



Step-growth Polymerization of 5-(Cyclohexylethanoylamino) isophthalic acid with Different Aromatic Diols

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ABSTRACT

A series of aromatic polyesters containing amide unit in the side chain have been synthesized with moderate molecular weights by direct polycondensation of 5-(Cyclohexylethanoylamino) isophthalic acid with different aromatic diols such as 4,4'-(pyridine-2-ylmethylene) diphenol, 4,4'-thiodiphenol, 4,4'-(propane-2,2-diyl) diphenol, 4,4'-sulphonyl diphenol, hydroquinone, 4,4'-dihydroxy biphenyl and 4,4'-(pyridine-2-ylmethylene)bis(2-methylphenol) by direct polycondensation. The prepared polyesters are readily soluble in solvents such as Dimethyl formamide, N,N-dimethylacetamide, Dimethyl sulphoxide, Pyridine, Tetrahydrofuran and N-methyl-2-pyrrolidone its show that the presence of bulky pendent group may play imported role to improve the solubility of polyester. The reaction was successfully applied to the preparation of thermally stable polyesters with good yields and inherent viscosities in the range of 0.61–0.66 dL/g which was measured by using Ubbelohde viscometer at 25°C. The prepared polyesters samples were characterized by FTIR spectral study. The molecular weights of polyesters were measured by gel permeation chromatography ranging from 81512-88211. Thermogravimetric analysis and differential scanning calorimetry were employed to study the thermal properties. Thermogravimetric analysis shows that these polyester exhibits good thermal stability. DSC analysis of polyester showed that glass transition temperature (T_g) in range of 229-260°C. Selected polyesters compounds were tested for their biological activity against bacteria and fungi. The results show that polyesters could inhibit the growth of the microorganisms to a considerable extent.

Introduction

One of the distinct areas within polymer science is that of high temperature or thermally stable polymeric materials. It has been recognized as a special area within polymer chemistry for nearly 60 years. The most important classes of these polymers are polyimides, polyamides, polyarylates, and their copolymers [1,2]. They show high thermal and oxidative stability, good chemical resistance, and excellent mechanical properties. Because of these useful properties, these polymers are of major commercial and industrial importance.

However, due to poor solubility in common organic solvent and high softening or melting temperature caused by high stiffness and intermolecular bonding lead to difficult processability (i.e. “engineering activity concerned with operations carried out on polymeric

materials or systems to increase their utility”) of these type polymers [3,4]. Polyesters are now widely used for textile fibers, technical fibers, films, and bottles because of their good thermal stability, mechanical properties, chemical properties and low production cost. Therefore, the main significant research work in this area have been focused on improving their several chemical properties, processability and solubility through the design and synthesis of new monomers to obtain polymers with improved solubility while maintaining thermal stability [5-8]. There are reports shows good approaches to improve the processability of polymers by making use of structurally modified monomer [9-16]. These include (1) To introduce bulky side groups in monomer which help in the separation of polymer chains and hinder the molecular packing and crystallization. (2) Introduction of

flexible groups in polymer backbone to reduces chain stiffness. (3) Introduction of flexible chains as pendent groups. (4) Use of enlarged monomers containing angular bonds which suppress coplanar structures. Several studies on polymer science have highlighted on the possibility of processability and improving solubility of polymer, such as, polyesters, polyimides, etc. by introducing flexible side chains, such as alkyl, alkoxy group [17-20]. Another suitable way for modification of polymer properties is copolycondensation, for instance modification of the properties of polyesters by incorporation of hydrogen-bonded amide groups or imide units produces copolymers with modified properties [21].

A survey of the literature reveals that polymers bearing 5-amino isophthalic acid moiety have many potential applications in various field and several scientists [22–26] published a series of papers on polymers and copolymers of 5-amino isophthalic acid either present as an end group or as a pendent group with a view to study their complexing ability and biological activity.

EXPERIMENTAL

Materials

5- amino isophthalic acid, cyclohexyl acetic acid, 4,4'-(propane-2,2-diyl) diphenol, 4,4'-sulphonyl diphenol, 4,4'-dihydroxy biphenyl, hydroquinone, 4,4'-thiodiphenol, 4,4'-(pyridine-2-ylmethylene)bis(2-methylphenol), 4,4'-(pyridine-2-ylmethylene)diphenol were purchased from Aldrich. Pyridine, thionyl chloride, HCl, N,N-dimethyl formamide and required solvents were procured from S.D. Fine chem. India. All the chemicals and solvents used for the synthesis purpose were of laboratory grade reagent and used after proper purification whenever it required.

