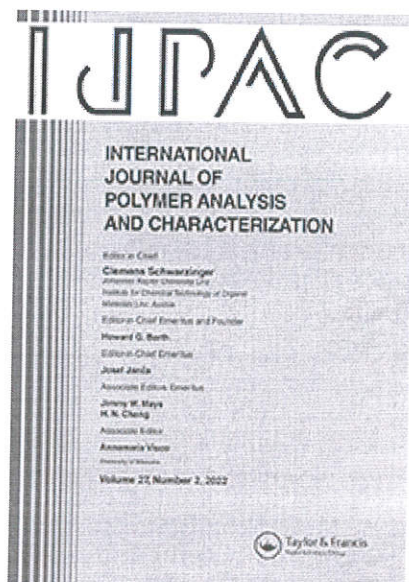


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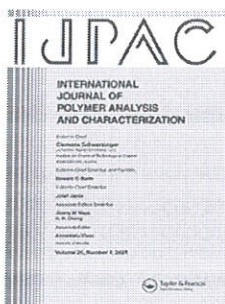
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# Synthesis and characterization of aliphatic aromatic polyamides containing pyridine and methylene spacer group in polymer backbone

Shivaji Ghodke<sup>a</sup>, Aslam Tamboli<sup>b</sup>, Arati Diwate<sup>c</sup>, Vijay Ubale<sup>d</sup>, Rajesh Bhorkade<sup>e</sup>, and Noormahmad Maldar<sup>e</sup>

<sup>a</sup>Department of Chemistry, Arts, Science and Commerce College, Naldurg, India; <sup>b</sup>Santosh Bhimrao Patil, ASC, Solapur, India; <sup>c</sup>Sangameshwar College Solapur, Solapur, India; <sup>d</sup>D.B.F. Dayanand College of Arts and Science, Solapur, India; <sup>e</sup>Punyashlok Ahilyadevi Holkar Solapur University, Solapur, India

## ABSTRACT

Thermally stable and organo-soluble polyamides synthesized by polycondensation reaction of novel diacid monomer, 2,2'-[pyridine-2,6-diylbis(oxy-4,1-phenylene)]diacetic acid with various commercially available aromatic diamines such as Pyridine-2,6-diamine, 4-(4-aminophenylsulfonyl) benzenamine, 4-(4-aminobenzyl)benzenamine and 4-(4-aminophenoxy)benzenamine by using Yamazaki's direct phosphorylation method. A 2,2'-[pyridine-2,6-diylbis(oxy-4,1-phenylene)]diacetic acid as a new compound containing methylene linkage and the heteroaromatic unit was successfully prepared by a displacement reaction. The resulted polymers show excellent thermal stability, good solubility in an aprotic polar solvent and inherent viscosities between 0.65 and 0.82 dL g<sup>-1</sup>. X-ray diffraction of all these polyamides shows amorphous nature. Polyamides showed glass-transition temperatures in the range of 193–199°C indicates that polymers are easily processable. Thermogravimetric analysis of the polyamides indicated a 10% weight loss ( $T_{10\%}$ ) in the temperature range of 460–518°C and char yields at 800°C under nitrogen atmosphere in the range of 50–65% depending on the diamine monomer used for the synthesis polyamides. The structure-property correlation among these polyamides were discussed. The limiting oxygen indexes of all polyamides are more than 37.87, thus such type polymers of can act as flame retardant materials. These polyamides will be useful for high-performance applications.

## ARTICLE HISTORY

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Polyamides; methylene; thermally stable; DSC; LOI

## Introduction

Aromatic Polyamides are well known for their high performance thermal polymeric material properties such as high thermal stability, excellent mechanical strength, heat-resistant properties, superior gas separation efficiency and good chemical resistance due to this property aromatic polyamides have a lot of application in various field such as military equipment, electronic field and automotive industry, etc. but there are few limitations associated in aromatic polymers because of their insolubility nature in many organic solvents and rigid nature of polymers which restrict their processability and practical application in many fields because strong intermolecular forces associated with them and rigid backbone nature of polyamides. A lot of research was made on these associated limitations during the last decade to overcome these limitations by using the

introduction of flexible ether linkages, methylene spacer group, non-coplanar moieties, cardo groups, a heterocyclic ring, replacing symmetrical aromatic rings with unsymmetrical ones and bulky pendant groups segments in their polymer backbone. All these parameters would decrease hydrogen bonding interaction and lowering chain packing of the polymer chain to reduce the crystallinity and rigid nature of the polymers.<sup>[1-19]</sup>

Works reported during the last decade related to the incorporation of flexible ether linkages, methylene spacer group, non-coplanar moieties, cardo groups, heterocyclic ring and bulky pendant groups in the polymer backbone chain to enhance the solubility and processability of these polymers without affecting physical and thermal properties to any great extent. Furthermore, in many cases, heterocyclic aromatic polyamides derived from heterocyclic ring containing monomers showed superior thermal stability and physical properties with improved solubility of polymers as a comparison with aromatic polyamide. The symmetry and aromaticity nature of the pyridine ring would assist to the thermal stability and physical properties of the resulting polymer and the polarization effect of a nitrogen atom in the pyridine ring would be suitable to enhance their solubility in many organic solvents.<sup>[20-27]</sup>

