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# Synthesis and properties of polyimides derived from bis-(Aminophenoxy) containing naphthalene, [Phenyl] propane and [Methyl] cyclohexane segment and 4, 4'-carbonyldipthalic anhydride

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## ABSTRACT

Three wholly, semi aromatic and aliphatic-aromatic polyimides containing bis(phenoxy) naphthalene, bis [(phenoxy) phenyl] propane and bis(phenoxy-methyl) cyclohexane segments by the two-step procedure from 2, 7-bis(4-aminophenoxy) naphthalene (BAPON), 2, 2-bis[4-(4-aminophenoxy)phenyl]propane (BAPOP), 1, 4-bis (4-aminophenoxy methyl) cyclohexane (BAPMC) as a diamine and 4,4'-carbonyldipthalic anhydride (CDPA) were prepared. The first step of this procedure including ring-opening polyaddition in a polar solvent to give poly(amic-acid)s, second step containing cyclodehydration reaction to form polyimides. Synthesized monomer and polyimides were characterized by FT-IR, <sup>1</sup>H NMR spectroscopy and elemental analyses (CHN) that obtained results gave the most powerful evidence. The polyimide synthesized from BAPON was characterized as semi-crystalline, whereas the other polyimides showed amorphous patterns by the x-ray diffraction studies. The inherent viscosity was ranging between 0.87–1.01 dL/g. Tensile strength, initial moduli, and elongation at break of the polyimide films ranged from 88–117 MPa, 1.98–2.32 GPa, and 5–8%, respectively. Thermogravimetric analysis in nitrogen atmosphere shows that these polymers having good stability, so 10% weight will be lost in the range of 500–630°C. The point of polyimide with BAPMC segment, is “adding of good thermal stability and processability” lower moisture absorption and dielectric constant (0.75% and 2.90).

## ARTICLE HISTORY

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## 1. Introduction

Aromatic polyimides are important and well-known high-performance polymer materials with outstanding properties such as high transition temperatures, excellent thermal stability, high mechanical properties, considerable optical and electrical properties, good dimensional and thermo-oxidative stability and solvent resistance. As a result, they are widely applied in various applications such as photoresist, electronics instrument, gas separation membranes, coatings, automobile, films, adhesives, printed circuit industries and composite materials in aerospace industries.<sup>[1–8]</sup> Recently, they are widely applied in the fields of aviation, aerospace, microelectronics, liquid crystal, separation film and laser.<sup>[9,10]</sup> However, the application of aromatic polyimides in fully imidized form are limited due to lack of solubility, processability and tractability.<sup>[11–15]</sup> In order to overcome the above said problems there are several approaches have been used as following: May by used poly(amic-acid) solutions in casting process and thermal treatment to prepared polyimide films. Essential problems of this method are released volatile byproducts (for instance, water) and instability of the poly(amic-acid). Other methods are random copolycondensation reaction<sup>[16]</sup> and applying synthesizing new dianhydrides or diamines with polar group, ether

linkages, bulky pendant groups, asymmetric moieties and meta- or ortho- substitution that are produced great variety of soluble and processable polyimides. The designed synthesized diamines incorporated cyclic ring or aliphatic chain can help improving solubility, processability and tractability of polyimides. The introduced flexible segments such as -C-, -O-, -CH<sub>2</sub>-, C (CH<sub>3</sub>)<sub>2</sub> and -COOH in polymer backbone can alter crystallinity and intermolecular interactions therefore solubility and processability are increased.<sup>[17]</sup> Generally, aromatic polymers containing both carbonyl groups and aryl ether linkages improved characteristics. At the result, three polyimides including these two groups have been prepared.

In this report, 4-bis (4-aminophenoxy methyl) cyclohexane were prepared as a designing monomer in two steps through the nucleophilic substitution reaction of 1,4-bis (hydroxymethyl) cyclohexane and 1-fluoro-4-nitro benzene, subsequent reduction with hydrazine/Pd-C. Meanwhile, polyimides were synthesized from the designing bis-amino compound and 4,4'-carbonyldipthalic anhydride. Due to the price of diols, consumption reagent, and low energy, lower cost of in situ reaction, which used for the preparation of polyimides, is considered. On the other hand the solubility and processability of the polyimides comparison with fully aromatic, due to

introduction of the flexible segments  $-O-$ ,  $C(CH_3)_2$  and cyclohexyl in the polyimide backbone, were increased.

## 2. Experimental

### 2.1. Materials

According to the reported method,<sup>[18,19]</sup> 2,2-Bis [4-(4-aminophenoxy)phenyl] propane (BAPOP) and 2,7-bis (4-aminophenoxy) naphthalene (BAPON) were prepared. 4,4'-Carbonyldiphthalic anhydride was recrystallized from acetic anhydride before use (CDPA, from Aldrich), *N,N*-Dimethylacetamide (DMAc, from Fluka) was purified by distillation under reduced pressure.