Techniques

Proton nuclear magnetic resonance (¹H NMR, 500 MHz) spectra

were recorded on a Bruker (Germany) advance 500 instrument. FTIR Spectra were recorded on Jasco-410 spectrophotometer. The spectra of solids were obtained using KBr pellets. The vibrational transition frequencies are reported in wavenumbers (cm⁻¹). Inherent viscosities were determined by using Ubbelohde viscometer at 25^oC in Dimethyl formamide. Thermal gravimetric analysis (TGA) data for polymers were obtained by TGA-Perkin Elmer (Pyris 1) in nitrogen atmosphere. DSC data for polymers were obtained by DSC- Perkin Elmer (Pyris 1). GPC data were obtained by using GPC instrument PSS win RI-71 detector Shodex equipped with 600E multi solvent delivery system.

Monomer Synthesis: Preparation of 5-(Cyclohexylethanoylamino) isophthalic acid

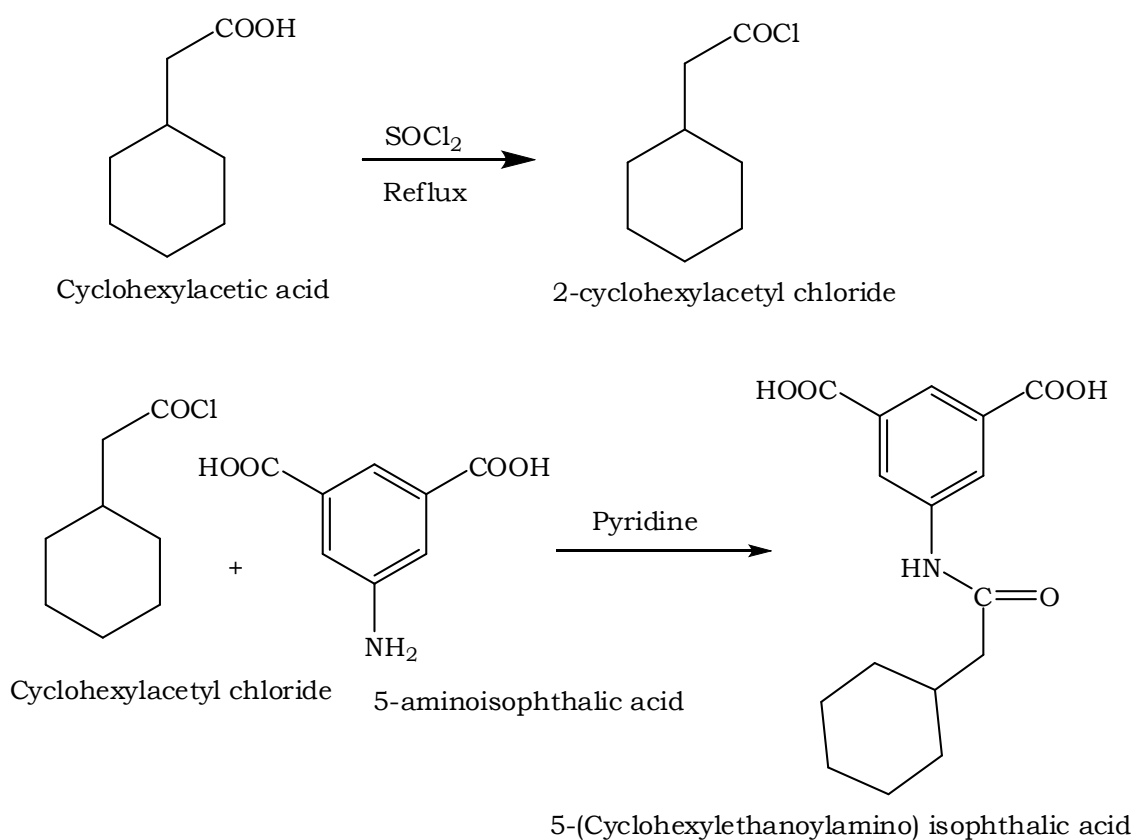
Cyclohexylacetic acid (0.05 mole) and thionyl chloride (0.15 mole) were taken in round bottom flask. The reaction mixture was refluxed in water bath for 4 hours with frequent shaking. Then unreacted thionyl chloride was distilled out by reducing pressure. 25 ml of pyridine was added in residue and then mixture was cooled up to 0^oC. In another flask, solution of 5-amino isophthalic acid (0.051mole) in 40 ml pyridine was taken and cooled up to 0^oC. This solution was added into the above prepared solution within the duration of half an hour with stirring. The reaction mass was then warmed and stirred for 12 hrs at room temperature. The reaction mass was quenched in cold HCl-water solution. The product obtained was filtered and washed with water till pH of the filtrate was found neutral. Isolated product was dried at 50^oC under vacuum. Crude product was crystallized in Toluene. Yield: 88%.

