# Preparation, Characterazation, and Thermal Properties of Newpoly (ester-imid)sderived from Poly condensation Reaction between Diimide-dicarboxylic Acid with Aromatic Diols

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#### Abstract

Polymeric systems based on poly (ester-imide)s (PEIs) are high-performance material, which combines the useful properties of polyester and polyimide resins. A chain of new poly(ester-imid) are obtained through polycondensation between diimide-dicarboxylic acid( Prepared from reaction pyromellitic anhydride(PMDA) with diamine) with aromatic diols such as Bis(2-bromo 4-hydroxy phenyl) cyclohexane (M1)and Bis(4-hydroxy phenyl)-2-hydroxy phenyl)methane(M2) in the presence of dibutyltine dilaurate as Catalyst, The corresponding new poly(ester-imid) (PEIs) thataregained in quantitative products are soluble in polar aprotic solvents, are ofhigh thermal stability. The structures of the Monomers and poly(esters-imide) were confirmed by FTIR and 1HNMRspectroscopies.

Keywords: Poly (esters-imide), Diamine, Pyromellitic Anhydride (PMDA), Polycondensation, Dicarboxylic Acid.

#### Introduction

Because of the growing needs for polymers of high-performance as a substitution for metals and ceramics in the aerospace, microelectronic and automotive products, polymers that are heating stable are gained a lot of attention during ten years. Polyimides and their copolymers are the most beneficial types of high performance, which are used in many applications in the industry [1-2]. Polyimides have attracted a lot of interest because of their extraordinary thermal stability, good mechanical and electrical characteristics [3]. Nevertheless, the hard task lies in its processing, resulted from poor flow features and infusibility, has limited their applications. The preraration of poly (ester-imide)s was evolved to solve such complications [4]. Employment of flexible ester association can decrease the heat of processing and enhance the flow property instead of the thermal stability.

Moreover, a lot of poly (ester-imide)s have been stated to show liquid crystalline phases [5-8]. Amongst them, poly (ester-imide) (PEIs) draw scientificattentionbecause they include good mechanical characteristicsof polyimides and the biodegradability of polyesters [9]. PEAs have various applications as single-use bags, agricultural films, drug carriers, and matrix resins for biomedical materials [10-13]. Poly (ester imide)s are researched only in the narrow viewpoints of film basic properties like thermal stability and solubility, and mechanical characteristics,[14]. Many of the formerly stated poly(ester imide)s were synthesized through direct polymerization of polyester from imide-containing di carboxylic acid with diols without the pathway of thermal imidization that plays animportant role for inplane Orientation[15].This spontaneous work includes preraration of new poly(ester-imide)(PEIs) from reaction diols such as (M1.M2) with diimidedicarboxylic acid such as compounds (M5,M6) in the presence of dibutyltine dilaurate and qualitative analysis of the monomers and polymers are achieved through FT-IR, and<sup>1</sup>HNMR spectroscopies, thermal stability have been thoroughly examined by (TGA).

## Experimental

### Materials

Dichloromethane. Chloroform. Cvclohexane. Sodium hydroxide, acetic acid ,pyridine ,methanol, and Nitric acid all from (BIOSOLVE); Hydrochloric ,Salicylaldehyde, 3-bromophenol, acid phenol Furfural, Hexane, pyromellitic anhydride, 4bromobenzaldehyde, Aniline, 2-chloroaniline, 3bromoaniline. Pvridine. acetic acid. dibutyltinedilaurate, Dimethyl Sulphoxide (DMSO), Tetrahydrofurane(THF), and thioglycolic acid all from(MERCK company ); N, N-Dimethylformamide (ALDRICH company), Ethanol (Scharlab S.L company).

