

# Studies on Synthesis and Characterization of some Novel Aromatic Copolyesters based on s-Triazine

Hemant S. Patel\*, Naitik B. Patel, and Keshav C. Patel

Department of Chemistry, Veer Narmad South Gujarat University, Surat-395 007, Gujarat, India

Received 17 September 2005; accepted 11 December 2005

## ABSTRACT

Ten copolyesters were synthesized by high temperature polycondensation of 2-(N-ethylanilino)-4,6-bis(phenoxy-2-carbonyl chloride)-s-triazine [EAPCCT] with each of the following mixtures of diols: [Bisphenol-A(BPA)+Bisphenol-C(BPC)], [BPA+Bisphenol-S(BPS)], [BPA+Phenolphthalein(Ph)], [BPA+1,8-dihydroxy anthraquinone(DHA-1,8)], [BPA+1,4-dihydroxy anthraquinone(DHA-1,4)], [BPA+catechol(C)], [BPA+resorcinol(R)], [BPC+Ph], [BPC+R], and [Ph+DHA-1,4]. All the copolyesters were characterized by solubility, density, and viscosity measurements, IR and NMR spectroscopy, and thermogravimetric analysis method. All the copolyesters were soluble at high temperature in acetone, dimethylformamide, dimethylsulphoxide, dimethylacetamide, etc. and have reduced viscosity in the range of 0.539-0.678 dL/g in dimethylformamide at 30°C. Copolyester derived from the diol mixture [BPA+DHA-1,4] showed the greater thermal stability than the other copolyesters.

### Key Words:

copolyesters;  
2-(N-ethyl anilino)-4,6-bis(phenoxy-2-carbonyl chloride)-s-triazine;  
high temperature polycondensation;  
viscosity determinations;  
IR spectroscopy.

### INTRODUCTION

Great attention has recently been paid to synthesize the high-performance polymers with increased thermal stability and heat resistance, and good mechanical properties, as these are required in the modern technological applications [1,2]. Aromatic

polyesters and copolyesters are thermally stable polymers with a favorable balance of physical and chemical properties [3]. They form one of the most important class of the polymers. They can compete with almost all the known conventional materials

(\* ) To whom correspondence should be addressed.  
E-mail: hemant7430@yahoo.co.in

in their characteristic applications [4,5]. They are commercially available as high performance engineering plastic materials. However, these polymers generally lack the properties essential for successful fabricating into useful forms. Many researchers have tackled these inherent problems over the last few decades. As an approach to improve the solubility and processibility of heterocyclic polymers, while maintaining the thermal stability, numbers of published reports have described the synthesis and properties of polyesters and their copolyesters containing an *s*-triazine ring [6-12]. The polymers containing *s*-triazine rings in their backbone are unique in the sense that they exhibit an unusual combination of properties such as high softening temperature and thermal stability together with solubility [13,14].

Many of the problems arising while processing and fabricating homopolyesters, for example, decomposition at processing temperature, melt stiffness etc., can be reduced or almost solved by using copolyesters of the mixture of diols and diacid chlorides. The present work describes synthesis and characterization of some aromatic copolyesters by condensation of EAPCCT with various mixtures of aromatic diols.

## EXPERIMENTAL

### Materials

Dimethylformamide, methanol, acetone and other common chemicals used were laboratory grade reagents. Cyanuric chloride (Fluka, mp 146°C) was purified by recrystallization from pure benzene solution. BPA (Resin grade, Cibatul, mp 156°C) was repeatedly crystallized from 50% aqueous acetic acid and finally recrystallized from benzene solution. Phenolphthalein (BDH, mp 262°C) were purified by crystallization from ethanol solution, Resorcinol and catechol (Sisco lab) were purified by recrystallization from their rectified spirit. 1,8-dihydroxy Anthraquinone and 1,4-dihydroxy anthraquinone (Sisco lab) were purified by recrystallization from aqueous alcohol solution.