### 2.2. Apparatus and measurements

$^1H$ -NMR spectra were determined on a Bruker AV-300 MHz NMR spectrometer. FT-IR spectra were recorded on a Galaxy series FT-IR 5000 Fourier Transform Infrared spectrophotometer. Elemental analysis was run in a Vario El mark. Inherent viscosities of all polymers were obtained at 0.5 g/dL concentration by used Technico Registered Trade Mark viscometers. TGA data of samples were taken on a Mettler TA 4000 System under an  $N_2$  atmosphere at a rate of  $10^\circ C/min$ . Melting points measured with Digital

Melting Point Apparatus. The thermal properties of the samples were measured by differential scanning calorimetry (DSC 200 F3, NETZSCH) in the range between 30 and  $350^\circ C$  at heating rate of  $10^\circ C/min$  in nitrogen atmosphere. To measure the mechanical properties of polymer films, they were cut into strips with a width of 5 mm in advance. The measurement was conducted on a SANTAM STM-50 with a gauge length of 20 mm and a crosshead speed of 5 mm/min. Each specimen was measured four times and the values were averaged.

### 2.3. Synthesis of 1,4-bis-(4-nitrophenoxy methyl) cyclohexane (BNPMC)

Into the round bottom flask which is equipped with a magnetic stirring and it contain 50 mL of DMF, the following materials including 1,4-bis (hydroxymethyl) cyclohexane (4.04 g, 28.01 mmol), 1-fluoro-4-nitro benzene (9.44 g, 66.90 mmol) and anhydrous potassium carbonate (9.70 g, 69.98 mmol) are added. After few minutes, the brown colored reaction mixture changed to yellow green. Reaction mixture stirred at room temperature for 24 h into the nitrogen atmosphere, and then filtered. The filtrate diluted with ethyl acetate (120 mL), and the organic layer separated. The organic layer washed three times with water, then dried with magnesium sulfate, and filtered.

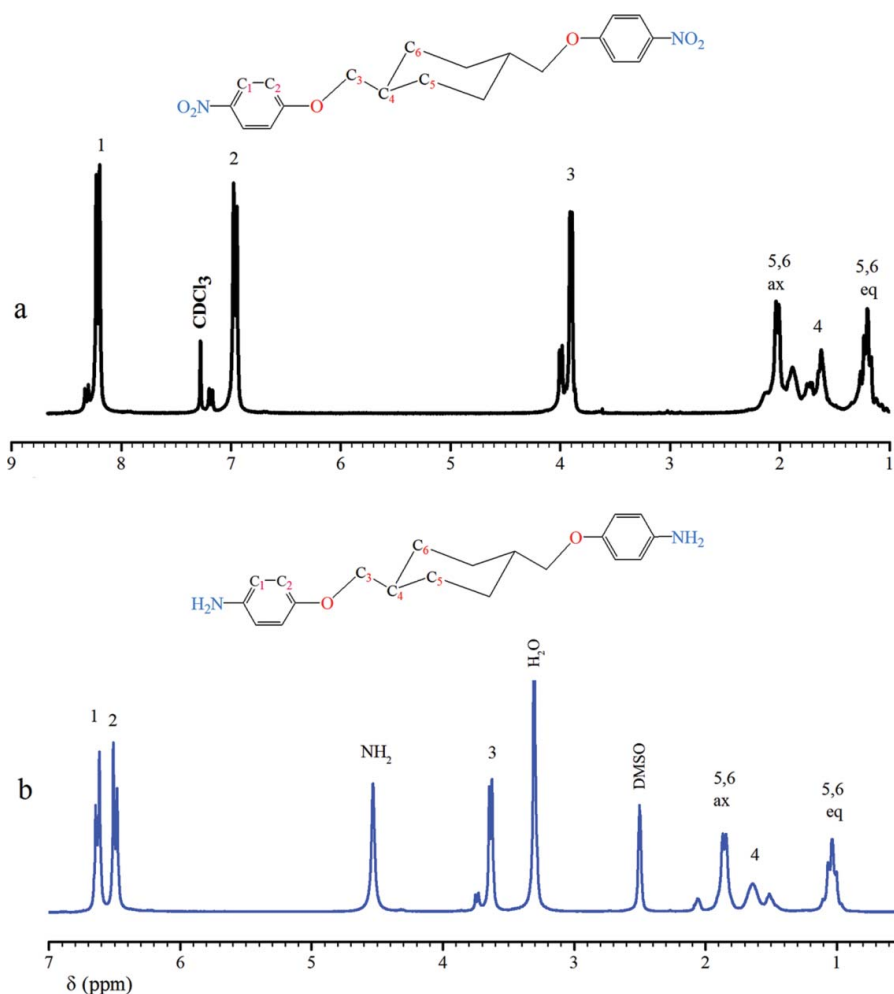


Figure 1.  $^1H$  NMR spectra of a) BNPMC and b) BAPMC.