The present investigation deals with the synthesis of new novel heterocyclic diacid monomer, 2,2'-[pyridine-2,6-diylbis(oxy-4,1-phenylene)]diacetic acid derived from 2,6-dichloro pyridine and 4-hydroxy phenyl acetic acid via aromatic nucleophilic substitution reaction. The structure of synthesized new novel diacid monomer, 2,2'-[pyridine-2,6-diylbis(oxy-4,1-phenylene)]diacetic acid was confirmed by spectroscopic techniques FT-IR, <sup>1</sup>H NMR and <sup>13</sup>C NMR. Polyamides containing pyridine ring moiety, methylene spacer group and flexible ether linkage derived from 2,2'-[pyridine-2,6-diylbis(oxy-4,1-phenylene)]diacetic acid with four various diamine by direct Yamazaki phosphorylation reaction. The prepared polyamides confirmed by FT-IR and their thermal and physical properties were studied by using Viscosity, solubility, thermogravimetric analysis (TGA) differential scanning calorimetry (DSC), and wide-angle X-ray diffraction methods techniques. The obtained results investigated that Polymers containing pyridine ring moiety in their backbone improved thermal stability, glass transition temperature and solubility because of its molecular symmetry and Aromaticity nature at elevated temperature and nitrogen atom present in pyridine ring. The incorporation of pyridine ring, methylene spacer group, ether flexibility disrupt the molecular packing to reducing intermolecular interaction and enhancing the thermal stability, lowering the glass transition temperature and the effect of polarization from the nitrogen atom in the pyridine ring would improve the solubility of these polyamides.

## Experimental

### Materials

P-hydroxyl phenyl acetic acid, 2,6-dichloro pyridine, dry NMP, dry toluene, dry pyridine, triphenyl phosphite (Sigma-Aldrich) LiCl was dried under vacuum at 140 °C for 6 hrs. Pyridine-2,6-diamine (DAP), 4-(4-aminophenylsulfonyl)benzenamine (SDA), 4-(4-aminobenzyl)-benzenamine (MDA), 4-(4-aminophenoxy)benzenamine (ODA). All these diamine monomers were recrystallized prior to use.

### Measurements

Synthesized organic compounds and polymers FT-IR spectra were performed on a Nicolet spectrometer using KBr pellets. <sup>1</sup>H NMR (400 MHz) and <sup>13</sup>C NMR (100 MHz) spectra were recorded on a Bruker Advance spectrometer at 25 °C using CDCl<sub>3</sub> and DMSO-d<sub>6</sub> as solvent and tetramethylsilane as reference solvent. Polyamides inherent viscosities were measured with a polyamide concentration of 0.5 g dL<sup>-1</sup> in NMP solvent at 30 °C using an Ubbelohde suspended level viscometer. Differential scanning calorimetry (DSC) was performed on a Mettler Toledo DSC

STARE instrument at a heating rate of 200 °C/min under nitrogen. Thermogravimetric analysis (TGA) was recorded on a Mettler Toledo STARE instrument at a heating rate of 10 °C/min under nitrogen. Wide angle X-ray diffraction (WAXD) was performed with a Rigaku X-ray diffractometer using polyamide powder.

### Synthesis of diacid

Synthesis of new novel monomer, 2,2'-[pyridine-2,6-diylbis(oxy-4,1 phenylene)]diacetic acid was prepared according to the literature procedure from P-hydroxyl phenyl acetic acid as starting material.<sup>[28]</sup> The obtained product was recrystallized in absolute ethanol and dried under vacuum at 55 °C. Yield: 90% and M.P. 150 °C.

FT-IR (KBr,  $\text{cm}^{-1}$ ): 3030  $\text{cm}^{-1}$  (–OH), 2922  $\text{cm}^{-1}$  aliphatic –CH, 1696  $\text{cm}^{-1}$  (Carbonyl).

$^1\text{H}$  NMR (DMSO, ppm): 3.47 ppm (s, 4H), 6.3 ppm (d, 2H), 6.9 ppm (d, 4H) 7.1 ppm (d, 4H), 7.5 ppm (dd, 1H).

$^{13}\text{C}$  NMR (DMSO, ppm): 40.64 ppm ( $\text{CH}_2$ ), 104.31, 120.96, 130.49, 131.05, 142.36, 152.66, 162.29 ppm (CH of aromatic), 173.44 ppm (–COOH).

### Polymerization

A mixture of 0.379 g (1 mmol) of 2,2'-[pyridine-2,6-diylbis(oxy-4,1-phenylene)]diacetic acid monomer, 0.109 g (1 mmol) of 2,6-Diaminopyridine, 0.200 g of lithium chloride, 0.63 ml of tri-phenyl phosphite, 0.5 ml of pyridine, and 2.5 ml of NMP was heated at 110 °C with stirring for 3 h under nitrogen atmosphere by Yamazaki phosphorylation polycondensation reaction. After completion of the reaction, the mixture was cooled at room temperature then poured into a large amount of methanol with constant stirring, formed a fibrous precipitate was filtered, washed thoroughly with methanol and hot water and dried at 120 °C under vacuum. The inherent viscosities were observed in the range of 0.65–0.82 dL  $\text{g}^{-1}$ .