### Material Synthesis

BPC and BPS were synthesized by published procedures [15] and were crystallized from benzene solution (mp 187°C and 239°C, respectively).

### Synthesis of Monomer EAPCCT

2-(*N*-ethyl anilino)-4,6-bis(phenoxy-2-carbonyl chloride)-*s*-triazine was synthesized by the reported method [16] as per reaction Scheme I. The procedure adopted for the synthesis is performed as follows.

#### 2-(*N*-ethyl anilino)-4,6-dichloro-*s*-triazine

A solution of cyanuric chloride (18.44 g, 0.1 mol) in a 60 mL acetone was added with stirring to a cold solution (0-5°C) of sodium bicarbonate (9.4 g, 0.1 mol) in 100 mL of distilled water, in a three necked flask (250 mL), equipped with a mechanical stirrer. This resulted in the formation of slurry of cyanuric chloride. A solution of *N*-ethylaniline (12.7 mL, 0.1 mol) in 10 mL acetone was added to the cold slurry of cyanuric chloride with continuous stirring. After addition was completed, the reaction mixture was stirred for 2 h at 0-5°C. The white colored product was filtered and recrystallized from ethanol and dried in a vacuum desiccator (yield 90 % and mp 127°C).

If the reaction is carried out in water, the reaction is left incompleting with low yield. But the yield is improved considerably if the cyanuric chloride is freshly precipitated by pouring an acetone or dioxane solution into ice cold water. The use of aqueous system allows the product to be isolated easily in higher yield [17].

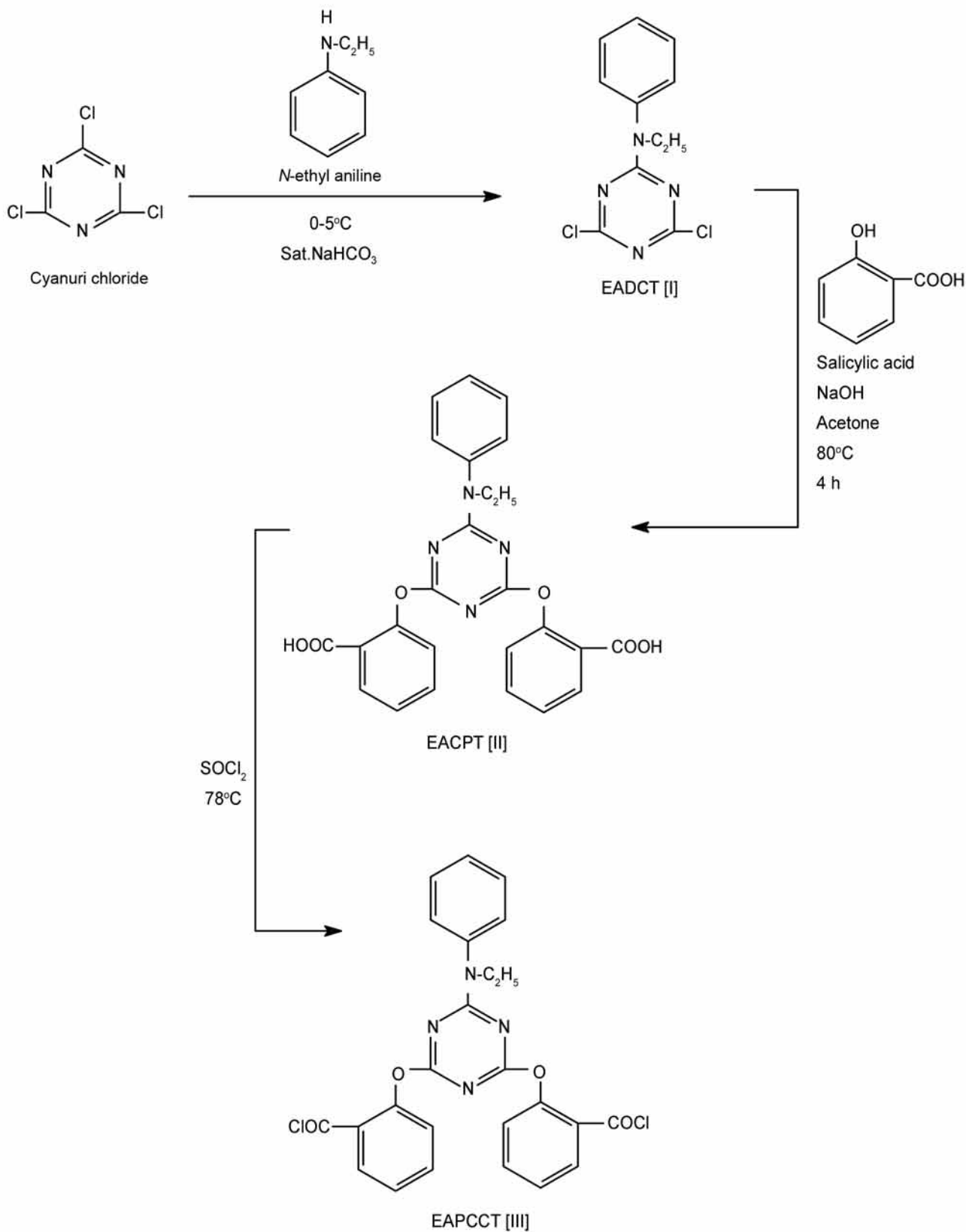
#### 2-(*N*-ethyl anilino)-4,6-bis(2-carbophenoxy)-*s*-triazine