Melting point: >350^oC

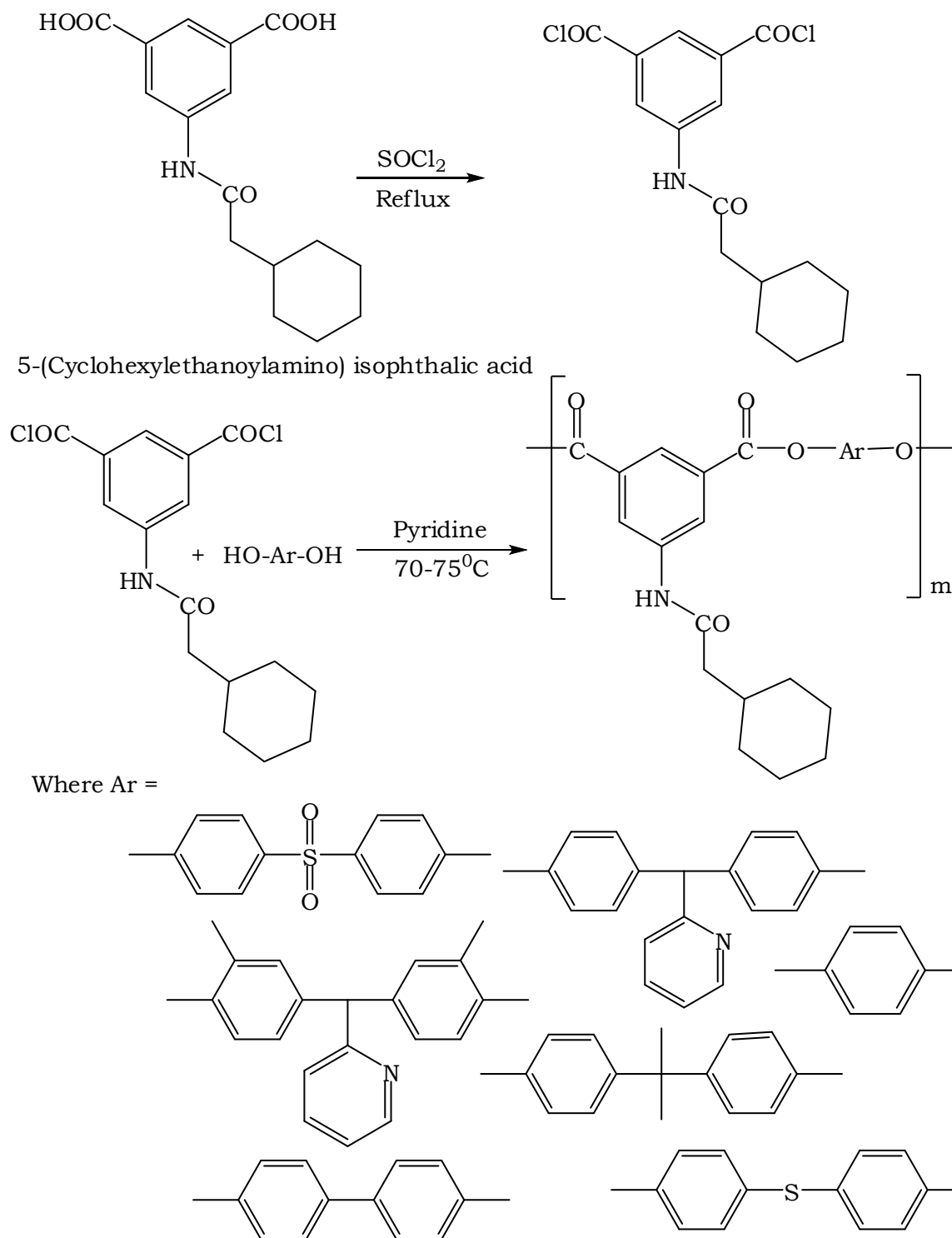
Synthesis of aromatic polyester of 5-(Cyclohexylethanoylamino) isophthalic acid²⁷.

