

## Thermally Stable and Soluble Polyesters Derived from Naphthyl-Benzamide Diol: Synthesis and Characterization

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### ABSTRACT

A novel diol, 3,5-dihydroxy-N-(naphthalen-8-yl)benzamide, was successfully synthesized via Yamazaki condensation between 1-naphthylamine and 3,5-dihydroxybenzoic acid in the presence of triphenyl phosphate and pyridine. The structure of the synthesized diol was confirmed through Fourier transform infrared spectroscopy (FT-IR), proton nuclear magnetic resonance, and carbon-13 nuclear magnetic resonance spectroscopy. This diol was subsequently polymerized with varying molar ratios of terephthaloyl chloride and isophthaloyl chloride to produce a series of polyesters (PE-1–PE-5) using a phase transfer catalyst system. The resulting polyesters were obtained in good yields, with inherent viscosities ranging from 0.40 to 0.88 dL/g. FT-IR analysis confirmed the successful formation of ester linkages. The polyesters demonstrated excellent solubility in polar organic solvents, attributed to the presence of bulky naphthalene pendant groups and polar amide linkages. Wide-angle X-ray diffraction patterns indicated low crystallinity. Thermal analysis via thermogravimetric analysis and differential scanning calorimetry revealed that the polyesters possessed high thermal stability, with decomposition temperatures reaching up to 488°C and glass transition temperatures in the range of 123–179°C. These findings suggest that the integration of naphthalene and benzamide units significantly improves the thermal properties and solubility of the polyesters, making them promising candidates for high-performance material applications.

**Key words:** Aromatic polyesters, Fourier transform infrared spectroscopy, Naphthalene-based diols, Nuclear magnetic resonance spectroscopy, Phase transfer catalysis, Solubility, Thermal stability, Yamazaki condensation

### 1. INTRODUCTION

Aromatic polyarylates are important class of high-performance engineering material due to good thermal and mechanical properties [1-3]. However most of these polyarylates shows high melting temperatures or glass transition temperatures (T<sub>g</sub>) and limited solubility in many organic solvents, as results leading to difficulties in processing [4-12]. Therefore, more effort have been made to overcome this drawback without lowering their thermal properties, bulky units were introduced into the polymer main chain, has been shown to impart greater solubility and enhanced rigidity, as well as better mechanical and thermal properties of the resulting polyarylates [7,13-20]. It has been reported that the presence of bulky units, such as binaphthalene units, tetraphenylfuran, phthalide, phthalimidine, tetraphenylthiophene, and anthrone, in the repeat unit of polyester results in enhanced solubility as well as higher thermal properties of polymer [21-25].

Present work deals with the synthesis of diol, 3,5-dihydroxy-N-(naphthalen-8-yl)benzamide derived from 1-naphthyl amine and 3,5-dihydroxy benzoic acid by Yamazaki phosphorylation condensation reaction. Polyesters were prepared through interfacial polymerization reaction of diol, 3,5-dihydroxy-N-(naphthalen-8-yl)benzamide with isophthaloyl chloride (IPC) and terephthaloyl chloride (TPC) (different molar ratio) in the presences of phase transfer catalyst benzyl triethyl ammonium chloride. These polyesters containing pendant naphthyl unit into the polymer chain have been shown to enhanced thermal stability as well as solubility in organic solvents. The structure of synthesized aromatic diol, 3,5-dihydroxy-N-(naphthalen-8-yl)benzamide was

investigated by infrared (IR) and nuclear magnetic resonance (NMR) spectroscopy. The prepared polyesters from diol, 3,5-dihydroxy-N-(naphthalen-8-yl)benzamide and IPC/TPC were characterized by IR, NMR spectroscopy and physical properties of polyesters, including inherent viscosity, solubility, thermal behavior, and X-ray diffraction.

The present studies describe a successful synthesis of a monomer containing naphthyl moieties and preparing related polyesters by the interfacial polymerization reaction of this diol with different diacid chlorides.