The filtrate evaporated to afford golden rod crystals, which recrystallized from ethanol to yield 9.74 g (90%) of off-white crystals; mp 105–107°C and  $^1\text{H}$  NMR spectra compound is shown in Figure 1a.<sup>[18]</sup>

FT-IR (KBr,  $\text{cm}^{-1}$ ): 2926 (H-C aromatic), 2856 (H-C aliphatic), 1593 ( $\text{NO}_2$ ), 1338 ( $\text{NO}_2$ ), 1508 (C-N), 1458 (C = C aromatic), 1236 (C-O-C).

$^1\text{H}$  NMR: ( $\text{CDCl}_3$ ,  $\delta$  in ppm): 1.16–1.25 (m, 4H, H5eq and H6eq), 1.52–1.68 (m, 2H, H4), 1.81–2.03 (m, 4H H5ax and H6ax), 3.86–3.91 (d, 4H,  $\text{CH}_2$ ), 6.94–6.97 (d, 4H, Ar-H), 8.19–8.22 (d, 4H, Ar-H)

Anal. Calcd for  $\text{C}_{20}\text{H}_{22}\text{N}_2\text{O}_6$ : C, 62.16%; H, 5.74%; N, 7.25%. Found: C, 62.72%; H, 5.5%; N, 7.01%.

## 2.4. Synthesis of 1, 4-bis (4-aminophenoxy methyl) cyclohexane (BAPMC)

In a round bottom flask with a magnetic stir (5.00 g, 12.87 mmol) of BNPMC and Pd/C (0.16 g of 10%) were dispersed in 20 mL of ethanol. A suspended solution refluxing and about 8 mL of hydrazine hydrate was added drop wise and the resultant reaction mixture was refluxed at 85°C for 12 h. To remove palladized charcoal the hot black colored solution was filtered. The filtrate was poured into a large amount of ice-cold water with efficient stirring. A light yellow precipitate of 1, 4-bis (4-aminophenoxy methyl) cyclohexane, was filtered, washed with ice cold water, and then dried at room temperature. The yield of the BAPMC was 90% (3.80 g) with mp 138–140°C and  $^1\text{H}$  NMR spectrum compound is shown in Figure 1b.

FT-IR (KBr,  $\text{cm}^{-1}$ ): 3456–3387 ( $\text{NH}_2$ ), 2987 (H-C aromatic), 2869 (H-C aliphatic), 1634 (N-H), 1236 (C-O-C), 829 (para-subst. arom.).

$^1\text{H}$  NMR ( $\text{DMSO}-d_6$ ,  $\delta$  in ppm): 0.96–1.11 (m, 4H, H5eq and H6eq), 1.45–1.60 (m, 2H, H4), 1.75–1.86 (m, 4H H5ax and H6ax), 3.62–3.64 (d, 4H,  $\text{CH}_2$ ), 4.53 (s, 4H,  $\text{NH}_2$ ), 6.48–6.51 (d, 4H, Ar-H), 6.61–6.64 (d, 4H, Ar-H).

Anal. Calcd for  $\text{C}_{20}\text{H}_{28}\text{N}_2\text{O}_2$ : C, 73.13%; H, 8.59%; N, 8.53%. Found: C, 72.21%; H, 8.80%; N, 8.28%.

## 2.5. Synthesis of polyimides

The typical procedure of polymerization is described as follow: 0.30 g (0.92 mmol) of BAPMC solution of in 10 mL of DMAc in several portions then 0.29 g (0.92 mmol) of CDPA was added. The mixture was stirred at room temperature for 10 h under nitrogen atmosphere. The inherent viscosity of the poly(amic-acid) in the DMAc solution was 1.98 dL/g, measured at a concentration of 0.5 g/dL at 25°C. In order to obtain casting film onto a glass plate, poly

(amic-acid) solution was spread. The imidization could be carried out by the thermal cyclodehydration of the poly (amic-acid) film by sequential heating at 60, 90, 120, 150, 180, 210 and 250°C for 45 min each, respectively. The  $\eta_{\text{inh}}$  of the flexible polyimide film was 1.38 dL/g in concentrated sulfuric acid, measured at a concentration of 0.5 g/dL at 25°C.