A solution of 2-(*N*-ethyl anilino)-4,6-dichloro-*s*-triazine (EADCT) (26.9g, 0.1 mol) in 40 mL of acetone, a mixture of NaOH (16.0g, 0.4 mol) and salicylic acid (2.76g, 0.2 mol) in 80 mL double distilled water was added slowly at room temperature in a solution of EADCT with constant stirring. Reaction was continued for 4 h; first 2 h at room temperature and then another 2 h at 80°C.

At the end of the reaction, the white solid separated from the reaction mixture was filtered and washed several times with hot water and dried in vacuum at 100°C. The product was recrystallized from acetone. The yield was 80% with mp 167°C.

#### 2-(*N*-ethyl anilino)-4,6-bis(phenoxy 2- carbonyl chloride)-*s*-triazine

Place 2-(*N*-ethyl anilino)-4,6-bis(2-carbophenoxy)-*s*-triazine (EACPT) (4.72g, 0.01 mol) in a dry round bottom flask. Thionyl chloride (11.9 mL, 0.1 mol) was



Scheme I.

added at the top of the condenser and the reaction mixture was refluxed at 78°C for 2 h. At the end of the reaction the excess of thionyl chloride was distilled off and dry product was recovered. The yield was about 80% and recrystallized from dimethylformamide (mp 198°C) (Scheme I).

### Synthesis of Copolyesters

A high temperature polycondensation is exemplified below for the synthesis of copolyesters from 2-(*N*-ethyl anilino)-4,6-bis(phenoxy 2-carbonyl chloride)-s-triazine (EAPCCT) with a mixture of diols [BPA+BPC].