### 2. EXPERIMENTAL

#### 2.1. Materials

1-naphthyl amine, methanol, anhydrous calcium chloride, 3,5-dihydroxy benzoic acid, lithium chloride, pyridine, N-methyl-

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2-pyrrolidone (NMP), triphenyl phosphate, sodium bicarbonate, IPC and terephthaloyl chloride (TPC), benzyl triethyl ammonium chloride.

## 2.2. Measurements

Synthesized diol and polyesters were characterized by spectroscopy and Fourier transform infrared spectroscopy (FT-IR) spectra were recorded on a Nicolet spectrometer using KBr pellets and NMR spectrum were obtained from Bruker Advance spectrometer at resonance frequency 400 MHz spectrometer for proton nuclear magnetic resonance ( $^1\text{H}$  NMR) and 100 MHz for carbon-13 nuclear magnetic resonance ( $^{13}\text{C}$  NMR) at  $25^\circ\text{C}$  using TMS as an internal reference. Inherent viscosities of polyesters were measured with concentration of 0.5 g/dL in NMP solvent at  $30^\circ\text{C}$  using an Ubbelohde suspended level viscometer. Differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) analysis was recorded on a Mettler Toledo DSC STARe instrument at heating rate of  $10^\circ\text{C}/\text{min}$  under nitrogen. Wide angle X-ray diffraction (WAXD) pattern obtained from Rigaku X-ray diffractometer using polyester powder.

## 2.3. Diol synthesis

### 2.3.1. 3,5-dihydroxy-N-(naphthalen-8-yl)benzamide

In 100 mL three necked round bottom flask equipped with a magnetic stirrer, a calcium chloride guard tube, a reflux condenser were charged with 4.20 g (0.027 mol) of 3,5-dihydroxy benzoic acid, 3.90 g (0.027 mol) of 1-naphthyl amine, lithium chloride (3.10 g), pyridine (7.75 mL), NMP (32 mL), triphenyl phosphate (11 mL) as condensing agent. The reaction mixture was heated at  $110^\circ\text{C}$  under nitrogen atmosphere for 14 h. Reaction mixture was cooled at room temperature and poured into cold water. The precipitated product was collected. The residue was continuously washed with sodium bicarbonate solution followed by hot water dried in vacuum for 8 h at  $80^\circ\text{C}$ . The residue was recrystallized from alcohol to get pure 3,5-dihydroxy-N-(naphthalen-8-yl)benzamide.

Yield: 4.46 g (94%), M.P.:  $160^\circ\text{C}$

IR:  $3361\text{ cm}^{-1}$  (-OH stretch),  $2924\text{ cm}^{-1}$  (amide stretch),  $1691\text{ cm}^{-1}$  (carbonyl stretch)

$^1\text{H}$  NMR (DMSO, 400MHz)  $\delta$  (ppm): 6.42 (s, 1H), 6.9 (s, 2H), 7.3 (d, 3H), 7.4 (d, 2H), 7.6 (d, 1H), 7.9 (d, 1H), 9.1 (s, 2H), 9.7 (s, 1H).

$^{13}\text{C}$  NMR (DMSO, 400MHz)  $\delta$  (ppm): 106.18, 106.37, 123.14, 123.43, 125.60, 125.94, 126.04, 126.26, 128.23, 129.24, 133.89, 134.11, 136.75, 158.66, 167.14.