The IR spectrum: carbonyl absorption of amide at 1654.45  $\text{cm}^{-1}$  and 3267.94  $\text{cm}^{-1}$  (–NH stretch of amide). The other three polyamides (PA-I, II and III) were prepared according to a similar procedure.

### Result and discussion

The Investigation of this study was focused on improving the solubility of the novel polyamides without affecting the physical and chemical properties of polyamides. We have synthesized series of heterocyclic aliphatic-aromatic polyamides containing methylene spacer group, ether linkages and pyridine moiety in the polymer backbone chain by Yamazaki's phosphorylation reaction to improve the thermal properties, solubility and inherent viscosity of these polyamides. The inherent viscosities of the heterocyclic aromatic polyamides were observed in the range of

**Table 1.** Thermal properties of polyamides.

Polymer code	$T_i^a$ (°C)	$T_{10\%}^b$ (°C)	$T_g^c$ (°C)	Residual wt. % at 800 °C	LOI
PA-I	230	558	198.70	65.13	43.55
PA-II	232	487	196.78	50.94	37.87
PA-III	221	514	193.10	59.72	41.38
PA-IV	225	460	194.42	58.75	41.00

<sup>a</sup> $T_i$ : Temp at which weight loss initiated.

<sup>b</sup> $T_{10\%}$ : Temp at which 10% weight loss was observed.

<sup>c</sup> $T_g$ : Determined by DSC measured at a heating rate 20 °C/min. under nitrogen atmosphere.

LOI: Limiting oxygen index.



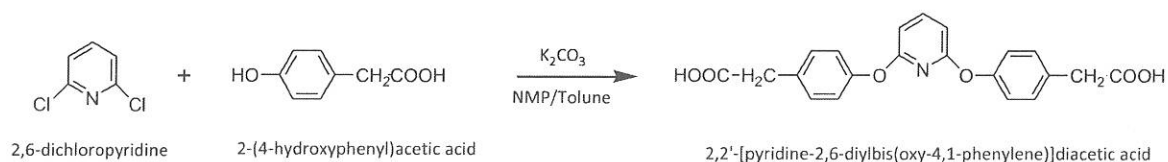
0.65–0.82 dL g<sup>-1</sup> which indicate a relatively high molecular weight of polymers as shown in Table 1. The synthesized polyamides and 2,2'-[pyridine-2,6-diylbis(oxy-4,1-phenylene)]diacetic acid monomer was characterized by TLC FT-IR, NMR (<sup>1</sup>H, <sup>13</sup>C) and elemental analysis techniques. The thermal behavior of polyamides was studied by the DSC showed glass transition temperature between 193.10 °C and 198.70 °C and the results are summarized in Table 1. The thermal stability of polyamides was measured by the TGA. The initial decomposition temperature was in the range of 221–232 °C and 10% weight loss temperature was in the range of 460–518 °C and the results are summarized in Table 1.

### Synthesis of diacid

The 2,2'-[pyridine-2,6-diylbis(oxy-4,1-phenylene)]diacetic acid monomer was synthesized from 4-hydroxy phenyl acetic acid and 2, 6 dichloro pyridine via aromatic Nucleophilic substitution reaction in the presences of base K<sub>2</sub>CO<sub>3</sub> by azeotropic distillation method using literature procedure (Scheme 1).<sup>[28]</sup>

### Characterization of diacid

The chemical structure of 2,2'-[pyridine-2,6-diylbis(oxy-4,1-phenylene)]diacetic acid monomer was confirmed by FT-IR and NMR (<sup>1</sup>H <sup>13</sup>C). FT-IR spectrum of 2,2'-[pyridine-2,6-diylbis(oxy-4,1-phenylene)]diacetic acid showed characteristic stretching absorption broadband at 3030 cm<sup>-1</sup> due to the hydroxyl group (–OH) of carboxylic acid, 2922 cm<sup>-1</sup> due to the aliphatic –CH stretching of methylene spacer group and absorption band at 1696 cm<sup>-1</sup> due to the carbonyl stretching



Scheme 1. Synthesis of 2,2'-[pyridine-2,6-diylbis(oxy-4,1-phenylene)]diacetic acid monomer.