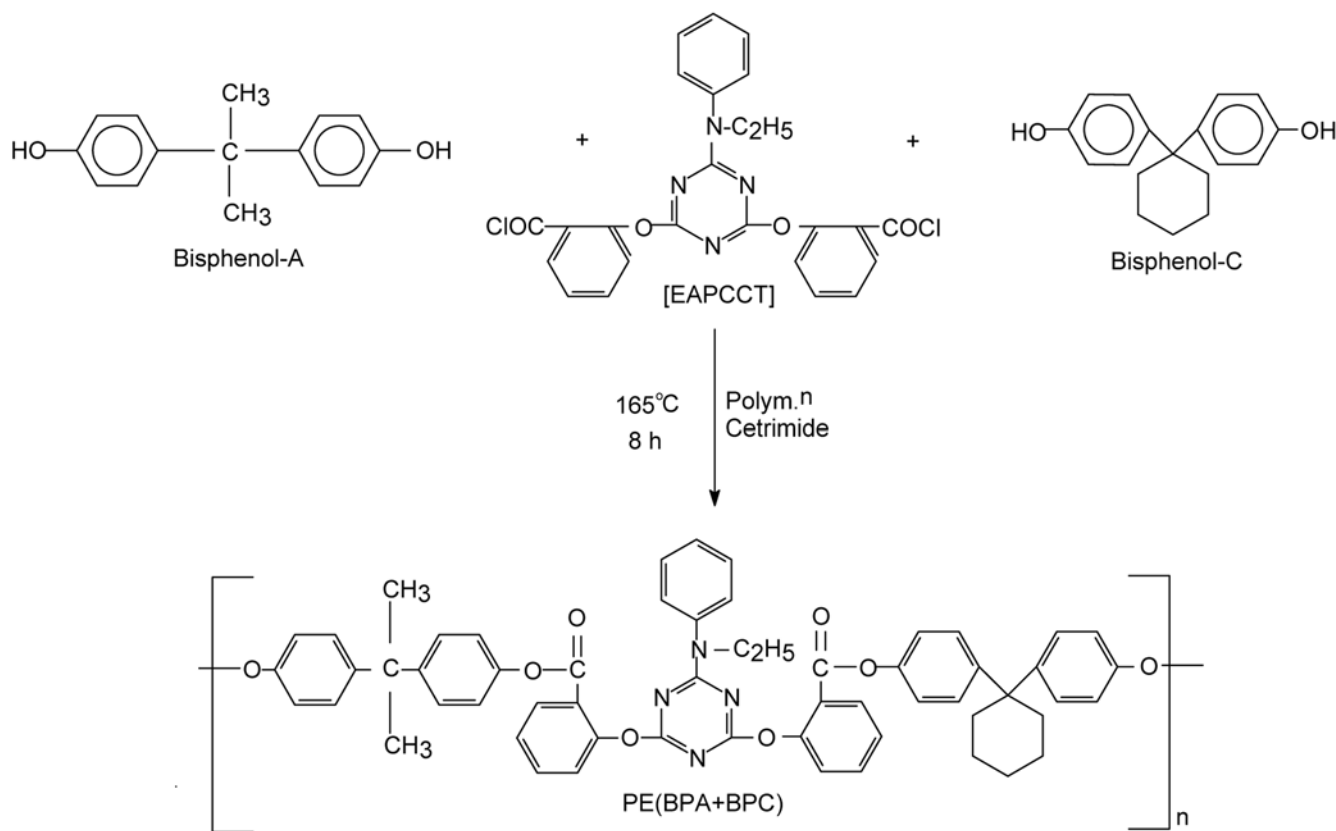
EAPCCT (5.09g, 0.01 mol) in minimum quantity of DMF was heated to about 150°C. Cetrimide (0.25g) and then the mixture of [BPA (2.28 g, 0.01 mol) + BPC (2.68 g, 0.01 mol)] were added. The reaction temperature was raised to 165°C and heated for 8 h. The reaction mixture was cooled and poured with constant stirring into 250 mL of ice cold water. The solid was filtered and washed with hot water and finally with

methanol to remove unreacted monomer and dried. The yield was about 90%.

The other copolyesters from EAPCCT and various mixtures of diols such as; [BPA+BPS], [BPA+Ph], [BPA+DHA-1,8], [BPA+DHA-1,4], [BPA+C], [BPA+R], [BPC+Ph], [BPC+R], [Ph+DHA-1,4] were synthesized by the similar method as is described above (Scheme II).