5-(Cyclohexylethanoylamino) isophthalic acid (0.01mole) and thionyl chloride(0.03 mole) were taken in round bottom flask. 25 mg dimethyl formamide was added in it. The reaction mixture was refluxed in water bath for 4 hours with stirring. Then unreacted thionyl chloride was distilled out by reducing pressure. Residual mass was cooled up to 0°C. 10 ml of pyridine was added in it. The solution of diol (0.01mole) in 15 ml

pyridine was prepared in another flask and it was cooled up to 0°C. This solution was added in above solution within half an hour with stirring. It was then heated up to 70-75°C and stirred for 20 hrs at 70-75°C. The reaction mass was quenched in HCl-water solution. The product obtained was filtered and washed with water till pH of the filtrate was found neutral. Isolated product was dried in oven.



Scheme-1: Synthesis of Monomer.



Scheme-2: Synthesis of polyester.

Table 1: Characterization of 5-(Cyclohexylethanoylamino) isophthalic acid

Molecular Formula	Molecular Wt. (g/m)	M.P. °C	% Yield	Color	Analysis % Found (Calculated)		
					C	H	N
C ₁₆ H ₁₉ NO ₅	305.33	>350°C	88	Light Yellow	62.90 (62.94)	6.25 (6.27)	4.62 (4.59)
IR	FTIR Spectra show the characteristic carbonyl absorption peaks for acid at 1693 cm ⁻¹ due to the carbonyl stretching vibrations and -OH(Stretching) at 2842cm ⁻¹						
Mass spectra (m/z)	305						

Solubility of Aromatic Polyesters

The solubility of polyesters was tested quantitatively in various solvents as listed in Table 2. All the poly-esters are soluble in organic solvents such as dimethyl formamide (DMF), tetrahydrofuran (THF), dimethylacetamide (DMAC), pyridine and H₂SO₄ at room temperature, and are insoluble in solvents such as methylene dichloride (MDC), ethyl acetate, toluene, hexane, acetonitrile and water. The incorporation of the pendant cyclopentanamide in the side chain polyesters enhanced their solubility in polar solvents.

Table-2: Solubility data of polyesters

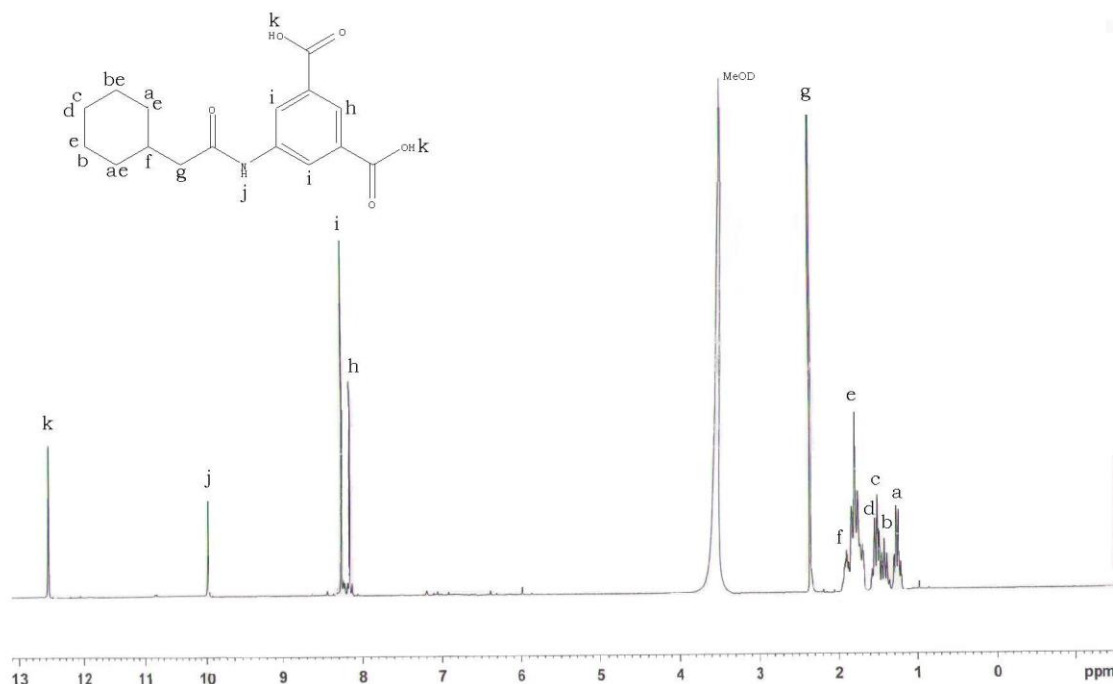
Polyester	DMF	NMP	DMAC	Sulpho lane	Pyridine	THF	Toluene	MDC	Water	Hexane	Ethyl acetate	Acetonitrile
PE-HQ	++	+	--	--	--	+	--	--	--	--	--	--
PE-HA	++	++	++	++	++	++	--	--	--	--	--	--
PE-HM	++	++	++	++	++	++	--	--	--	--	--	--
PE-HP	++	++	++	++	++	++	--	--	--	--	--	--
PE-HT	++	++	++	++	++	++	--	--	--	--	--	--
PE-HD	++	++	++	++	++	++	--	--	--	--	--	--
PE-HS	++	+	--	--	--	+	--	--	--	--	--	--