## 3. Results and discussion

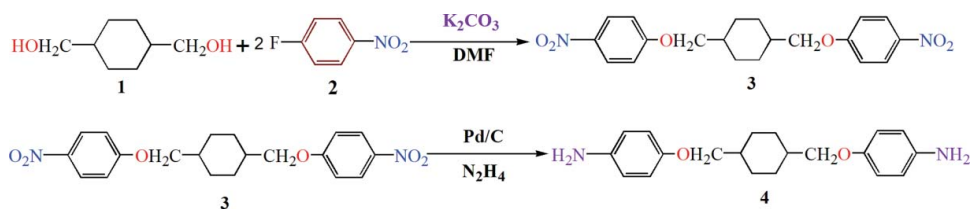
### 3.1. Monomers synthesis

1, 4-bis(4-aminophenoxy methyl) cyclohexane (BAPMC) was prepared by using a two-step process (Scheme 1).

In the first step, BNPMC was prepared from 1,4-bis (hydroxymethyl) cyclohexane and 1-fluoro-4-nitrobenzene in the presence of potassium carbonate. Nucleophilic substitution with present electron-withdrawing group ( $\text{NO}_2$ ) in the 1-fluoro-4-nitrobenzene is occurred and activated fluoro-atom for displacement. In the second step, BNPMC was reduced in ethanol in the presence of hydrazine hydrate and trace amount of palladium on the activated carbon as a catalyst at 80°C to produce light yellow crystals of BAPMC. The structure of intermediate BNPMC and diamine (BAPMC) were identified by elemental analysis, FT-IR and  $^1\text{H}$  NMR spectroscopy. According to experimental, the elemental analysis values of the BNPMC and BAPMC compounds agreed with the calculated values of their structures. In particular,  $^1\text{H}$  NMR and FT-IR spectra gave the most powerful evidence. In the FT-IR spectrum of BNPMC compound show symmetric and asymmetric stretching of  $-\text{NO}_2$  group appearing around 1338 and 1583  $\text{cm}^{-1}$ . By reducing nitro groups in BNPMC peak absorption of the nitro groups is disappeared and instead the peak of amino groups will be observed at 3387–3456  $\text{cm}^{-1}$  ( $\text{NH}_2$  stretching). Other characteristic absorption of amino groups appeared at 1635  $\text{cm}^{-1}$  (N-H bending) that is shown in the Figure 2. According to experimental, when BNPMC compound was converted into BAPMC, a broad peak at 4.94 ppm in the  $^1\text{H}$  NMR spectrum was appeared related to amino group.

### 3.2. Polymer preparation

Conventional two-step procedure was used to prepare polyimide containing BAPMC segment. At the first step from the reaction, aromatic-aliphatic diamine and dianhydride in DMAc, poly(amic-acid) will be produced. In the polymerization reaction, adding monomers has very important role. This effect was investigated by viscosity of poly(amic-acid) containing BAPMC and CDPA. Obtained results of this study presented in Table 1. The results show that mode addition of



Scheme 1. Synthesis route of BNPMC and BAPMC.

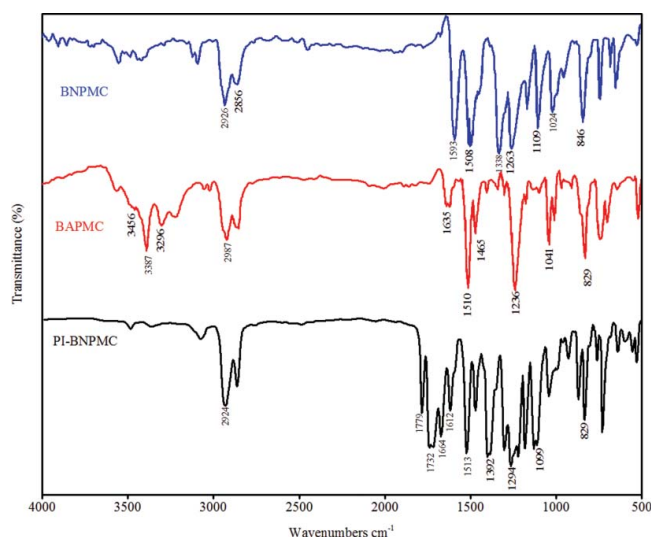


Figure 2. FT-IR spectra of BNPMC, BAPMC Polyimide.

Table 1. Effect of the monomer addition in the viscosity of poly(amic-acid).

| Run | Process <sup>a</sup>   | $\eta_{inh}^b$ of poly(amic-acid) (dL/g) |
|-----|--|--|
| 1   | BAPMC and CDPA added into DMAc in one portion  | 1.24                                     |
| 2   | First BAPMC was dissolved in DMAc, then at the once CDPA was added                     | 1.38                                     |
| 3   | First CDPA was dissolved in DMAc, then at the BAPMC once was added                     | 0.85                                     |
| 4   | First BAPMC was dissolved in DMAc, and every 30 min once, CDPA was added in four steps | 1.48                                     |
| 5   | First CDPA was dissolved in DMAc, then BAPMC was added in four portions within 30 min  | 0.80                                     |

<sup>a</sup>in each process 0.30 g BAPMC, 0.29 g CDPA and 10 mL of DMAc were used.