### RESULTS AND DISCUSSION

High temperature polycondensation of EAPCCT with [BPA+BPC] has been investigated in detail to examine the effect of variables such as the nature of organic solvent, reaction time and temperature. The remaining variables, such as concentration of reactants, their ratio, rate of stirring and initiator used, were maintained nearly constant for all of the experiments. The quantity and quality of the copolyester of [BPA+BPC] obtained at various temperature are presented in Table 1. Based on



Scheme II.

**Table 1.** Systematic study of high temperature polycondensation technique of EAPCCT (0.1 mol) with [BPA(0.1 mol) +BPC(0.1 mol)] for optimization of reaction conditions.

System	Reaction temperature (°C)	Yield (%)	Product appearance	$\eta_{sp}$ (dL/g)
DMF	100	55	Powdery	0.615
DMF	125	65	Powdery	0.635
DMF	150	70	Powdery	0.705
DMF	165	85	Powdery	0.661
DMF	200	70	Powdery	0.638

these data, suitable reaction conditions have been selected for the synthesis of all of the copolyesters of various mixtures of aromatic diols. The reduced viscosity of the polymer solution ( $1 \text{ g dL}^{-1}$ ) in dimethylformamide is used as a criterion of the quality of the polymer formed in a given set of reaction conditions.

### Solubility

Solubility of the copolyesters was determined for powdery samples in excess solvents. Most of the copolyesters were soluble in a variety of solvents such as; dimethylformamide, dimethylacetamide, dimethylsulfoxide, methanol, tetrahydrofuran, and ethyl acetate. Aliphatic and aromatic chlorinated solvents like chloroform, carbon tetrachloride, chlorobenzene, mono and

dichloro ethane are non-solvents for polyesters. The copolyesters were also insoluble in diethyl ether, benzene, bromo benzene, and nitro benzene.

Dimethyl formamide and ethyl acetate are the good solvents for all these copolyesters.

### Viscosity Measurements

Dilute solution viscosity measurements were carried out using Ubbelohde suspended level kinematic viscometer. Reduced viscosities for all the solutions (1%) of copolyesters in DMF were determined at  $25 \pm 0.1^\circ\text{C}$ . Typical Huggins's and Kraemer's plots were used to obtain intrinsic viscosity for each copolyesters.

Results for reduced viscosity of copolyesters are given in Table 2. From the results, it reveals that PE(BPA+Ph) has the highest solution viscosity and hence highest molecular weight amongst all of these copolyesters, where as the PE(BPC+R) has the lowest solution viscosity. The reduced viscosity of the copolyesters obtained from EAPCCT and various diols mixtures follows the sequence given below:

$$\text{PE(BPA+Ph)} > \text{PE(BPA+BPC)} > \text{PE(BPC+Ph)} > \text{PE(Ph+DHA-1,4)} > \text{PE(BPA+DHA-1,4)} > \text{PE(BPA+DHA-1,8)} > \text{PE(BPA+BPS)} > \text{PE(BPA+C)} > \text{PE(BPA+R)} > \text{PE(BPC+R)}$$

### Density Measurements

Density of all of these copolyesters was determined at  $25 \pm 1^\circ\text{C}$  by suspension method by pycnometer [18]. The results are presented in Table 2. Density of copolyesters

**Table 2.** Physical Characteristics of copolyesters.

Polymer Code	Mixture of diols used	Product appearance	Yield (%)	Density (g/mL)	$\eta_{sp}$ (dL/g)
PE(BPA+BPC)	(BPA+BPC)	Grey powder	75	1.254	0.6609
PE(BPA+Ph)	(BPA+Ph)	Reddish Brown	90	1.246	0.6783
PE(BPA+DHA-1,4)	(BPA+DHA-1,4)	Red	85	1.257	0.6348
PE(BPA+DHA-1,8)	(BPA+DHA-1,8)	Green	90	1.260	0.6174
PE(BPA+BPS)	(BPA+BPS)	Black	90	1.222	0.5823
PE(BPA+C)	(BPA+C)	Black	85	1.198	0.5652
PE(BPA+R)	(BPA+R)	Black	85	1.191	0.5478
PE(BPC+Ph)	(BPC+Ph)	Grey	90	1.250	0.6522
PE(BPC+R)	(BPC+R)	Black	80	1.194	0.5391
PE(Ph+DHA-1,4)	(Ph+DHA-1,4)	Reddish Brown	88	1.252	0.6404

varies in the range of 1.260 -1.191 gm/cm<sup>3</sup>. The highest density is displayed by PE(BPA+DHA-1,8), and the lowest by PE(BPA+R).