## 2.4. Polymerization

A 100 mL three necked round bottom flask equipped with a magnetic stirrer and a calcium chloride guard tube were charged with 0.371 g (0.001 mol) 3,5-dihydroxy-N-(naphthalen-8-yl)benzamide, sodium hydroxide 0.080 g (0.002 mol) and benzyl triethyl ammonium chloride used as phase transfer catalyst 0.100 g and 12.5 mL distilled water the whole reaction mixture was stirred rapidly then introduced IPC 0.203 g (0.001 mol) in dichloromethane 8 mL. The stirring was vigorously continued at  $30^\circ\text{C}$  for an hour. The resulting viscous polyester solution was poured slowly into excess hexane to form polyester precipitate that was collected by filtration, washed thoroughly with water and dried under vacuum at  $80^\circ\text{C}$  for 10 h. Yield: 95%, the polyester exhibited inherent viscosity 0.45 dL/g. The other polyester was prepared by similar procedure with changing IPC/TPC mole ratio solvent concentration temperature. The yield of prepared polyesters was above 92%.

## 3. RESULTS AND DISCUSSION

### 3.1. Diol Synthesis

Synthesis of diol, 3,5-dihydroxy-N-(naphthalen-8-yl)benzamide from starting material 1-naphthyl amine and 3,5-dihydroxy benzoic acid which undergo Yamazaki condensation reaction in the presences of triphenyl phosphate as condensing agent and base pyridine to afford 3,5-dihydroxy-N-(naphthalen-8-yl)benzamide. The chemical structure and purity of prepared diol, 3,5-dihydroxy-N-(naphthalen-8-yl)benzamide was confirmed by FT-IR,  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR. In FT-IR spectrum of diol [Figure 1], the characteristics band of hydroxy group and amide group was observed at  $3361\text{ cm}^{-1}$  and  $2924\text{ cm}^{-1}$ . In  $^1\text{H}$  NMR spectrum the characteristic proton resonance peak of -OH and amide group observed at 9.1 ppm (b) and 9.76 ppm (d), the aromatic proton para to amide group and ortho to hydroxyl group resonance at 6.3 ppm (a) in upfield region due to more shielding character. All aromatic proton resonance peaks were observed in the range between 6.4 ppm to 7.9 ppm [Figure 2]. The  $^{13}\text{C}$  spectrum of 3,5-dihydroxy-N-(naphthalen-8-yl)benzamide showed 15 different signal corresponding to 15 non-equivalent carbons. The signal at 167.14 ppm (e) is assigned to carbonyl carbon of amide group and the signal observed at 158.66 ppm (b) is assigned to hydroxyl carbon of aromatic ring. All aromatic protons resonance peak observed in the range between 106.18 ppm to 136.75 ppm [Scheme 1 and Figure 3].

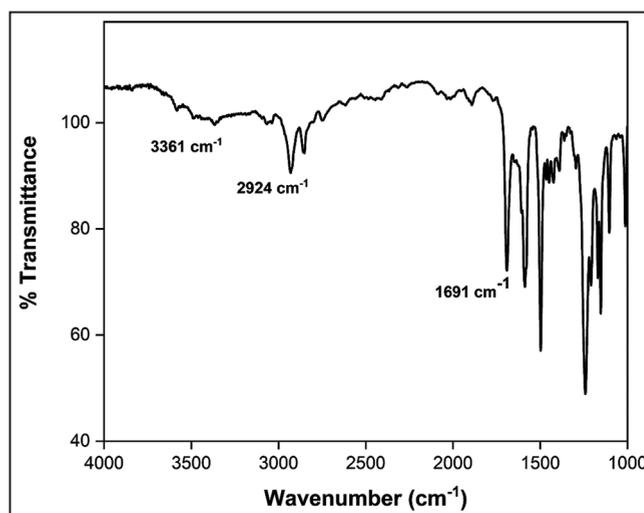
### 3.2. Synthesis of Polyesters

In this study series of (PE-11–PE-15) were synthesized by polycondensation of 3,5-dihydroxy-N-(naphthalen-8-yl)benzamide with different molar ratio of TPC and/or IPC in dichloromethane and aqueous sodium hydroxide system. In this reaction benzyl triethyl ammonium chloride was used as phase transfer catalyst. The physical properties of prepared polyesters are summarized in Table 1. All the polyesters were obtained in almost quantitative yields with inherent viscosity were in the range of 0.40–0.88 dL/g [Scheme 2 and Table 1].