The instruments: Its showed in the table (1):

Table 1: The Instrumentsused in the Study

Instrument	Company	The state
FTIR 8400S, Fourier Transform infrared spectrophotometer	SHIMADZU	Japan
Oven	Trip International Corp	Italy
Hot plate stir	BibbyStrlintd	UK
Thermogravimetric analysis	Polymer	England
(TGA)	laboratories Co	
Stuart smp30	Measuring the	
	degree of fusion	
Shiel 300MHZ	recorded NMR spectra	Switzerland
Land by using (DMSO-d6)	University's	Tehran
	Educational teacher	Iran
Model PL-TG	Iran polymer & petrochemical institute	Iran

Preraration of monomer

Bis(2-bromo 4-hydroxy phenyl) cyclo hexane) (M1) Amixture of each following:

- 1- 3-bromo phenol 4gramm (42mmol).
- 2- cyclohexanone2.72gram(16mmol).
- 3- thioglycolic acid(0.5gram)
- 4- HCl catalyst (37.5gram)

All the pervious compounds kept at the flask and made mechanical stirrer and heating at (60) °C for six hours. Then the mixture should be cold to stop the reaction. Follow washing and drying [16]. Yellow crystals are (85) % of the products, M.P is (187-190)  $^{\circ}C$ .

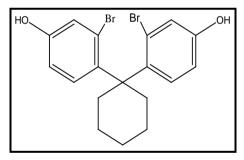
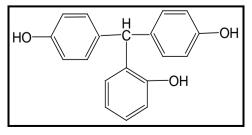


Figure-1: Structure of M1

Bis (4-hydroxy phenyl) -2-hydroxy phenyl)methane (M2)

To a 0.1M solution of H2SO4 in salicylaldehyde (1.5gram) (12mmol), phenol (21mmol) (2gram), methanol (60- 40) %, and salicylaldehyde (1.5gram) (12mmol) aremixed for ten hours at (120) °C with the continuous stirring. After completing the reaction, the solution is distance beneath vacuum (17). Brown crystals (71) % is Products, M.P= (80°C) crystals.



Bis (4-amino 2-chloro phenyl)4-bromo phenyl methane (M3)

To a 0.1M solution of H2SO4 in methanol (60-40) %, 2-chloro Aniline (21mmol) (2gram) and4-Bromo benzaldehyde (1.5 gram) (12mmol) arejoined, and the combination was refluxed for ten hours at 120°C with the continuous mixing. After completing the reaction, the solution was distanced beneath vacuum [17]. The producttook the form of (71%) brown crystals, M.P=  $(230^{\circ}C)$  crystals.

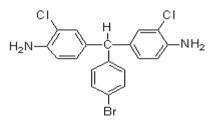


Figure-3: M3 (BACPBPM) structure

Themonomer4, 4'-(furan-2-ylmethylene)dianiline(M4)synthesized by the similar method that (M3)is made, but by employing ingredients such as Aniline, and Furfural with the mixture of methanoland H<sub>2</sub>SO<sub>4</sub> as a catalyst.

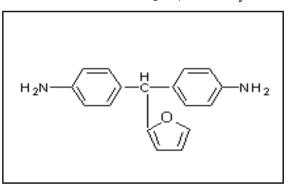


Figure-4: Structure of M4 (BAPFM)

Table-2: Properties of aromaticdiamines

N O	Mono mer	Substance		Col our	Yiel d%	М. Р/°с
1	BACP BPM	and4- Bromo benzalde hyde (1.5gm,1 2mmol)	2- chloro Aniline (2gm,2 1mmol )	Bro wn	71 %	230 °C
3	FMD A	Furfural 1.5gm(15 mmol)	Aniline 3gm(32 mmol)	Bla ck	90 %	200 °C

Preraration of dicarboxylicacid compounds

Preraration of 2, 2'-(((4-bromophenyl) methylene) bis (3-chloro-4,1-phenylene)) bis (1,3dioxoisoindoline-5-carboxylicacid (M5)

In a 250 mL round-bottomed flask are joined (20 mmol) (3.84 gram) of pyromellitic anhydride,

Figure-2: Structure of M2

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2gm(4mmol) of (BACPBPM), acetic acid (80) ml and pyridine (3:2), and a moving bar. The combination was mixed at room temperature overnight and then refluxed for 4 hours. The solution was distanced beneath condensed pressure, and the remainder was melted in 100 mL of cold water. Then 5 Ml of concentrated HCl was joined. The solventwas moved till a yellow precipitate was shaped, and the precipitate was filtered off and dried [18] to yield4.95 g (90.3%) of the yellow crystal composite.