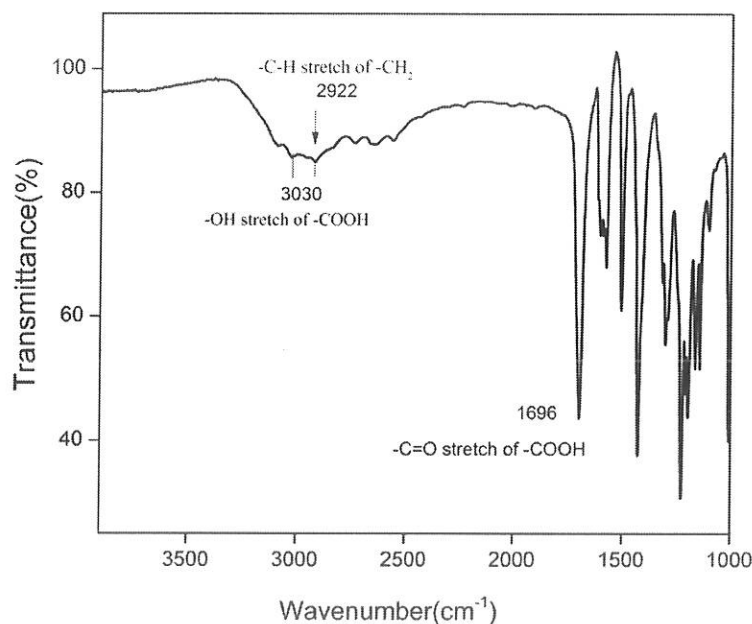


Figure 1. FT-IR Spectrum of 2,2'-[pyridine-2,6-diylbis(oxy-4,1-phenylene)]diacetic acid.



The heterocyclic aliphatic-aromatic polyamides were prepared by direct polycondensation of 2,2'-[pyridine-2,6-diylbis(oxy-4,1-phenylene)]diacetic acid with various aromatic diamines in NMP by the Yamazaki et al. method using TPP and Pyridine as condensing agents (Scheme 2). The obtained polyamides were quantitative yields with inherent viscosity observed in the range of 0.65–0.82 dL g<sup>-1</sup>. All structural features of these resulting polyamides were verified by FT-IR (Figure 5). They exhibit characteristic absorption band of amide group at 3267.94 cm<sup>-1</sup> is due to the –NH stretching of amide indicates the disappearance of carboxylic group in polyamide and carbonyl stretching absorption band at 1654.61 cm<sup>-1</sup>.



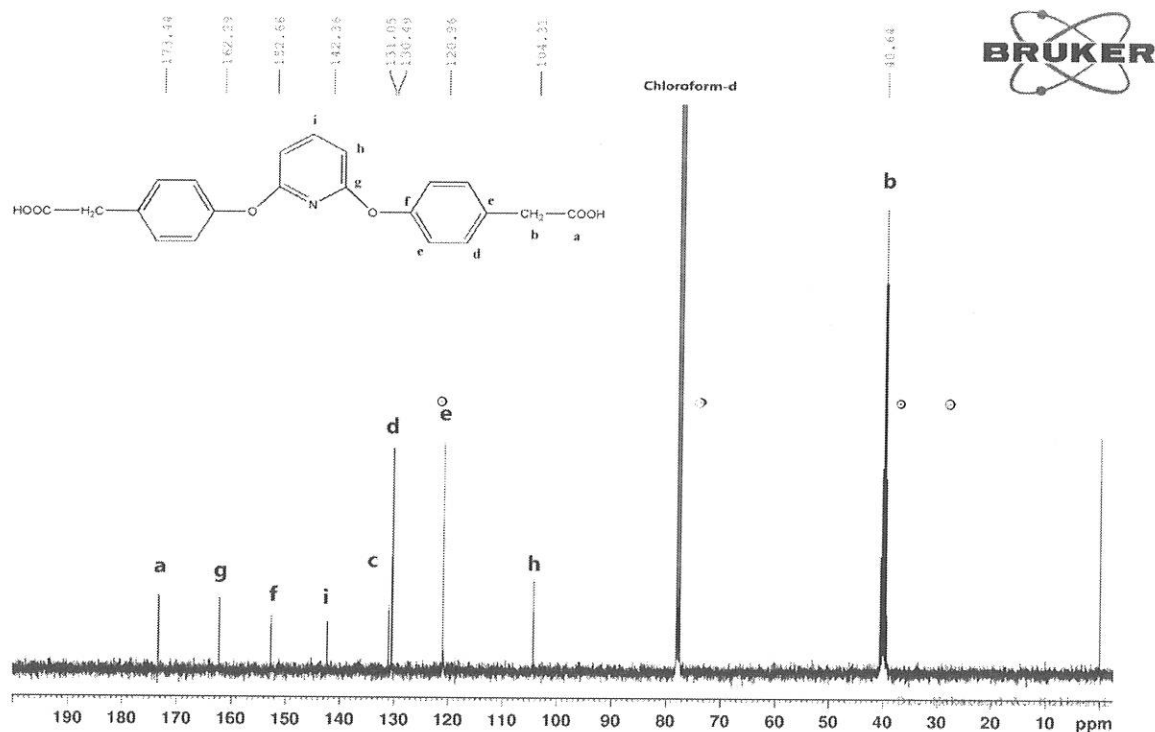


Figure 3.  $^{13}\text{C}$  NMR Spectrum of 2,2'-[pyridine-2,6-diylbis(oxy-4,1-phenylene)]diacetic acid.