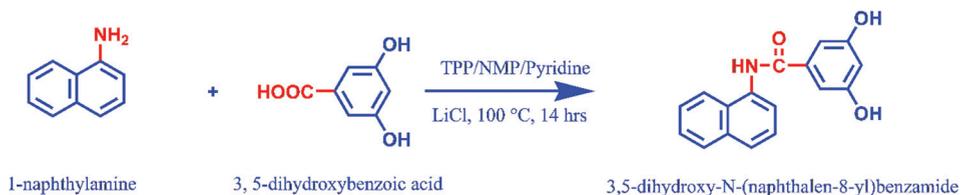
Structural characteristic of the polyesters were confirmed by FT-IR spectra. They showed the characteristic absorptions band of the ester group at around  $3216\text{ cm}^{-1}$  (ester carbonyl stretching).

### 3.3. Solubility of Polyesters

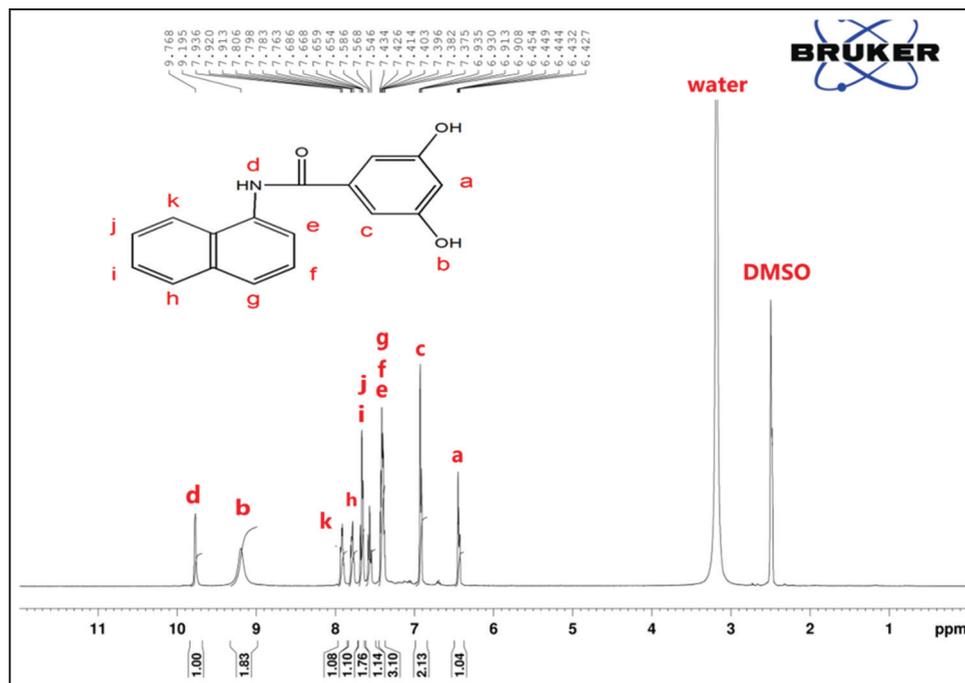
Synthesis of modified polyesters having improved solubility was the main objective of our work. The solubility of the polyesters



