56.02	3.19	17.20	56.18	3.10	17.28	273-275	88.6
BABP	3370-3332 3223-1673 1601-1585	9.85 s (2H, NH) 7.79-7.77 m (3H, pyridine) 7.69 d (4H, phenyl) 6.55 d (4H, phenyl) 5.80 s (4H, amine)	65.71	4.90	20.17	65.80	5.01	20.09	298-300	87.6



Scheme II. Preparation of polyimides.

polymer is usually processed in the form of poly(amic acid), which is imidized in place [27].

Accordingly, a series of polyimides (PIs) containing pyridine and amide units were synthesized by a conventional two-step method using reaction of diamine with different tetracarboxylic dianhydrides including pyromellitic dianhydride (PMDA), benzophenone tetracarboxylic dianhydride (BTDA) and hexafluoroisopropylidene diphthalic anhydride (6FDA). Ring-opening polyaddition of the diamine to dianhydride led to preparation of poly(amic acid) and subsequent chemical cyclo-dehydration resulted in preparation of polyimide as shown in Scheme II. In this process, formation of poly(amic acid) was a very important process due to the growth of molecular weight. Therefore, purity of monomers and solvents were extremely critical at this stage. Thus, polyimides were formed by chemical cyclization of the corresponding poly(amic acid)s using acetic

anhydride and pyridine. The PIs were characterized by spectroscopic methods and elemental analysis technique and the results are tabulated in Table 2.

The characteristic absorption bands of imide ring were observed at about 1785 and 1725  $\text{cm}^{-1}$  (asymmetrical and symmetrical C=O stretching vibration), 1372  $\text{cm}^{-1}$  (C-N stretching vibration) and 719  $\text{cm}^{-1}$  (imide ring deformation) in FTIR spectroscopy.

The inherent viscosity of the PIs in NMP at a concentration of 0.5  $\text{g.dL}^{-1}$  at 30°C was measured and the results are shown in Table 2. Since inherent viscosity is a good criterion for estimation of molecular weights in these polyimides, the polymers showed reasonable molecular weights.

The organosolubility behaviour of these PIs was tested qualitatively in various organic solvents. The prepared PIs were soluble in dipolar aprotic solvents such as NMP, DMAc, DMF, DMSO and also *m*-cresol due to the presence of pyridine polar unit and amide

**Table 2.** Polymer characterization.

Polymer	IR (KBr, cm <sup>-1</sup> )	NMR (DMSO-d <sub>6</sub> , $\delta$ , ppm)	Elemental analysis						Inherent viscosity (dL/g)	Yield (%)
			Calc.			Found				
			C	H	N	/	C	H		
BABP-PMDA	3400-1776 1727-1673 1589-1365 719	10.38/2H, 8.39/2H 8.17/4H, 7.94/1H 7.86/2H, 7.69/4H	65.78, 2.84, 13.23 / 65.65, 2.93, 13.10						0.51	87.9
BABP-BTDA	3383-1779 1725-1670 1601-1366 715	10.38/2H, 8.31/2H 8.28/2H, 8.18/2H 8.15/4H, 7.89/3H 7.66/4H	68.25, 3.00, 11.06 / 68.11, 2.88, 11.25						0.48	86.8
BABP-6FDA	3398-1785 1725-1681 1604-1372 719	10.66/2H, 8.20/2H 8.12/4H, 7.96/2H 7.89/3H, 7.74/2H 7.59/4H	60.40, 2.52, 9.27 / 60.29, 2.71, 9.11						0.47	83.5

**Table 3.** Thermal data of the polyimides.

Polymer	T <sub>g</sub>	T <sub>0</sub>	T <sub>10</sub>	T <sub>Max</sub>	Char yield at 600°C (%)
BABP-PMDA	283	360	453	550	71
BABP-BTDA	268	345	435	542	58
BABP-6FDA	244	318	420	539	49

T<sub>g</sub>: glass transition temperature; T<sub>0</sub>: initial decomposition temperature; T<sub>10</sub>: temperature for 10% weight loss; and T<sub>max</sub>: maximum decomposition temperature.

structures in the polyimide backbones that caused increased dipole-dipole interaction. The improved solubility of BTDA- and 6FDA-derived PIs in comparison to PMDA-derived PI was attributed to the presence of carbonyl and isopropylidene flexible groups in these PIs. However, the films cast from solutions of polymers (films were cast from NMP and heated to 300°C and held for 30 min) were hard and brittle and their mechanical properties could not be measured.