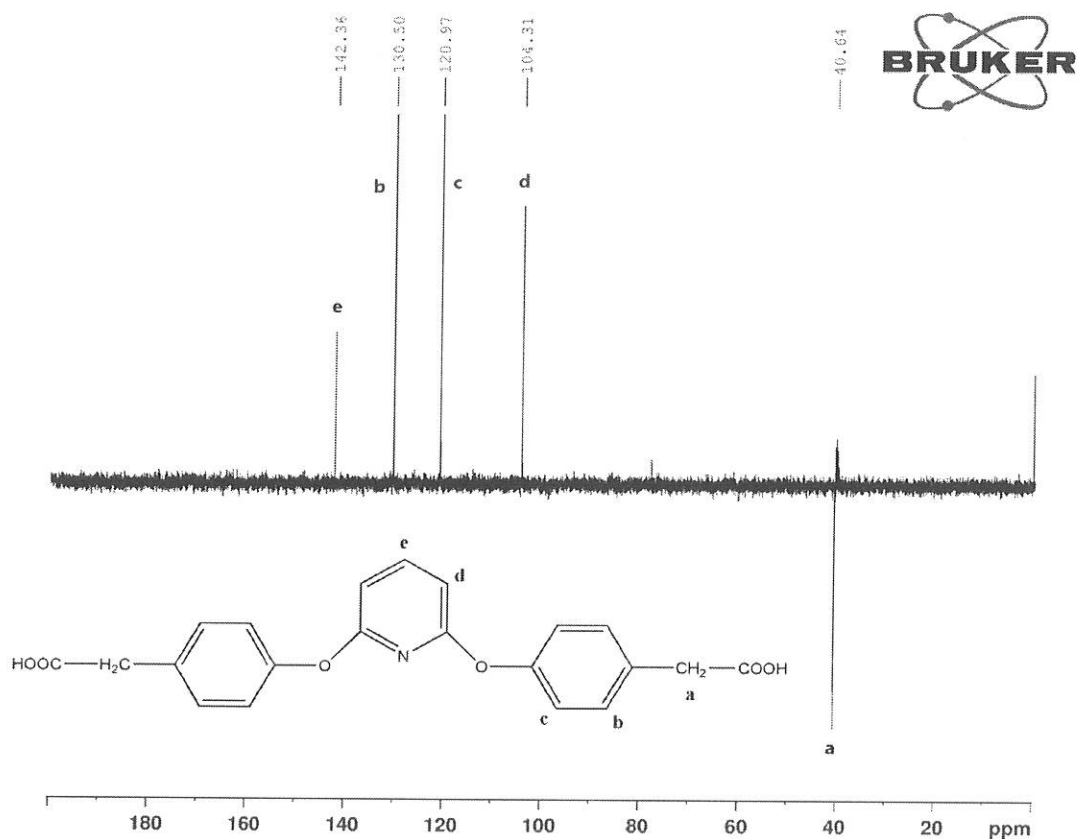
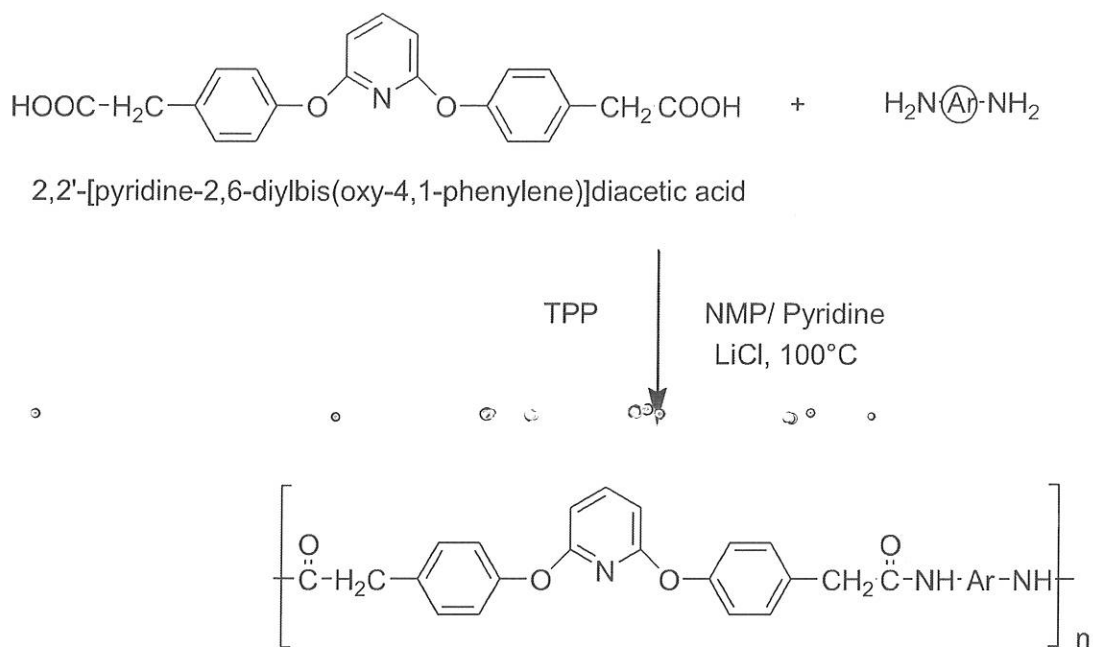
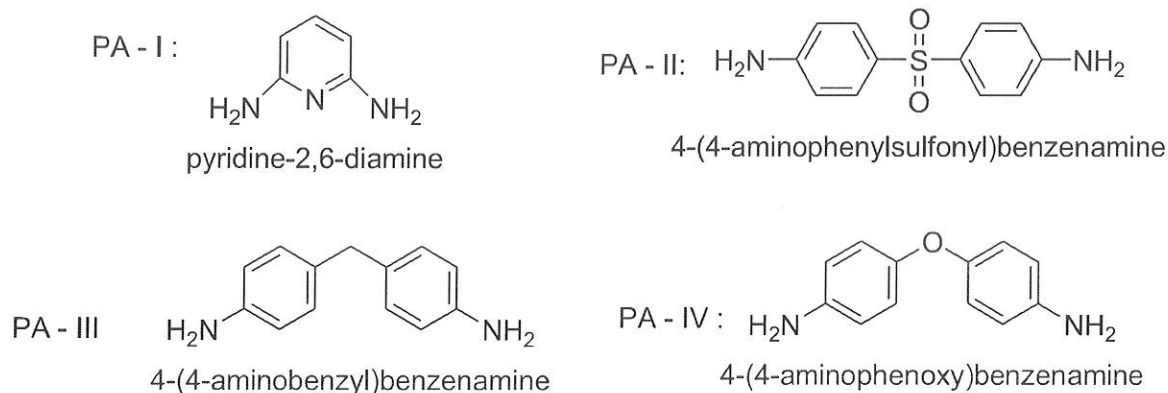