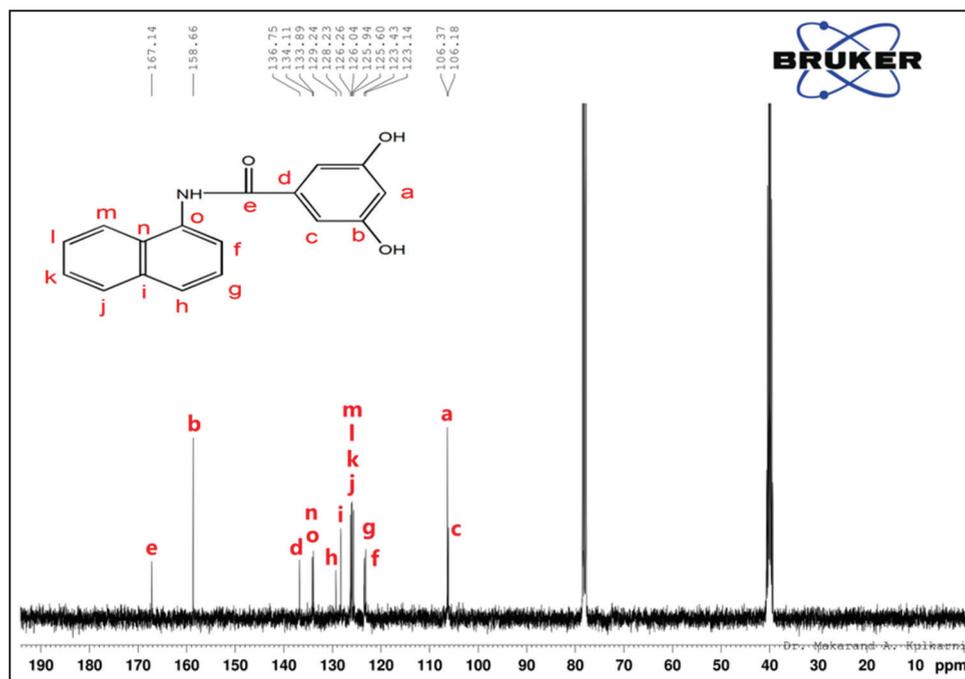
**Figure 1:** Fourier transform infrared spectroscopy spectrum of 3,5-dihydroxy-N-(naphthalen-8-yl)benzamide.



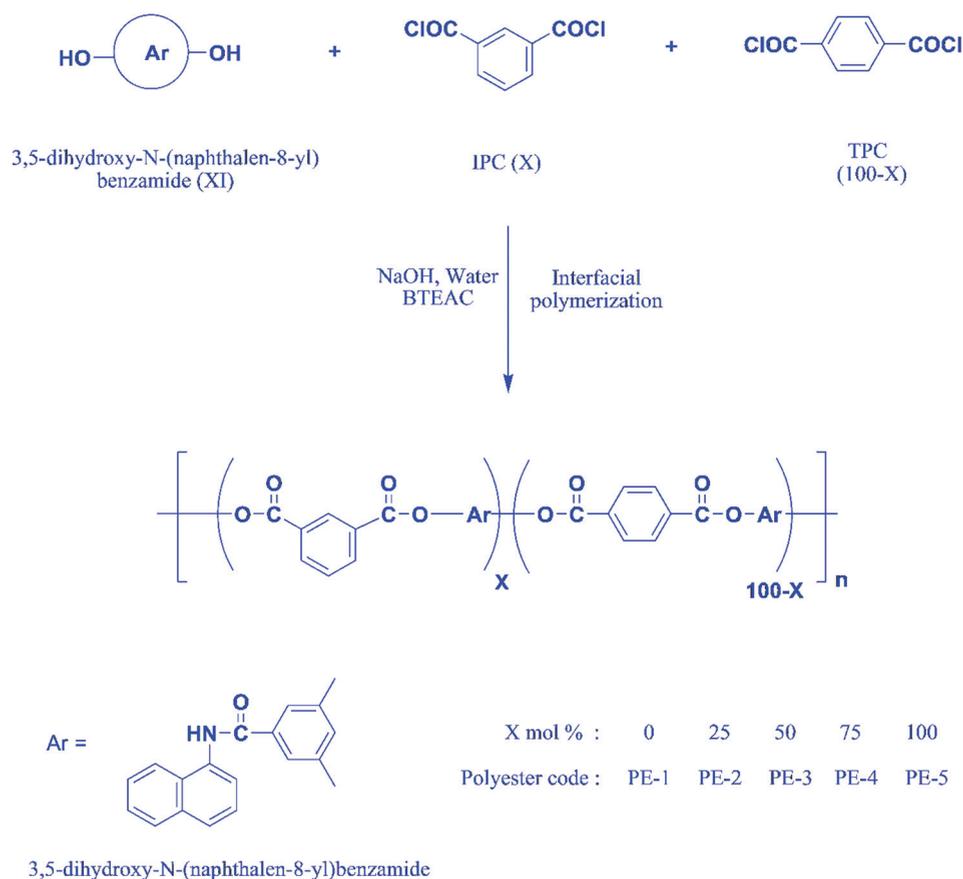
**Scheme 1:** Synthesis of 3,5-dihydroxy-N-(naphthalen-8-yl)benzamide.