The density of the copolyesters obtained from EAPCCT and various mixtures of diols decreases in the following order:

PE(BPA+DHA-1,8) > PE(BPA+DHA-1,4) > PE(BPA+BPC) > PE(Ph+DHA-1,4) > PE(BPC+Ph) > PE(BPA+Ph) > PE(BPA+BPS) > PE(BPC+C) > PE(BPC+R) > PE(BPA+R)

### IR Spectral Characteristics

IR spectra of all of these copolyesters were scanned in KBr on a Perkin Elmer FTIR Paragon 1000 SPIR S. No. 42825. The IR spectra of the copolyesters (Figure 1) exhibited several common characteristic absorption frequencies (cm<sup>-1</sup>). The structural features of the monomer employed for copolyester synthesis are such that all the ten copolyesters formed will be linear.

A broad band, which is a characteristic of bonded -OH group, extends from 3020 to 3680 cm<sup>-1</sup> with a maximum around 3400-3420 cm<sup>-1</sup>, most probably due to -OH bond of end -COOH group of the copolyesters. Certain inflections are observed in the above mentioned broad band. The inflection at 3050-3080 cm<sup>-1</sup> is attributed to aromatic nucleus stretching [19,20].

All the copolyesters exhibit band at 720-795 cm<sup>-1</sup> and 840-890 cm<sup>-1</sup> may be due to bending vibration of the C-H bands of aromatic ring. The presence of s-triazazine ring is supported by the appearance of band at 1440-1510 cm<sup>-1</sup>, which is due to in plane bending vibration of s-triazazine ring. The bands observed around

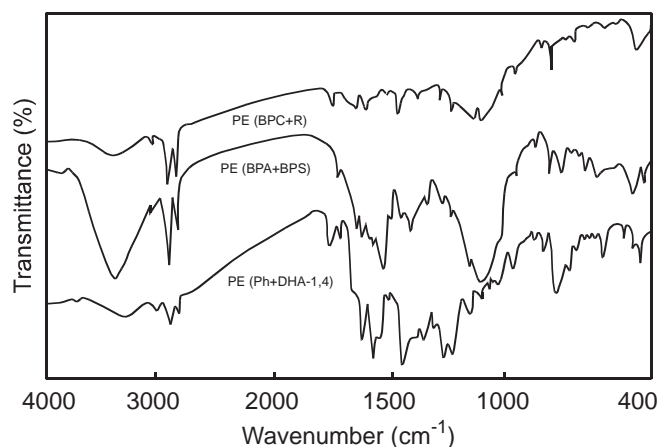


Figure 1. IR Spectrum of some copolyesters.

1020-1050 cm<sup>-1</sup> are attributed to vibration of aryl-ether linkage and 1080-1110 cm<sup>-1</sup> may be due to in plane bending vibration of aromatic C-H bond. The presence of aryl-ether band is further supported by the appearance of band at 1220-1240 cm<sup>-1</sup>.

In spectra of all the copolyesters, two bands are observed, one at 1140-1160 cm<sup>-1</sup> and the other at 1260-1300 cm<sup>-1</sup> which are attributed to symmetric and asymmetric stretching vibrations of C-O-C (ester) group [21]. In spectra of all these copolyesters, a band around 1720-1740 cm<sup>-1</sup> is observed which confirms the presence of ester carboxyl group. A band around 3050-3085 cm<sup>-1</sup> indicates the presence of C-H stretching vibration and a band around 3400-3425 cm<sup>-1</sup> is due to stretching vibration of O-H (bonded).