Figure 4. DEPT  $^{13}\text{C}$  NMR spectrum of 2,2'-[pyridine-2,6-diylbis(oxy-4,1-phenylene)]diacetic acid.



Whereas -Ar- =



Scheme 2. Synthesis of polyamides from 2,2'-[pyridine-2,6-diylbis(oxy-4,1phenylene)]diacetic acid and various aromatic diamine.

### Solubility of polyamides

The prepared polyamides were readily soluble in common organic solvents. The introduction of ether linkages, methylene spacer group and pyridine units into the backbone of polymers was the main reason for enhancing the solubility of the polymers.<sup>[30-31]</sup> The ether linkages and methylene spacer group provided more free volume due to the low internal rotational energy about these bonds and the polarization effect of pyridine units between polymer and solvent. These new heterocyclic aliphatic polyamides exhibited higher solubility than the conventional aromatic polyamides as shown in Table 2.

The X-ray studies revealed that all the polymers were completely amorphous. The representative XRD curves of polyamides (PA-I to IV) are shown in Figure 6. Thus, the amorphous nature of these polymers was reflected in their excellent solubility and this could be attributed to the incorporation of the heterocyclic pyridine ring, methylene spacer group and ether linkages in the polymer backbone.

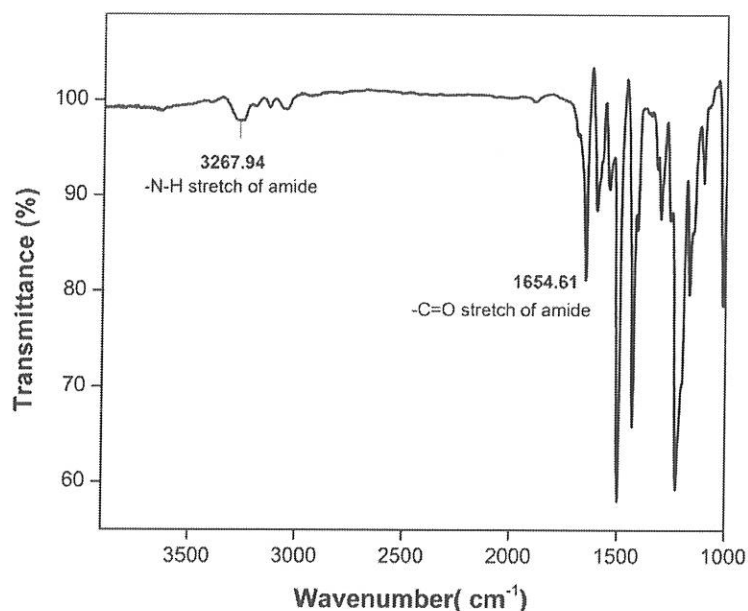


Figure 5. FT-IR Spectrum of PA-I.

Table 2. The solubility of polyamides.

Polymer → Solvent ↓	PA-I	PA-II	PA-III	PA-IV
NMP	++	++	++	++
DMF	++	++	++	++
DMSO	++	++	++	++
DMAc	++	++	++	++
THF	+-	+-	++	++
m-Cresol	++	++	++	+-
DCM	+-	+-	+-	+-
CHCl <sub>3</sub>	+-	--	+-	--
H <sub>2</sub> SO <sub>4</sub>	++	++	++	++

++: Soluble; +-: partially soluble; --: insoluble.