Table-3: Physical property of polyester

Polyester	Diphenol	Description	Yield (%)	η_{inh} (dL/g) ^a
PE-HQ	HQ	Yellow power	94	0.66
PE-HA	BPA	Light yellow powder	96	0.61
PE-HM	BPM	Light yellow powder	94	0.62
PE-HP	BPP	Light yellow powder	95	0.63
PE-HT	TDP	Light yellow powder	95	0.62
PE-HD	DPD	Light yellow powder	92	0.65
PE-HS	BPS	Light yellow powder	96	0.64

(a) Measured at a concentration of 0.5 g/dL in DMF at 25°C.

Figure-1: NMR of CHAA

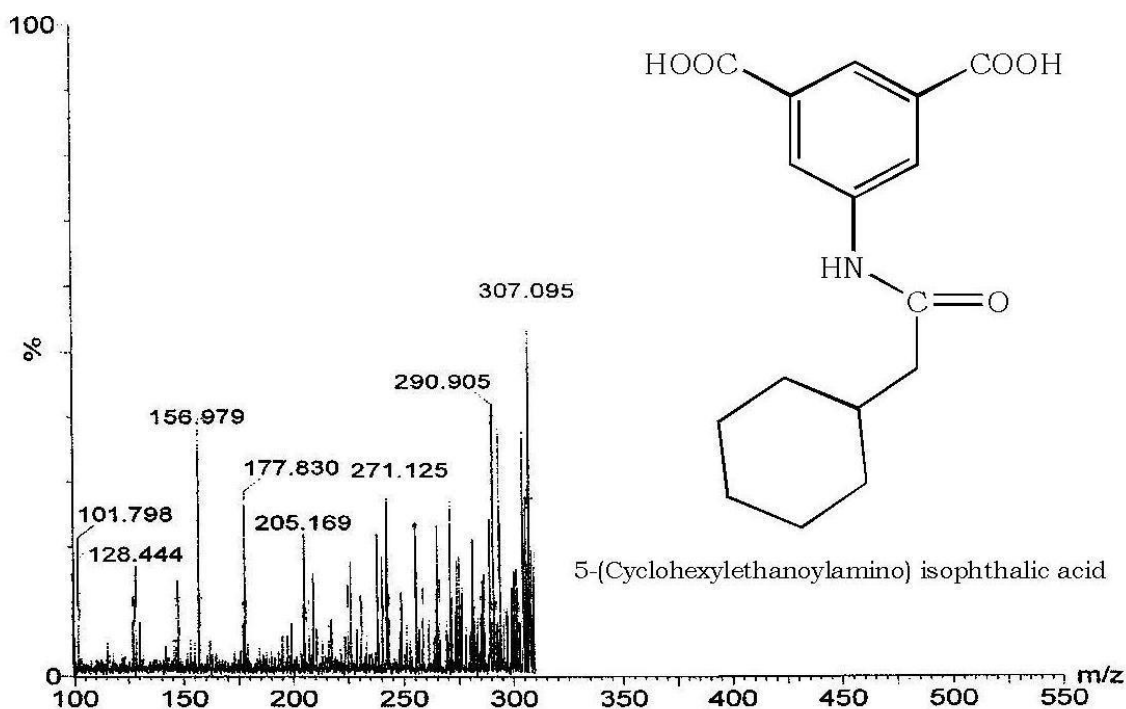


Prediction of ¹H- NMR of 5-(Cyclohexylethanoylamino) isophthalic acid

Sr. No	Node	Shift	Remarks
1	-CH ₂ -	1.2 (m), 1.5(m)	2H cyclo hexane
2	-CH ₂ -	1.4 (m), 1.5(m)	2H cyclohexane
3	-CH-	1.6 (m)	1H cyclohexane
4	-CH ₂ -	2.2 (d)	2H methylene (1alpha to -C=O)
5	-CH-	8.5 (s)	1H ArH
6	-NH-	10.0 (s)	1H -NH-
6	-OH	12.7 (s)	-COOH group