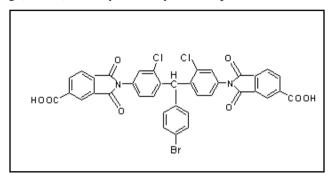


Figure-5: Structure of (M5)

Thedicarboxylicacid2, 2'-((furan-2-ylmethylene) bis (4,1-phenylene)) bis(1,3-dioxoisoindoline-5carboxylic acid) (M6) synthesized in the same method above using materials BAPFM, pyromellitic anhydride acetic acid and pyridine.

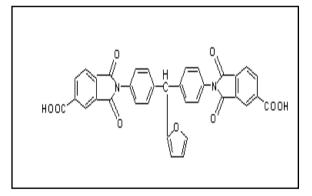


Figure-6: Structure of(M6)

Table-3: Diacids properties

NO	Dicarboxylicacids	Color	Yield%	<b>M.P</b> /°c
1	M5	Brown	61%	396°C
2	M6	yellow	70%	375°C

Preraration of poly (ester-imide)s

As shown in (Table3), Dihydroxy monomer and Di Carboxylic acidareblended at  $120^{\circ}$ C beneath continuous stirring.

When the acid contents are dissolution imide in Dihydroxy monomers, the dibutyltinedilurate (0.15 wt (%) are joined, and the temperature was raised to  $160^{\circ}$ C for 1hour. The constituents are dried beneath the vacuum[19].

**Table-4:** Properties of poly (ester-imide) [PEI<sub>1</sub>-PEI<sub>2</sub>]

polyester	Mor	10mers	Diacid	Dihydroxy	Yield	Color
polycsici	Diacid	Dihydroxy	gm/mmol	gm / mmol	(%wt.)	COIOI
P.E1	M5	M1	1.2gm	1gm	88%	white
P.E2	M6	M2	2.4gm	1.3gm	85%	Orang

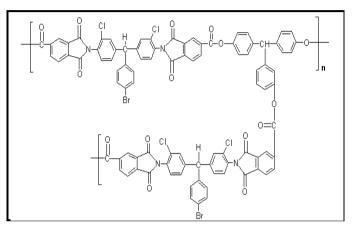


Figure-7: structure of PEI2

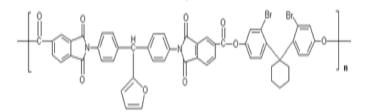


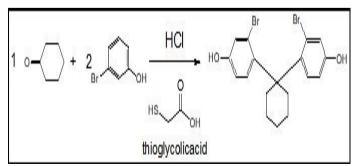
Figure-8: Structure of PEI2

**Results and Discussion** 

Preraration of monomers

Preraration and characterization (M1)

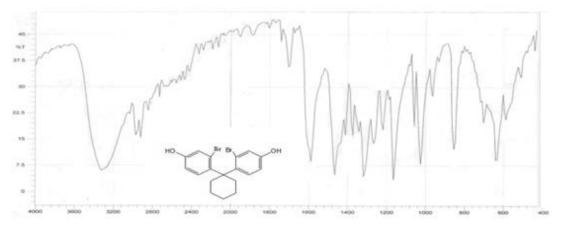
Cyclohexanon (One molecule) with 3-bromo phenol (two molecules) will produce monomer in the existence of thioglycolic acid and acid as a catalyst for six hours at  $60^{\circ}$ C. It's defined by FTIR.