### Thermal properties

The thermal characteristic and stability of the prepared polyamides was evaluated by means of TGA and DSC in a nitrogen atmosphere at a heating rate of 10 °C/min and the results are reported in Table 3. The DSC and TGA curves of polyamides PA I to PA IV are shown in Figures 7 and 8. This series of polyamides showed glass transition temperature in the range of 193.10–198.70 °C. The  $T_g$  values polyamides increased with the increasing rigidity effect of the polymer repeat unit. The thermal stability of polyamides was investigated by TGA. The polyamides started to lose weight, due to the thermal degradation was in the range of 221–232 °C. TGA thermograms indicated that all the polymers have good thermal stability with 10% decomposition temperatures were in the range of 460–518 °C.

The thermal properties of the heterocyclic Polyamides depend not only on the structural composition but also on the rigid nature of polymers. The amount of carbonized residue (char yield) value was 65.13% (PA-I), 50.94% (PA-II), 59.72% (PA-III), 58.75% (PA-IV) at 800 °C respectively as shown in Table 3. The amount of carbonized residue (char yield) value at 800 °C of polyamides containing pyridine (PA-I) moiety are more than the other polyamides containing sulfone linkages (PA-II), methylene spacer (PA-III), and flexible ether (PA-IV) moiety, due to the disrupt the molecular packing to reducing intermolecular interaction and enhancing the thermal stability. A higher order of thermal stability is achieved in this polyamides.

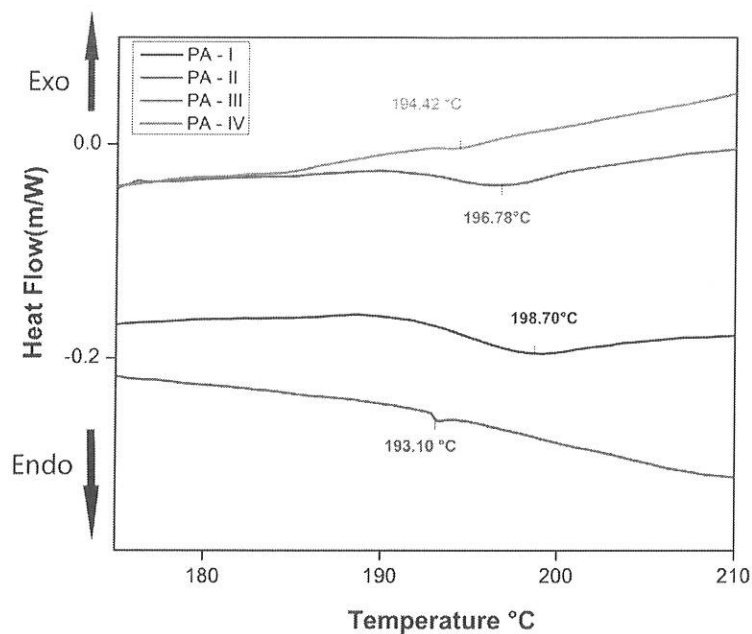


Figure 6. DSC curves of polyamides, PA-I to PA-IV.

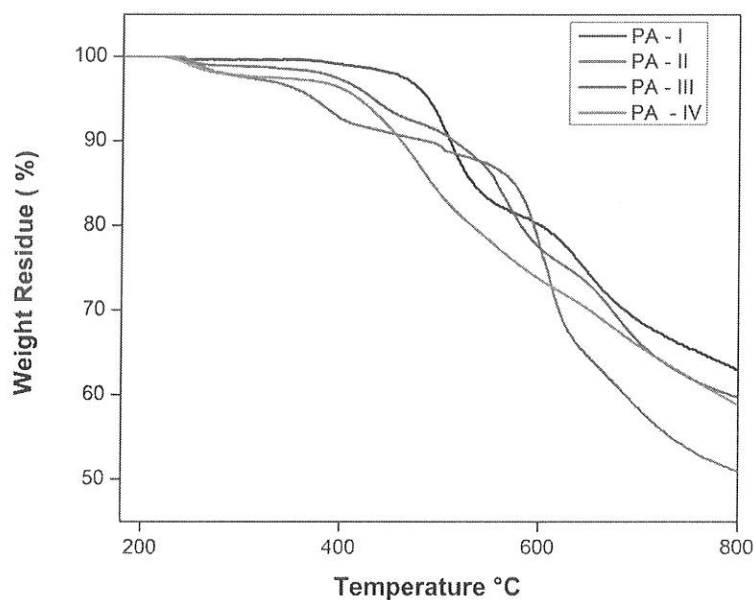


Figure 7. TGA curves of polyamides, PA-I to PA-IV.

Comparative Thermal Stability Study of heterocyclic aliphatic aromatic polyamides with literature reported work on the basis of heterocyclic aromatic polyamides.<sup>[32–35]</sup> The obtained results are summarized in Table 4.

According to the obtained results, heterocyclic aliphatic aromatic polyamides showed suitable thermal stability and solubility than the heterocyclic aromatic polyamides due to the presence of rigid pyridine ring, ether linkages and methylene spacer group respectively, in the polyamide's backbone.