NMR of CHAA shows that NMR spectra matched with ideal structure

Figure -2 : Mass Spectra of CHAA



Interpretation of Mass Spectra of CHAA

Sr. No.	Fragment	m/z
1	Molecular Ion peak (M+2)	307.095
2	C ₁₆ H ₁₈ N ₀₄ ⁺	290.905
3	C ₁₆ H ₁₇ N ₀₃ ⁺	271.125
4	C ₉ H ₇ N ₀₃ ⁺	177.830

Characterization of Polyesters by FTIR Spectra

The formation of aromatic polyesters was confirmed by FTIR spectroscopy analysis. FTIR Spectra of all polymers show the characteristic carbonyl absorption peaks for ester at 1720-1750 due to the carbonyl stretching vibrations. The vibrations due to aromatic ring are assigned to absorptions in the range of 3000-2900 cm⁻¹. The polyesters showed characteristic absorptions at 2960-2850 cm⁻¹ due to aliphatic ring. Interpretation of FTIR spectra is listed in Table-4

Tabel-4: Interpretation of FTIR Spectra

Type	Group vibration Mode	PE-HQ	PE-HS	PE-HD	PE-HM	Expected Frequencies (cm ⁻¹)
Aromatic	=C-H (Str)	2972	2931	3020	3080	3080-2900
	C=C (Str)	1605	1589	1603	1608	1600-1450
		1447	1488	1500	1510	
		759	744	1450	1453	540-1400
	C-H (i-p-d)	1020	1292	1245	1190	1350-1000
			1153	1020	1119	
			1172			
	C-H (o-o-p-d)	805	918	866	990	960-675
		725	837	808	810	
		701	682	725		
			701			
-(CH ₂)-	C-H (Str)	2878	2854	2932	2931	2960-2850
-C-N-	C-N(Str)	1320	1319	1340	1320	1310-1380
-NHCO-	N-H (Ben.)	1549	1542	1551	1544	1540-1600
	N-H (Str)	3110	3120	3230	3243	3080-3450
-COO-	C=O (Str)	1713	1747	1720	1714	1735-1720
	C-O (Str.)	1206	1292	1202	1270	1260-1225
Sulphone	S=O (Str)		1195			1300-1160
	S-O (Str)		682			700-500
	C-S (Str)		555			700-570

Gel permeation chromatography: The molecular weight and Polydispersity value of polyesters are listed in Table-5. These amount shows that the prepared polyesters are medium molecular weight in the range of 81512-88211.

Table-5: GPC data of polyesters

Polyether	Diphenol	Molecular weight ^a		Polydispersity Mw/Mn
		Mn	Mw	
PE-HA	BPA	81512	84982	1.042
PE-HT	TDP	82514	84759	1.027
PE-HD	DPD	88211	89966	1.019

a: Measured by GPC in Dimethyl formamide (DMF), polystyrene was used as a calibration standard.

Thermal Properties

Thermogravimetric Analysis (TGA).

Pyrolysis of many polymers yields the TG curve, which follows a relatively simple sigmoid curve. In such a case weight of the sample decreases slowly as reaction begins, then decreases rapidly over a comparatively narrow range of temperature and finally levels off as the reaction gets completed. The shape of the curve depends on the kinetic parameters viz., reaction order (n), frequency factor (Ao) and activation energy (E*). The values of these parameters have been shown to be of major importance to elucidate the mechanism involved in polymer degradation and to estimate the thermal stability.

The thermal stability of polymers was studied by TGA under a nitrogen atmosphere. The 5%, 10% and 50% weight loss (T5, T10 and T50) of the polymers and residue at 600°C (char yield) for these polymers are summarized in Table 4. IPDT is calculated by Doyle equation²⁸. Activation energy calculated by using broido method²⁹.

Table-6: IPDT and Activation energy of Polyesters.

Sr. No	Polyester	IPDT	Activation energy (KJ/Mole) Ea	
			Slope-I	Slope-II
1	PE-HA	515.0°C	25.21	57.69
2	PE-HD	488.5°C	32.98	59.72
3	PE-HM	908.3°C	55.78	67.02

Table-7: Thermal properties of polyesters

Polyester	Decomposition temperature			Char yield ^d (%)
	T5 ^a	T10 ^b	T50 ^c	
PE-HA	165°C	244°C	425°C	24.39%
PE-HD	205°C	232°C	336°C	27.09%
PE-HM	186°C	203°C	384°C	34.20%

(a) Temperature at which 5% weight loss was recorded by TGA

(b) Temperature at which 10% weight loss was recorded by TGA

(c) Temperature at which 50% weight loss was recorded by TGA

(d) Percentage weight of material left undecomposed after TGA analysis at maximum temperature 600°C in a nitrogen atmosphere.