**Scheme-1**: Preraration of (M1) The FTIR spectra of (M1) as displayed in a table (5):

Spectra of (M1)	absorption band	
c-c aromatic	1589cm <sup>-1</sup>	
C-H Aromatic	3031 cm <sup>-1</sup>	
ОН	3286 cm <sup>-1</sup>	
C-H aliphatic	2970 cm <sup>-1</sup>	
C-0	1164 cm <sup>-1</sup>	
C-Br	656cm <sup>-1</sup>	

Table-5: FTIR spectra of (M1) and absorption band



### Figure-9: FTIR spectra of (M1)

Η

2

0

1

Preraration and Characterization of (M2)

This monomer is made through the concentration of salicylaldehyde (one molecule) with phenol(two molecules) in the company of  $H_2SO_4$  and methanol as catalysts at 120 °C for 10 hours. It's defined by FTIR.

(60% H<sub>2</sub>SO<sub>4</sub>:40%Methanol

120 °C / 10h

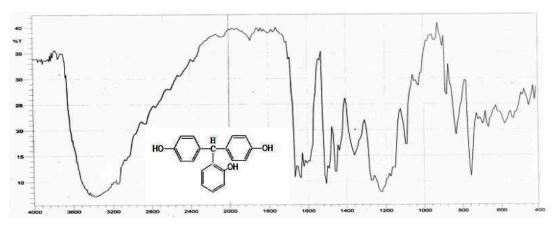


M2 Characterization and Preraration M2 FTIR spectra as a revealedtable (6)[20].

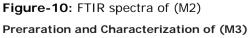
 $M_2$  FTIR spectra as a revealed table (6)[20].

Table-6: M2FTIR spectra and absorption band

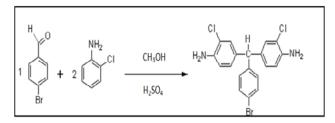
The FTIR spectra	absorption band
C=C Aromatic	(1666) cm <sup>-1</sup>
C-H Aromatic	(3062) cm <sup>-1</sup>
OH	(3379) cm <sup>-1</sup>
C-H aliphatic	(2862) cm <sup>-1</sup>
C-0	(1095) cm <sup>-1</sup>



ΩН

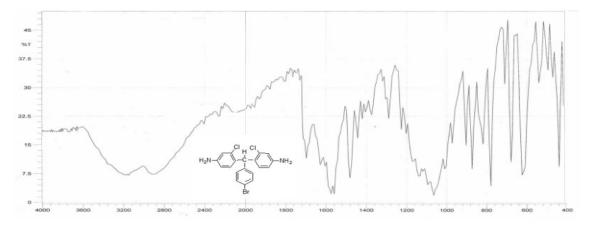


This monomer was made by the concentration4bromo benzaldehyde (one molecule) and 2-Chloro aniline (two molecules) in the company of H2SO4and methanol as catalysts at 120  $^{\rm O}$ C for 10 hours. It's defined by FTIR.



Scheme-3: Preraration of (M3) Characterization of [M3] FTIR spectrum

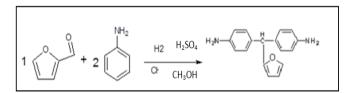
FTIR spectrum	absorption band	
(C=C Aromatic),	1579) cm <sup>-1</sup>	
(C-H Aromatic),	(3116) cm <sup>-1</sup>	
(NH2),	(3300) cm <sup>-1</sup>	
(C-H aliphatic),	(2862) cm <sup>-1</sup>	
(C-N)	(1226) cm <sup>-1</sup>	
C-Cl,	(779) cm <sup>-1</sup>	
C-Br	(672) cm <sup>-1</sup>	



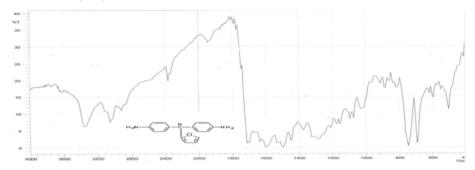
## Figure-12: FTIR spectra of (M4)

## Preraration and Characterization of (M4)

The monomer produced from Furfural (one molecule) and the aniline (two molecules) in the attendance of  $H_2SO_4$  and methanol as catalysts at 120  $^{\rm O}C$  for 10 hours. It's defined by FTIR.