<sup>b</sup>Measured at a concentration of 0.5 g/dL in DMAc at 25°C

CDPA in 4 portions to the diamine solution produced poly(amic-acid) with a higher molecular weight than other modes. The highest inherent viscosity in this mode is 1.48 dL/g (see run 4).<sup>[20]</sup>

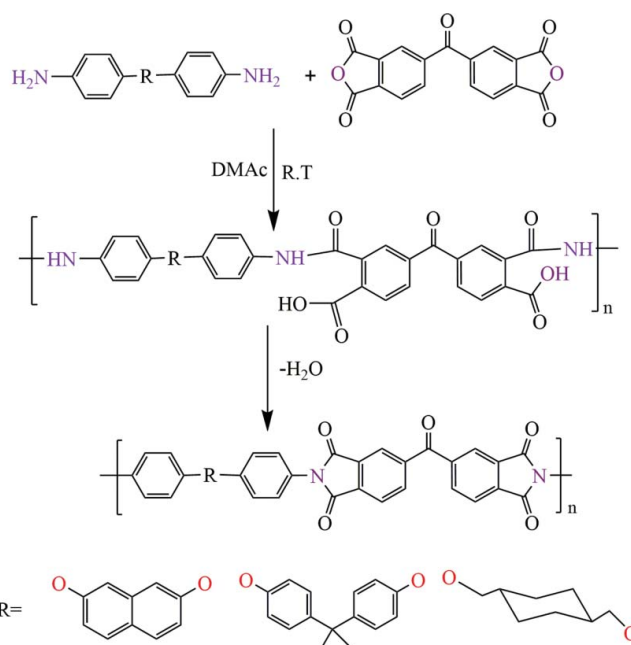
According to run 4 in Table 1 other poly(amic-acid) were prepared (see Scheme 2). Inherent viscosities of all poly(amic-acid)s with those of the corresponding polyimides are shown in the Table 2. The ring opening polyaddition in DMAc at room temperature produced poly(amic-acid)s with inherent viscosities in range 1.11–1.48 dL/g. Among them, the highest inherent

Table 2.  $\eta_{inh}$  of poly(amic-acid) and Polyimide with elemental analysis.

| poly(amic-acid) |                                  |                                  | polyimide   |                    |       |       |
|-----------------|----------------------------------|----------------------------------|---|--------------------|-------|-------|
| Bis-amine Code  | $\eta_{inh}$ (dL/g) <sup>a</sup> | $\eta_{inh}$ (dL/g) <sup>b</sup> | Formula   | Elemental Analysis |       |       |
|                 |                                  |                                  | Formula Wight   | C (%)              | H (%) | N (%) |
| BAPOP           | 1.11                             | 0.98                             | (C <sub>44</sub> H <sub>28</sub> N <sub>2</sub> O <sub>7</sub> ) <sub>n</sub> | Calcd:76.00        | 4.02  | 3.99  |
|                 |                                  |                                  | 701.07  | Found:76.17        | 4.07  | 3.78  |
| BAPON           | 1.46                             | 0.87                             | (C <sub>39</sub> H <sub>20</sub> N <sub>2</sub> O <sub>7</sub> ) <sub>n</sub> | Calcd:74.67        | 3.18  | 4.43  |
|                 |                                  |                                  | 632.45  | Found:74.91        | 3.12  | 4.72  |
| BAPMC           | 1.48                             | 1.01                             | (C <sub>37</sub> H <sub>28</sub> N <sub>2</sub> O <sub>7</sub> ) <sub>n</sub> | Calcd:72.70        | 4.58  | 4.54  |
|                 |                                  |                                  | 616.30  | Found:72.61        | 4.48  | 4.75  |

<sup>a</sup>in each process 0.30 g BAPMC, 0.29 g CDPA and 10 mL of DMAc were used. The concentration of 0.5 g/dL in DMAc at 25°C

<sup>b</sup>Measured at a concentration of 0.5 g/dL in H<sub>2</sub>SO<sub>4</sub> at 25°C



Scheme 2. Polyimides pathway synthesis.

viscosity of poly(amic-acid) related to polymer containing BAPMC.

Immediately after completing poly(amic-acid) the solutions should be casted into polyimide films, because water hydrolysis it. This is second-step that poly(amic-acid) was heated according to program. As the result, flexible polyimide films were obtained by casting from polymer solution. At 250°C on plate glass, the thermal conversion has happened by poly(amic-acid) to polyimide films. The polyimide from BAPON formed brown flexible and tough films. The brown color of the films is probably attributable to the formation of charge transfer complex between electron-donating BAPON unit and strongly electron-accepting phthalimide unit.<sup>[18]</sup> Rang of inherent viscosities is from 0.87 to 1.01 dL/g in concentrated sulfuric acid. Polyimide with BAPMC segment has higher inherent viscosity. Results of elemental analyses of polyimides are listed in Table 2 that agree with the calculated values based on the repeating unit of polymers.