**Figure 2:** Proton nuclear magnetic resonance spectrum of 3,5-dihydroxy-N-(naphthalen-8-yl)benzamide.



**Figure 3:** Carbon-13 nuclear magnetic resonance spectrum of 2,4-dihydroxy-N-(naphthalen-8-yl)benzamide.



**Scheme 2:** Synthesis of polyesters from 3,5-dihydroxy-N-(naphthalen-8-yl)benzamide and IPC/TPC by interfacial polycondensation method.

**Table 1:** Yield and inherent viscosity of polyesters<sup>a</sup> (PE-1–PE-5) from diol<sup>b</sup> and diacid chloride

S. No.	Polyester code	Diacid chloride (mol%)		Yield (%)	Inherent Viscosity <sup>c</sup> $\eta_{inh}$ (dL/g)
		IPC	TPC		
1.	PE-1	100	00	91.4	0.58
2.	PE-2	75	25	90.7	0.46
3.	PE-3	50	50	92.2	0.48
4.	PE-4	25	75	90.6	0.52
5.	PE-5	00	100	91.8	0.40

<sup>a</sup>Polymerization was carried out with 1 mmol each of 3,5-dihydroxy-N-(naphthalen-7-yl) benzamide and different mol proportion of diacid chloride. <sup>b</sup>3,5-dihydroxy-N-(naphthalen-7-yl) benzamide. <sup>c</sup>Measured with a 0.5% (w/v) polyester solution in NMP at 30°C

was determined qualitatively in various organic solvents, such as dimethyl sulfoxide (DMSO), 1-methyl-2-pyrrolidone (NMP), dimethylformamide (DMF), pyridine, m-cresol, chloroform, dichloromethane, tetrahydrofuran etc. the obtained results are summarized in Table 2. The solubility of polyesters improved was attributed due to the introduction bulky pendant naphthalene moiety and amide polar group into the polymer chain.

The WAXD X-Ray diffraction patterns of polyesters (PE-1–PE-5) was in the region of  $2\theta = 5-50^\circ$  as represents in Figure 7. The bulky pendant naphthyl benzamide moiety and amide linkage decreases

**Table 2:** Solubility of polyesters (PE-1–PE-5)

Polyester→Solvent ↓	PE-1	PE-2	PE-3	PE-4	PE-5
NMP	++	++	++	++	++
DMF	++	++	++	++	++
DMSO	++	++	++	++	++
DMAc	++	++	++	++	++
THF	++	++	++	++	++
m-Cresol	++	++	++	++	++
DCM	++	+-	+-	++	++
CHCl <sub>3</sub>	+-	+-	+-	+-	+-
H <sub>2</sub> SO <sub>4</sub>	++	++	++	++	++

++: Soluble, +-: Partially soluble, --: Insoluble

disrupting chain regularity and packing. This could be attributed to low crystallinity nature of polyesters shows good solubility in common organic solvents.