## Scheme-4: (M4) Preraration



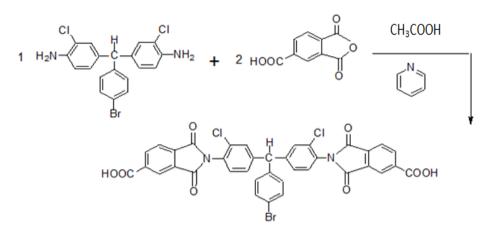
## Figure-13: (M4) FTIR spectra

Dicarboxylic acid Preraration and Characterization (M5) Preraration and Characterization The monomer acid was made by (BACPBPM) (1 molecule) and pyromelliticdianhydride

Table-8: FTIR spectra of (M4) and absorption band

The FTIR spectra	absorption band
C=C Aromatic	(1597) cm <sup>-1</sup>
C-H Aromatic	(3055) cm <sup>-1</sup>
NH2	(3355) cm <sup>-1</sup>
C-H aliphatic	(2916) cm <sup>-1</sup>
C-N	(1280) cm <sup>-1</sup>
C-0	(1175) cm <sup>-1</sup>

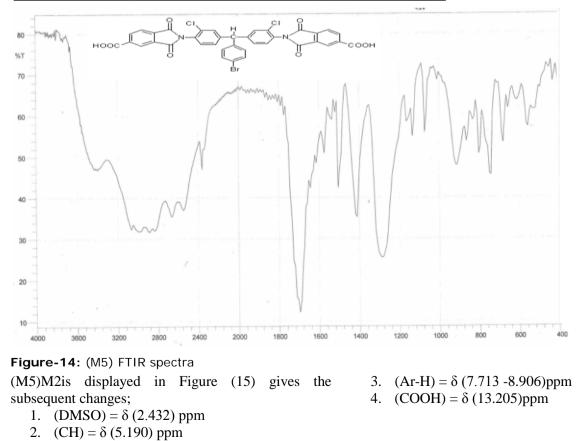
(2molecules) with the present of pyridine and acetic acid at 25 C for 24 hours then refluxed for 4 hours. This composite was defined by FTIR.

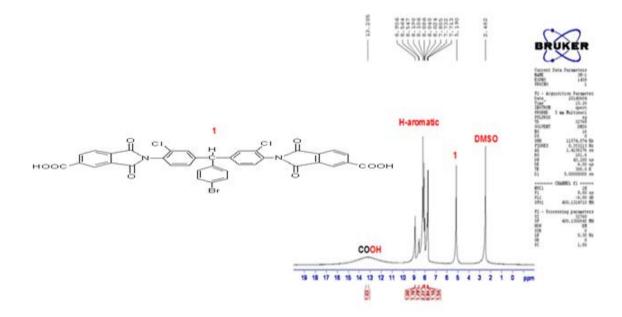


Scheme-5: Preraration of M5

Table-9: FTIR spectra of (M5) and absorption band

The FTIR spectra	absorption band
C=C Aromatic	(1597) cm <sup>-1</sup>
C-H Aromatic	(3062) cm <sup>-1</sup>
ОН	(3394) cm <sup>-1</sup>
C-H aliphatic	2977 cm <sup>-1</sup>
C-N	(1288) cm <sup>-1</sup>
C-0	(1194) cm <sup>-1</sup>
(C=O)	1694cm <sup>-1</sup>
C-Br	610cm <sup>-1</sup>
C-Cl	740cm <sup>-1</sup>

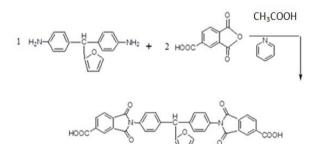




### Figure 15<sup>1</sup>: HNMR spectra of (M5)

## (M6) Preraration and Characterization

The monomer was created by the concentration of (BAPFM) (1 molecule)with pyrotrimellitic dianhydride (2 molecules)in the company of pyridine and acetic acid and after that, it refluxed for 4 hoursat room temperature overnight. This composite was defined by FTIR.