FT-IR spectrum of polyimide was shown in Figure 2. The characteristic absorption bands of the imide ring are observed near 1779 cm<sup>-1</sup>, 1709 cm<sup>-1</sup> (asymmetrical and symmetrical C = O stretching vibration) and 1392 cm<sup>-1</sup> (C = N stretching vibration). Those of the amide and carboxyl groups in the



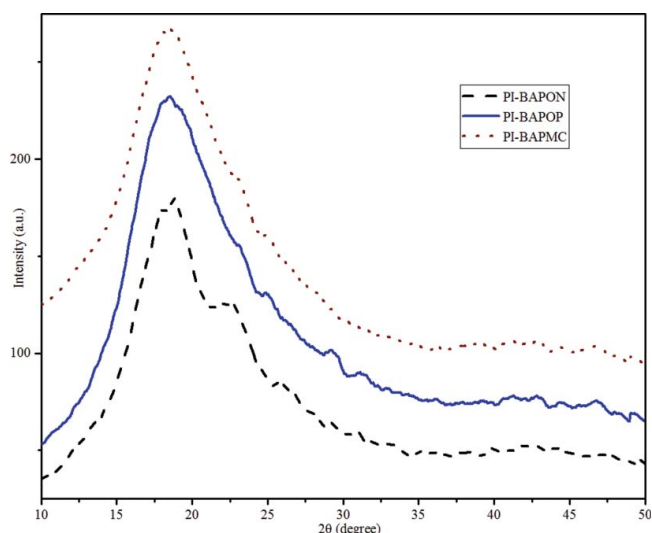


Figure 3. XRD pattern for PI-BAPON, PI-BAPOP and PI-BAPMC.

regions of  $2900\text{--}3200\text{ cm}^{-1}$  (COOH and NH<sub>2</sub>),  $1713\text{ cm}^{-1}$  (C = O (COOH)),  $1658\text{ cm}^{-1}$  (C = O (CONH)) and  $1546\text{ cm}^{-1}$  (C-NH-) disappear, indicating a virtually complete conversion of the precursor into polyimide.

### 3.2.1. Solubility and XRD pattern

Polyimides Solubility test as the qualitatively were performed in various solvents. The solubility properties of polyimides are as follow. The polyimides derived from BAPON and BAPOP almost insoluble at the various organic solvents. Despite the fact that polyimides with BAPOP segment have amorphous structure should be has solubility in amide form solvent but insoluble this phenomena may be due to partial crosslinking during the imidation. BAPMC soluble on heating at  $100^\circ\text{C}$  or boiling temperature of the amide form solvent this related to cyclohexyl group in the backbone of polyimide.

The crystallinity of prepared polyimides films was evaluated by x-ray diffraction measurement with  $2\theta$  from  $5^\circ$  to  $50^\circ$ . As shown in Figure 3, polyimide derived from BAPON showed the degree of crystalline character and exhibited two strong peak reflections at  $19^\circ$  and  $25^\circ$ . While others patterns of polyimides were interpreted as amorphous ones.

### 3.2.2. Hydrolitic stability and dielectric measurement

Moisture absorption of polyimide is determined by using difference between the pre-dried ( $W_0$ ) and wet film sample ( $W$ )

of approximately  $1 \times 1\text{ cm}$  square after soaking in the de-ionized water for 100 h:

$$\% \text{ Moisture absorption} = \frac{W - W_0}{W_0} \times 100$$

Dielectric constant ( $\epsilon$ ) of the PI films are measured by scratch coated with ultrathin gold layer vacuumed evaporation deposition for 40 s on both sides by using JFC-1200 Fine Coater (model fitted with FC-TM10 Thickness Monitor). Coated film was placed between the upper and lower thin film plate sensors and measurement was taken at the different frequency. This procedure excluded any contact problems.

As shown in Table 3, polyimide containing BAPMC segment has lower dielectric constants (2.96 at 10 kHz) than other polyimides. The decreased dielectric constants might be partly attributable to the cyclohexyl groups, which resulted in less efficient chain packing and increased free volume. The moisture absorption of the polyimides ranged from 0.75 to 0.86% is depicted in Table 3. Polyimide containing BAPMC segment in comparison, of the other polyimides exhibited lower moisture absorption (0.75%). The moisture absorption can be partly attributable to the cyclohexyl as a hydrophobic group.