### 3.4. Thermal Properties of Polyesters

Thermal studies of polyesters (PE-1–PE-5) were evaluated by TGA and DSC in nitrogen atmosphere with heating rate 10°C/min. Figures 5 and 6 shows the TGA and DSC thermograms of PE-1–PE-5. The thermal data of polyesters PE-1–PE-5 are summarized in Table 3. The TGA curves of PE-1–PE-5 shows initial decomposition temperature were in the range of 150–195°C. The temperatures for 10% weight loss were in the range of 324–488°C. The char yields at 900°C

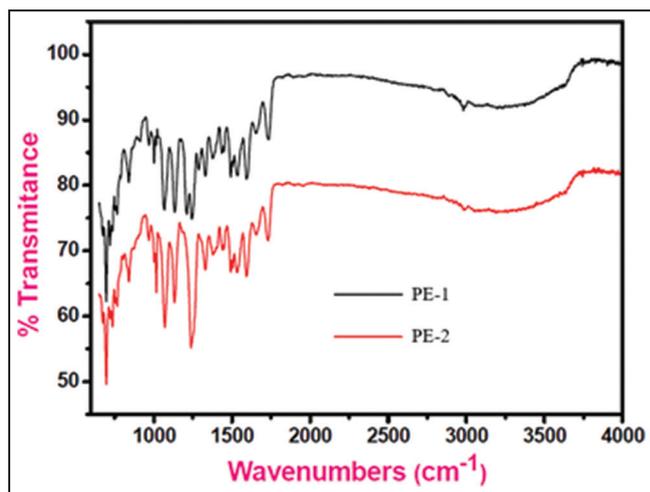


Figure 4: Fourier transform infrared spectroscopy spectrum of polyesters.

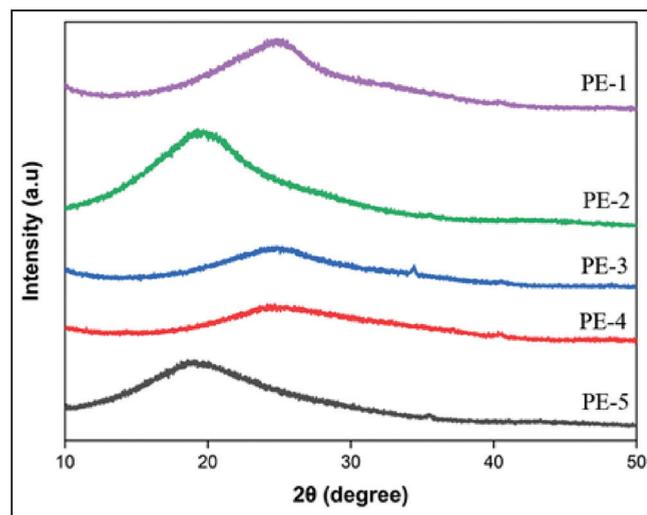


Figure 7: X-ray diffraction curves of polyesters, PE-1–PE-5.

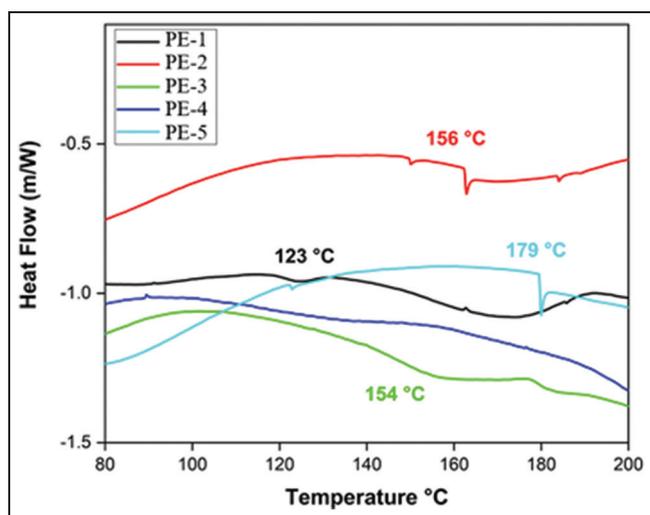


Figure 5: Differential scanning calorimetry curves of polyesters, PE-1–PE-5.