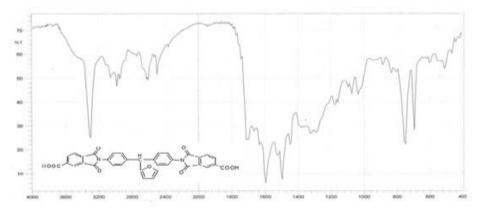


### The FTIR spectra of (M6) as a table (10).

 Table-10: FTIR spectra of (M6) and absorption band

FTIR spectra	absorption band
C=C Aromatic,	(1604) cm <sup>-1</sup>
C-H Aromatic	(3100) cm <sup>-1</sup>
OH	(3250) cm <sup>-1</sup>
C-H aliphatic	(2800) cm <sup>-1</sup>
C-N,	(1296) cm <sup>-1</sup>
C-0	(1172) cm <sup>-1</sup>
C=0	(1692) cm <sup>-1</sup>

#### Scheme-6: Preraration of M6



## Figure-16 : FTIR spectra of (M6)

1- DMSO =  $\delta$  (2.147) ppm

<sup>1</sup>H-NMR spectrum of (M6), is revealed in Figure(17) gives the subsequent changes;

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3- Ar-H = \delta (7.547 - 7.639)ppm
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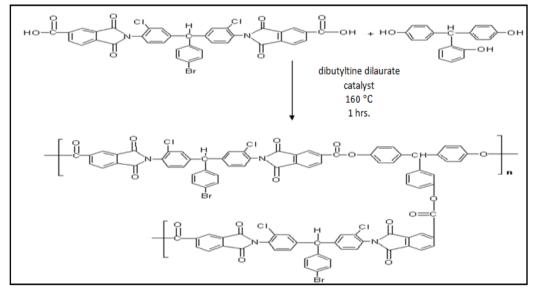
4- COOH =  $\delta$  (13.172) ppm

2- CH  $\delta = (7.547, 7.639)$  ppm 639 747 434 434 133 133 1465 495 495 147 181 181 801 801 NUCI P1 PL1 SF01 F2 SI 14 15 13 10 16 12 11 8 ÷ 3 2 ó -

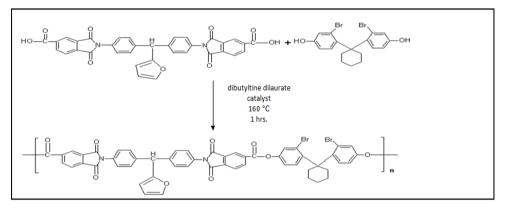
## Figure-17<sup>1</sup>: HNMR spectra of (M6)

Preraration of polyester [PEI1-PEI2]

Polycondensation procedure used for preraration of sequences(M5,M6)dicarboxylic acidof new diverse aromatic dihydroxyl monomer [M1,M2], by employing the dibutyltin dilaurate catalyst(0.15 wt.(%). It has a delicatepartlike methylene unit and a ring of the pyridine heterocyclicin key series for increase reaction with organic compounds. (esterimide)s were defined by FT-IRand<sup>1</sup>H-NMRspectra.



Scheme-7: Preraration of (PEI1)



Scheme-8: Preraration of (PEI)

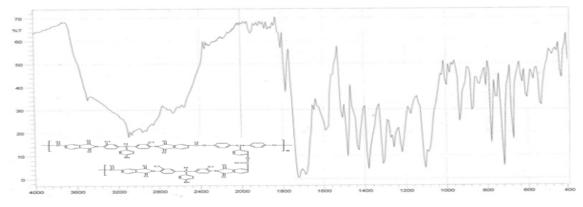
Characterization of poly (ester-imide)s [PEI1-PEI2]

The FTIR spectra of (PEI1) as in table (11).