### 3.2.3. Thermal properties of polyimides

Polyimide is a state of art heat-resistant engineering plastics that causes widely used in aerospace and military applications due to their thermal properties.<sup>[9]</sup> Thermal behavior of the polyimides was determined by thermogravimetric analyses (TGA) and DSC that summarized in Table 3. One step pattern of decompositions is appeared in all the polyimides with no weight loss below  $480^\circ\text{C}$  in nitrogen atmosphere. Wholly and semi-wholly polyimide have higher thermal stability this attribute to the fact that the C = C bond of the aromatic group is stronger than C-C bond of the ether-amine moiety. Initial Temperature at which 5% mass loss was recorded in the range of  $490\text{--}608^\circ\text{C}$ . The decomposition temperatures of 10% weight loss were in the range of  $500\text{--}630^\circ\text{C}$  in nitrogen and depending on the diamine used.

Depolymerization, random chain rupture and removal of side groups are three mechanisms of thermal decomposition in Polyimides, which are numerous numbers of radicals produced. Random chain breaking which is the main of decomposition could be produced by heating as much as needed. The initial fragments of

Table 3. Moisture absorption, Dielectric constant, Thermal properties data and Glass transition temperatures obtained by TGA and DSC of the Polyimides.

| Bis-amine Code | Film Thickness ( $\mu\text{m}$ ) | Moisture absorption (%) | Dielectric constant ( $\epsilon$ ) |       | Thermal properties in N <sub>2</sub> atmosphere |  |   |                             |
|----------------|----------------------------------|-------------------------|------------------------------------|-------|---|--|---|-----------------------------|
|                |                                  |                         | 10 kHz                             | 1 MHz | Tg/ $^\circ\text{C}$ <sup>a</sup>               | T <sub>initial</sub> / $^\circ\text{C}$ <sup>b</sup> | T <sub>10</sub> / $^\circ\text{C}$ <sup>c</sup> | Char yield (%) <sup>d</sup> |
| BAPOP          | 34                               | 0.80                    | 3.2                                | 3.18  | 205   | 608  | 630   | 66                          |
| BAPON          | 81                               | 0.86                    | 3.25                               | 3.20  | 262   | 500  | 510   | 65                          |
| BAPMC          | 74                               | 0.75                    | 2.96                               | 2.90  | 198   | 490  | 500   | 63                          |

<sup>a</sup>Glass transition temperatures by DSC

<sup>b</sup>Temperature at which 5% mass loss was recorded

<sup>c</sup>Temperature at which 10% mass loss was recorded

<sup>d</sup>Mass percentage of material left after TGA analysis at a maximum temperature of  $700^\circ\text{C}$

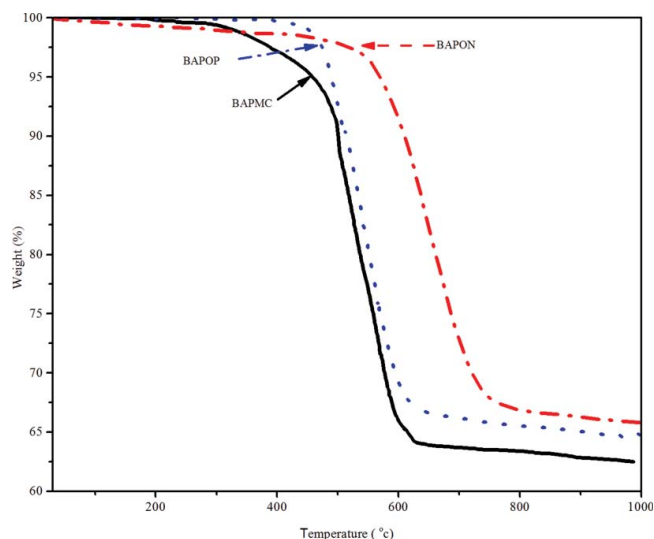


Figure 4. TGA curves of BAPON, BAPOP and BNPMC films.

thermal degradation are imide rings and ether linkages could produce CO<sub>2</sub> and CO. At the resolute, the active free radicals could recombine and cause more weight loss of PI.<sup>[21,22]</sup>

In the nitrogen atmosphere, the amount of carbon residual for all polyimides at 800°C was higher than 60% and the polyimide containing BAPON has maximum char yield (up to 66%). Figure 4 was shown TGA curves of polyimides with heating rate of 10°C/min under flowing nitrogen.