Differential Scanning Calorimetry of the Polyester.

The thermal properties of all the polyesters have been investigated by DSC measurements. Their phase transition temperatures of first heating scan are listed in Table 8. According to the DSC analysis the prepared polyesters showed glass transition temperature (T_g) in range of 229-260°C.

Table- 8: Transition temperatures of the polyester determined by DSC

Polyester	Thermal transition from	
	DSC (°C, J/g)	
	T _g °C	ΔH
PE-HP	235.35°C	38.14 J/g
PE-HM	229.01°C	135.3J/g
PE-HQ	260.35°C	66.42 J/g

NMR Spectra of Polyesters: H¹- NMR and C¹³- NMR spectra confirmed the structure of polyester in all aspect.

Figure-3: H¹- NMR Spectra of PE-HA

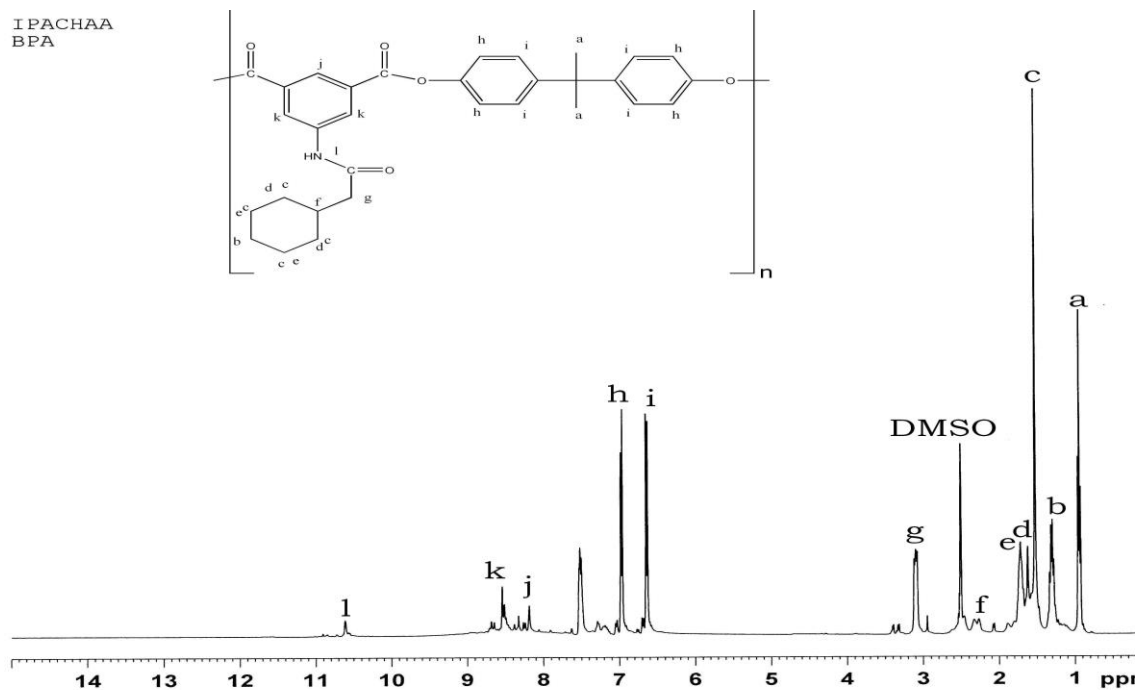
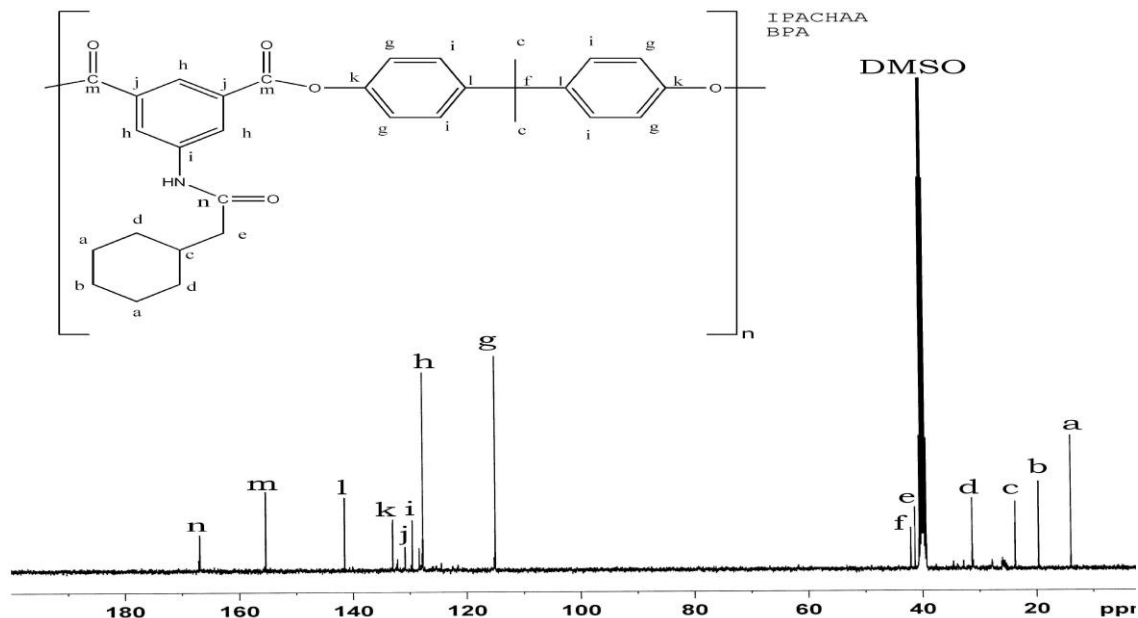