#### Flame-retardant property of polyamides (PA-I to PA-IV)

According to Van Krevelen and Hoftyzer's equation,<sup>[36]</sup> the char yield can be useful as a conclusive factor for valuing the limited oxygen index (LOI) of polymers using the following equation,



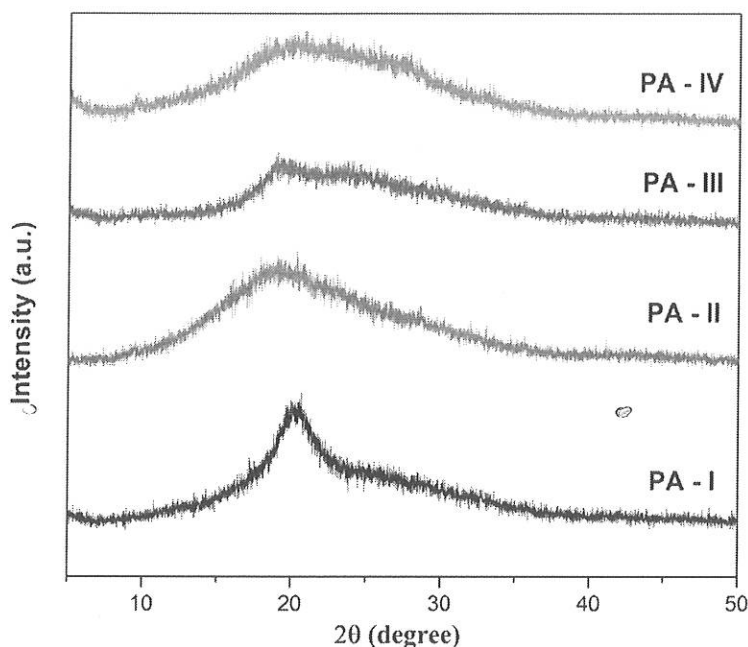


Figure 8. TGA curves of polyamides, PA-I to PA-IV.

Table 3. Yield and inherent viscosity of polyamides (PA-I to PA-IV).

Sr. no.	Polymer code	Diamine	Yield (%)	Inherent viscosity $\eta_{inh}$ (dL/g)
1.	PA-I	Pyridine-2,6-diamine (DAP)	92	0.68
2.	PA-II	4-(4-aminophenylsulfonyl)benzenamine (SDA)	94	0.65
3.	PA-III	4-(4-aminobenzyl)benzenamine (MDA)	96	0.76
4.	PA-IV	4-(4-aminophenoxy)benzenamine (ODA)	97	0.82

<sup>a</sup>Polymerization was carried out with 1 mmol each of 2,2'-[pyridine-2,6-diylbis(oxy-4,1-phenylene)]diacetic acid and various aromatic diamine.

<sup>b</sup>Measured with 0.5% (w/v) polymer solution in NMP at  $30 \pm 0.1$  °C.

Table 4. Comparative study of thermal stability.

Polymers	$T_g$ value range (°C)	$T_{10\%}$ value range (°C)
Current work polymers	193–198	460–558
Reported work by Soumendu Bisoi et al. <sup>[32]</sup>	235–248	390–425
Reported work by Khalil Faghihi et al. <sup>[33]</sup>	–	345–490
Reported work by G. A. Koohmareh et al. <sup>[34]</sup>	60–180	390–410
Reported work by Saharam Mehdipour-Ataei et al. <sup>[35]</sup>	138–229	345–465

$$LOI = 17.5 + 0.4CR,$$

where CR is the percentage of polymer remaining at 800 °C.

In general, the material constituents showing LOI values higher than 26 show self-extinguishing behavior and reflecting as flame retardant. On the basis of the LOI values the resulting polymers can be classified as self-extinguishing materials. All polyamides have LOI more than 37.87 and such polymers can act as flame-retardant materials are summarized in Table 1. The LOI of all polyamides (PA-I to PA-IV) estimated in the range of 37.87–43.55.

## Conclusion

A new 2,2'-[pyridine-2,6-diylbis(oxy-4,1-phenylene)]diacetic acid was successfully synthesized via Azeotropic distillation method with good yield. Heterocyclic aliphatic aromatic polyamides were

synthesized from 2,2'-[pyridine-2,6-diylbis(oxy-4,1-phenylene)]diacetic acid monomer with various aromatic diamines by Yamazaki phosphorylation reaction. Prepared polymers appeared amorphous in nature with good solubility in many aprotic solvents. Incorporation heterocyclic pyridine ring, ether linkages and methylene spacer group in polymer backbone enhance the solubility and thermal stability of polymers. From above result it was confirmed that the introduction of methylene spacer group and ether linkages into the polymer backbone was highly effective for the preparation of Heterocyclic aliphatic aromatic polyamides having both high  $T_g$  and high  $T_{10}$  value than the polymers derived from only Pyridine moiety containing polyamides. The developed polymer could be used as a high-performance polymeric material.

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Arts Science & Commerce College  
Naldurg, Dist.Osmanabad-413602