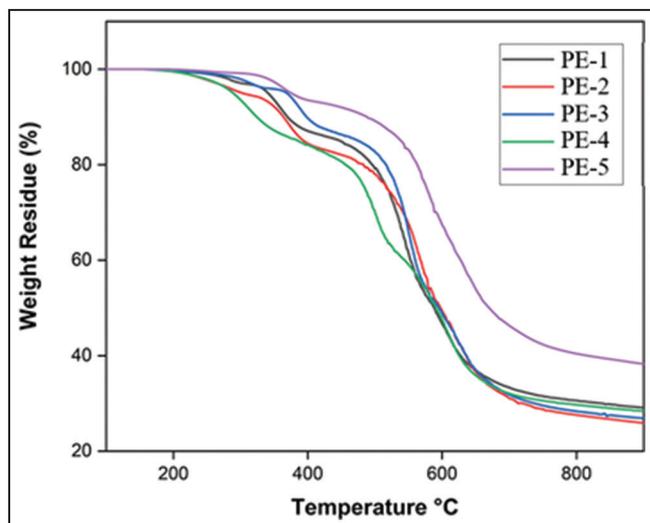


Figure 6: Thermogravimetric analysis curves of polyesters, PE-1–PE-5.

Table 3: Thermal properties of polyesters (PE-1–PE-5)

Polyester code	<sup>b</sup> T <sub>i</sub> (°C)	<sup>c</sup> T <sub>10%</sub> (°C)	<sup>d</sup> T <sub>g</sub> (°C)	Residual wt. % at 900°C
PE-1	150	370	123	29
PE-2	172	364	156	25
PE-3	175	398	154	26
PE-4	150	324	138	28
PE-5	195	488	179	38

<sup>a</sup>Thermogravimetric analysis was conducted at a heating rate of 10°C/min. Under nitrogen atmosphere. <sup>b</sup>T<sub>i</sub>: Temp at which weight loss initiated. <sup>c</sup>T<sub>10%</sub>: Temp at which 10% weight loss was observed. <sup>d</sup>T<sub>g</sub>: Determined by DSC measured at a heating rate 20°C/min. under nitrogen atmosphere. LOI: Limiting oxygen index

for these PE-1–PE-5 polyesters were in the range of 25–38%. Figure 4 shows polyesters PE-1–PE-5 have good thermal stability which may be attributed to stability of naphthalene moiety, amide linkages and ether linkages in these polyesters.

The glass-transition temperatures (T<sub>g</sub>) of polyesters determined by using DSC thermograms. The T<sub>g</sub> of naphthyl unit containing polyesters PE-1–PE-5 were in the range of 123–179°C. All these polyesters shows lowest T<sub>g</sub> due to the presence of benzamide linkage in the main chain of a given polymer lowers the rigidity of its backbone and lower the T<sub>g</sub> values.

#### 4. CONCLUSION

A novel diol, 3,5-dihydroxy-N-(naphthalen-8-yl)benzamide, was successfully synthesized via Yamazaki condensation and characterized using FT-IR, <sup>1</sup>H NMR, and <sup>13</sup>C NMR spectroscopy. A series of aromatic polyesters (PE-1–PE-5) were synthesized through interfacial polycondensation of the diol with various molar ratios of TPC and IPC. The resulting polyesters exhibited excellent solubility in common organic solvents, which can be attributed to the presence of bulky pendant naphthyl and polar amide groups disrupting regular chain packing and crystallinity, as confirmed by WAXD patterns.

Thermal analysis revealed that these polyesters possess good thermal stability with decomposition temperatures ranging from

324–488°C and char yields up to 38%. The T<sub>g</sub> were found between 123–179°C, indicating moderate rigidity of the polymer backbone. The lower T<sub>g</sub> values are associated with the flexible benzamide linkages.

Overall, the introduction of naphthyl and amide moieties into the polyester backbone not only enhanced solubility but also provided thermal robustness, making these materials promising candidates for high-performance, solution-processable polymer applications.

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