Figure-4: C^{13} - NMR Spectra of PE-HA



Antimicrobial Activity

Polyester compounds were tested for their response against microorganisms, interesting results were obtained. The results indicate that the polyesters inhibit significantly the growth of microorganisms. The bacterial subcultures for *Bacillus subtilis*, *Staphylococcus aureus*, *Escherichia coli*, *Pseudomonas aeruginosa* were obtained from central diagnostic laboratory, Surat, Gujarat, India. An antifungal susceptibility test was used *Candida albicans*. *Bacillus subtilis*, *Staphylococcus aureus*, *Escherichia coli*, *Pseudomonas aeruginosa* and *Candida albicans* were cultured on Brain Heart Infusion Broth (BHI) for the antibacterial and fungal activity

Each compound was dissolved in Dimethyl formamide (DMF) at a concentration of 50, 100, 150, 200, 400, 600, 800, 1000, 1200 and 1500 $\mu\text{g/mL}$ concentrations. In table 9 zone of inhibition in mm is described at concentration of 1500 $\mu\text{g/mL}$.

Table- 9: Zone of inhibition in mm of polyesters.

Sample	Zones of inhibition in mm				
	B. subtilis	S. aureus	E. coli	P. aeruginosa	C.albicans
PE-HQ	20	22	11	12	20
PE-HP	20	24	12	13	19
PE-HM	21	23	13	14	18
PE-HA	22	24	11	13	21
PE-HS	19	23	12	13	20
PE-HT	21	22	13	12	19
PE-HD	19	21	12	12	21
DMF	10	10	10	10	10

CONCLUSION

In this research work, polyesters compounds were prepared by direct polycondensation reaction of diacid monomer with several aromatic diols. The polycondensation leads to the formation of polymers having inherent viscosity ranging from 0.61–0.66 dL/g. By introducing bulky pendent group to the aromatic polyesters, polymers with substantially increased solubility and good thermal stability were obtained. Since the resulting polyesters have they can be used for film forming and coating materials.

SYMBOLS AND ABBREVIATIONS

- CHAA** : 5-(Cyclohexylethanoylamino) isophthalic acid
- PE-HQ** : Poly(Hydroquinone)-alt-(5-(Cyclohexylethanoylamino) isophthalic acid)

PE-HA : Poly(4,4'-(Propane-2,2-diyl)diphenol)-alt-(5-(Cyclohexyl ethanoyl amino) isophthalic acid)

PE-HM : Poly(4,4'-(Pyridin-2-ylmethylene) bis (2-methyl phenol))-alt-(5-(Cyclohexylethanoylamino) isophthalic acid)

PE-HP : Poly(4,4'-(Pyridin-2-ylmethylene)diphenol)-alt-(5-(Cyclohexyl ethanoylamino) isophthalic acid)

PE-HT : Poly(4,4'-Thiodiphenol)-alt-(5-(Cyclohexylethanoylamino) isophthalic acid)

PE-HD : Poly(Biphenyl4'4-diol)-alt-(5-(Cyclohexylethanoylamino) isophthalic acid)

PE-HS : Poly(4,4'-sulphonyldiphenol)-
alt-((5-(Cyclohexylethanoyl
amino) isophthalic acid)

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