Characterization of PEI1 FTIR spectrum of PEI1

Table-11: FTIR spectra of (PEI1) and absorption band

The FTIR spectra of	absorption band
C=C Aromatic	1589cm <sup>-1</sup>
C-H Aromatic	3093 cm <sup>-1</sup>
C-H aliphatic	2962 cm <sup>-1</sup>
C-N	1272 cm <sup>-1</sup>
C-0	1103cm <sup>-1</sup>
C=O ester	1741 cm <sup>-1</sup>
C-Cl	733 cm <sup>-1</sup>
C-Br	672 cm <sup>-1</sup>



# Figure-18: FTIR spectra of (PEI1)

(PEI1) Spectrumis revealed in Figure (19) as:

- 1- DMSO =  $\delta$  (2.358 -2.986) ppm
- 2- CH =  $\delta$  (1.200-1.889) ppm

- 3- Ar-H =  $\delta$  (6.588-8.588)ppm 4- OH =  $\delta$  (2.086-2.728) ppm
- 4- OH =  $\delta$  (3.986-3.728) ppm.

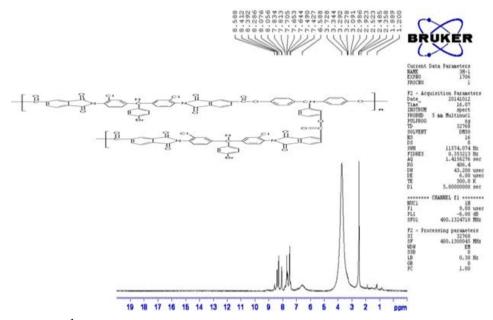


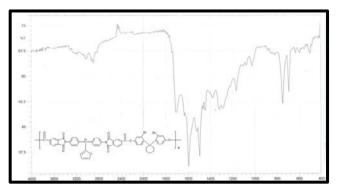
Figure-19<sup>1</sup>: HNMR spectra of PEI1

#### FTIR spectrum of PEI2

The FTIR spectra of (PEI2) as presented in table (11).

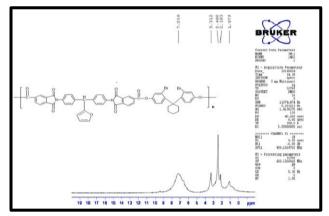
**Table-12:** FTIR spectra of (PEI2) and absorption band

The FTIR spectra	absorption band		
C=C Aromatic	1596cm-1		
C-H Aromatic	3055 cm <sup>-1</sup>		
C-H aliphatic	2954 cm <sup>-1</sup>		
C-N	1288 cm <sup>-1</sup>		
C-0	1164cm-1		
C=O ester	1704 cm <sup>-1</sup>		
C-Br	684cm-1		



**Figure-20:** FTIR spectra of (PEI2) (PEI2)OHis displayed in Figure(21) as

- 1- DMSO =  $\delta$  (2.183-2.460) ppm
- 2- CH =  $\delta$  (1.979) ppm
- 3- Ar-H =  $\delta$  (7.216) ppm
- 4- OH =  $\delta$  (3. 312) ppm



# Figure 21<sup>1</sup>: HNMR spectra of PEI2

### The solubility of poly (ester-imide)s

solubility of polyesters PEI1-PEI2is The qualitatively examined in organic solutions; the outcomes were shown in (Table 4). The way which tries to improve the process capabilities and solubility were presenting plaint linkages, bulky groups, or molecular corresponding. The supplement of bulky pendant groups in polymer backbone not only could afford an improved solubility due to reduce packing density and crystallinity [21]. Main purposes of our study were making better solubility polyesters. The Solubility is testedas (0.01) gram of polymeric sample insolvent (2) ml. Most new artificial polyesters have high soluble in common dipolar and polar a protic solvents and does not require heating.