The thermal properties and behaviours of the PIs were studied by TGA, DSC and DMTA techniques. The glass transition temperatures (T<sub>g</sub>) of the polymers were about 244-283°C (Table 3) according to DSC (the midpoint of the change in slope of baseline was considered as T<sub>g</sub>) and DMTA (decreasing in storage

modulus with increasing of tan $\delta$  was considered as T<sub>g</sub>) techniques. Higher T<sub>g</sub> value of PMDA-based polyimide was attributed to more rigidity of this polymer in respect to other polyimides. Also, higher T<sub>g</sub> value of BTDA-based polyimide in comparison to 6FDA-based polyimide might be related to higher inherent viscosity (molecular weight) of this polyimide. No transition associated with softening or melting was observed for the polymers. The thermal stabilities of PIs were evaluated by thermal gravimetric analysis in air at a heating rate of 10°C/min. The polyimides started to lose weight, because of thermal degradation, around 318-360°C. The 10% weight loss of polymers as a main criterion for their thermal stability was in the range of 420-453°C indicating good thermal stability. Char yield (weight of polymer

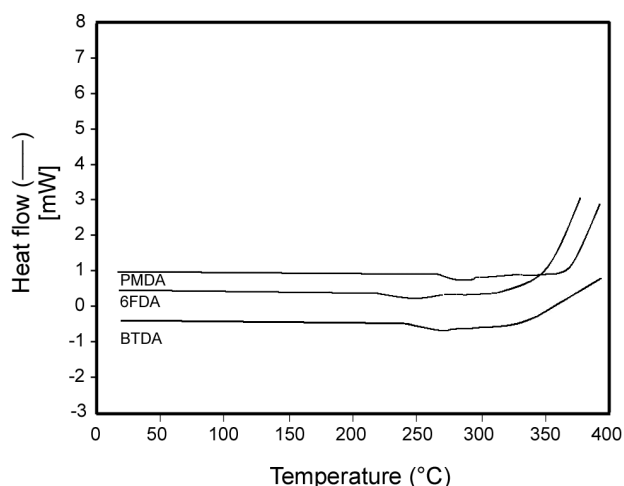


Figure 1. DSC Curves of polyimides.

remained at 600°C) was about 49-71%. Maximum decomposition temperature ( $T_{max}$ ) of the polymers that was derived from TGA curve was in the range of 539-550°C. These results are collected in Table 3 and the DSC and TGA curves of polymers are shown in Figures 1 and 2, respectively. The polymers showed high thermal stability that could be attributed to the incorporation of imide backbone, phenylation of chain, symmetry of structures to avoid weak linkages. PMDA-derived PI showed higher heat-resistant than BTDA and 6FDA derived PIs that was related to the more rigidity of the structure.

Comparison of the prepared BABP diamine and related polyimides with similar pyridine-based diamines and polyimides showed that the presence of preformed amide units in the structure of pyridine-diamine affected some properties of final polyimides. The thermal stability of BABP-based

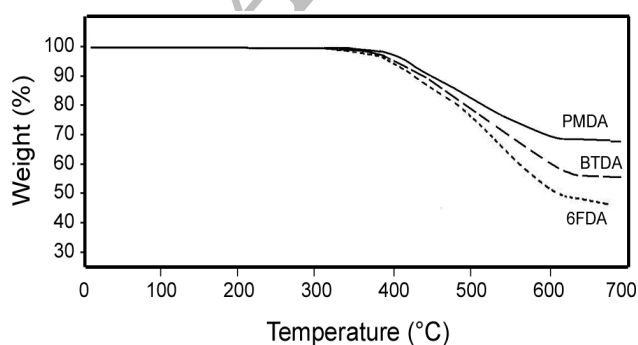


Figure 2. TGA Curves of polyimides.

polyimides was higher than, and solubility was lower than previously reported 2,6-bis(4-aminophenoxy) pyridine-based polyimides [28], while 2,6-diaminopyridine-based polyimides revealed higher thermal stability and lower solubility in respect to BABP-based polyimides [16].

## CONCLUSION

In this study a novel diamine (BABP) was successfully prepared in high purity and good yield via two steps. Nucleophilic chloro-substitution reaction of 2,6-diaminopyridine with 4-nitrobenzoyl chloride provided BNP and subsequent reduction of BNP nitro groups to amino groups led to BABP diamine. The related PIs were prepared via a two-step polycondensation reaction of BABP with different aromatic dianhydrides. Introduction of pyridine unit in addition to amide groups from one side, and avoidance of any weak linkages and presence of fully aromatic structures from the other side resulted in formation of polyimides with enhanced solubility and high heat-resistant.

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