The glass transition temperature (T<sub>g</sub>) is a macroscopic expression to the beginning of the movement of molecular chain segments. It directly affects the processing performance and application of materials. Thus, we measured T<sub>g</sub> by DSC (Figure 5). Curves PI-BAPON, PI-BAPOP and PI-BAPMC show the glass transition temperatures (T<sub>g</sub>s) in the range of 198–262°C. With the increasing of aromatic content, the molecular chains contain less flexible ether linkages and incorporation -C(CH<sub>3</sub>)<sub>2</sub>- and cyclohexyl more flexible with large volume structure. In brief, the rigidity of backbone is increased, resulting in increased T<sub>g</sub>. DSC reveals both glass transition and melting of the resultant polymers (Figure 5). Melting is observed between 198–262°C, which follows the increasing order of chain rigidity

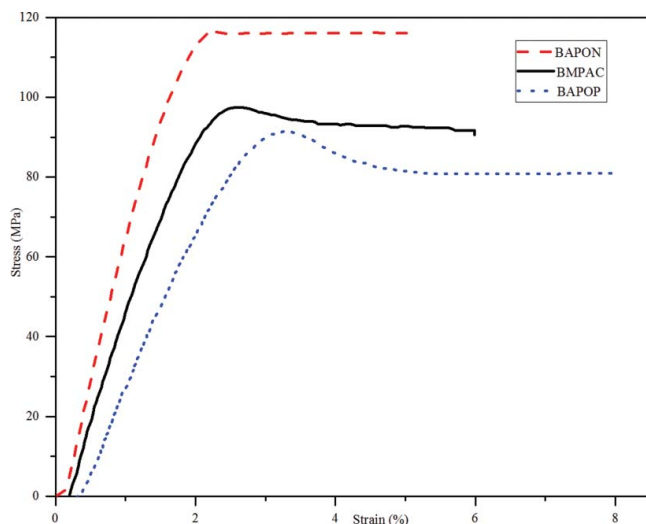


Figure 5. DSC curves of BAPON, BAPOP and BNPMC films.

Table 4. Mechanical properties of the Polyimides films.

| Bis-amine Code | Tensile strength (MPa) | Young's modulus (GPa) | Elongation at break (%) | Strain at yield point (MPa) | Toughness |
|----------------|------------------------|-----------------------|-------------------------|-----------------------------|-----------|
| BAPON          | 117                    | 2.32                  | 5                       | 2.12                        | 5.35      |
| BAPOP          | 88                     | 1.98                  | 8                       | 3.10                        | 4.62      |
| BAPMC          | 98                     | 2.11                  | 6                       | 2.6                         | 4.93      |

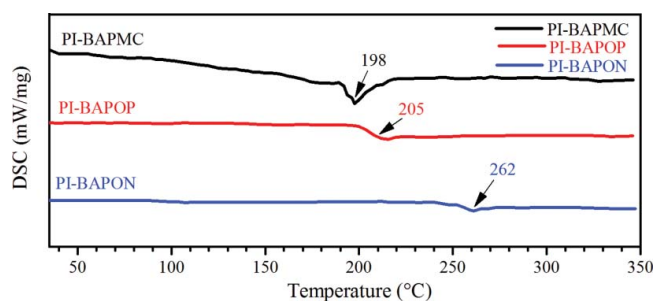


Figure 6. Stress-strain curves of BAPON, BNPMC and BAPOP films.

and polarity of the ether amines. The incorporation of aliphatic units into the PI backbone results in decreasing glass transition temperature, while incorporation of aromatic units increases the T<sub>g</sub>. These results are shown PI-BAPMC has more solubility, processability and tractability than other does.

#### 3.2.4. Mechanical properties of the PEAs polyimide

Three synthetic polyimides afforded good-quality films with a light color except BAPON that is dark color. These films were subjected to a tensile test, and their tensile properties are summarized in Table 4. Ultimate tensile strengths of films, elongations to break and initial moduli are 88–117 MPa, 5–8 and 1.98–2.32 GPa respectively.

These synthetic polyimide films exhibited a high tensile strength and moduli and low elongation, indicating that they belong to a hard and strong material. The tensile properties are shown in Figure 6. The needs of energy to rupture the films are calculated from area under the stress-strain isotherm curve and are known as toughness.

## 4. Conclusions

Synthetic diamine with high yielding is prepared directly by using two-steps procedure. Including the Williamson etherification reaction with nucleophilic substitution reaction of diol and 1-fluoro-4-nitro benzene in the presence of potassium carbonate, and reduction by catalytic reaction consequently. Three polyimides were obtained from bis(ether-amine) with CDPA by two-step chemical and thermal imidization methods. Structures of monomers and polyimides were confirmed by FT-IR and <sup>1</sup>H NMR spectroscopy and elemental analyses. The measurement of TGA and mechanical properties indicate that polyamides were thermal stable at temperatures up to 480°C. Polyimide films possessed excellent tensile properties and show typical yield behavior of thermoplastic polymers. Polyimide containing BAPMC segment has good transparency, low dielectric constant and moisture absorption that is suitable for using in optoelectronics and microelectronics.

Otherwise solubility of this polymer in organic solvent comparison others so good thermostability is introduced as one of the high performance materials.

### Compliance with ethical standards

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