# Table-13: Solubility of poly (ester-imide)s

Solvent	P Poly(ester- imide)				
	PEI1	PEI <sub>2</sub>			
pyridine	+ -	++			
CHCl <sub>3</sub>	+ -	++			
CH <sub>2</sub> Cl <sub>2</sub>	++	++			
NaOH	+ -	+-			
H <sub>2</sub> SO <sub>4</sub>	+ -	+++			
HNO <sub>3</sub>	++	+-			
Acetone	+ -	+ -			
Benzene	++	++			
DMF	++	+-			
DMSO	+++	++			
THF	+ +	+ -			

**1-** +++ complete soluble

2- ++Soluble at 25 C room temperature

**3-** +- partially or semi–complete Soluble

Poly (ester-imide)sthermal analysis

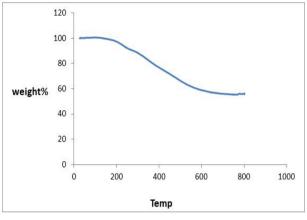
Thermogravimetric Analysis includes defining variations in mass temperature function. It is usually employed to study temperatures, absorbed of materials, concentrations of inorganic and organic parts [22]. It uses a delicate electronic balance by a temperature programmer. The thermal characteristics of four examples of this polyester were examined through thermogravimetric analysis (TGA) in Argon atmosphere at a heating rate of 10°C/min and the outcomes as  $Ti, T_{op}, T_f, T_{50\%}$ , percentage of the residue at temp (800)°C, and produces of the char at temp (500) °Care shown in (Table 4).50% weight loss temperatures of (PEI<sub>1</sub>-PEI<sub>2</sub>)as a standard reference are all temp (700-800) °C, char produces of (PEI<sub>1</sub>-PEI<sub>2</sub>) at temp (500) °C is58% until66% in Argon atmosphere that refers to they could encounter high-temperature resistant needs as particular distinctive supplies. Weight residue of (PEI<sub>1</sub>) at temp (800) °C is higher (56%) than (PEI<sub>2</sub>) (51%).

Aromat	DT/•C				Resid	Cha	
ic poly(est er-	T <sub>i</sub>	<b>T</b> <sub>op</sub> 1	<b>T</b> <sub>o</sub> <sub>p2</sub>	$\mathbf{T}_{f}$	T <sub>50</sub> %	ue at °C800	r % <i>at</i> 500•

imide)s						C
PEI1	23	40	 80	80	56%	66%
	4	0	0	0		
			<			
PEI2	22	30	 80	79	51%	58%
	0	0	0	9		
			<			

DT:temperature of Decomposition.

 $T_i$ : Initial temperature of decomposition.  $T_{op}$ :temperature of Optimum decomposition.  $T_f$ : temperature of Final decomposition.  $T_{50\%}$ :50% weight loss Temperature, got from TGA. Char% at 500°C: Residual weight percentage at 500°C in Argon by TGA





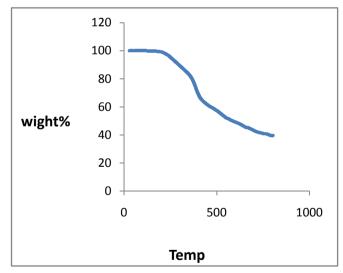


Figure-23: TGA curve of PEI2

## Conclusion

The current research work, direct polycondensation was achieved by the reactionof imide containing dicarboxylic acid and several aromatic diols to prepare aromatic poly (ester-imid)s. The polycondensation resulted in the formation of polymers that has high Glass transition. The structures of polymers and monomers wereaffirmed by FTIR and<sup>1</sup>H NMR.The polymers display good thermal characteristics and good solubility in aprotic polar solvents. Resulting poly (ester-imide)s can be used for film forming, coating material and in electric or electronic industry materials.

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