IR spectra of copolyesters of [BPA+BPC], [BPA+BPS], [BPA+Ph], [BPA+DHA-1,8], [BPA+DHA-1,4], [BPA+C], and [BPA+R] exhibit a distinct band at 550-580 cm<sup>-1</sup> confirming the presence of C-C deformation vibration of propyl link due to BPA while in spectra of copolyesters of [BPA+BPC], [BPC+Ph] and [BPC+R] two bands exhibit around 540 cm<sup>-1</sup> and 960-965 cm<sup>-1</sup> due to deformation vibration of cyclohexane ring of BPC.

### NMR Spectral Characteristics

High resolution (300 MHz) spectra of solution of copolyesters sample were measured in deuterated (DMSO-d<sub>6</sub>) using TMS as an internal reference. NMR spectrum of PE(BPA+C) is shown in Figure 2. The chemical shift in δ ppm for the copolyesters presented in Table 3.

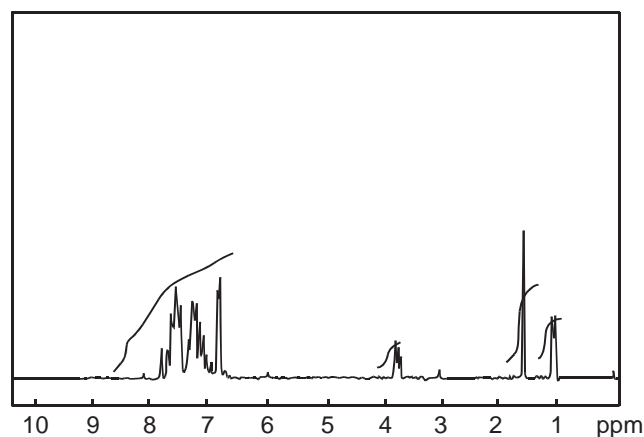


Figure 2. <sup>1</sup>H NMR Spectrum of PE(BPA+C).

**Table 3.** Characteristic data of the 300 MHz spectra of copolyesters.

PE(BPA+BPS)	PE(BPA+C)	Assignment
6.84-7.94 (m)	6.84-7.92 (m)	Aromatic protons (Ar-H)
1.05 (t)	1.06 (t)	Methyl protons of -CH <sub>2</sub> CH <sub>3</sub> group
3.78 (q)	3.81 (q)	Methylene protons of -CH <sub>2</sub> CH <sub>3</sub> group
1.51 (s)	1.52 (s)	Methyl protons propyl link

**PE(BPA+BPS)**

The <sup>1</sup>H-NMR spectra of PE(BPA+BPS) shows chemical shift  $\delta$  at 6.84-7.94 ppm may be due to the presence of aromatic protons. 3 H due to methyl group and 2 H due to methylene group of ethyl appear around 1.05 and 3.78 ppm, respectively. 6 H due to methyl protons of isopropyl groups produces singlet at  $\delta$  1.51 (s) ppm.

**PE(BPA+C)**

The <sup>1</sup>H NMR spectra of PE(BPA+C) shows chemical shift  $\delta$  at 6.84-7.92 ppm may be due to the presence of aromatic protons. 3 H due to methyl group and 2 H due to methylene group of ethyl appear around 1.06 and 3.81 ppm, respectively. 6 H due to methyl protons of isopropyl groups produces singlet at  $\delta$  1.52 (s) ppm.

**Thermal Decomposition Characteristics**

All the thermograms of copolyesters have been obtained on the thermobalance Mettler TA-4000 system at a heating rate 10°C/min in the temperature range from ambient temperature to 800°C. Thermograms obtained at a scan rate of 10°C/minute for all the copolyesters. Thermograms of some copolyesters are shown in Figure 3.

The characteristics temperatures for assessment of relative thermal stability of polymers such as initial decomposition temperature  $T_0$ , temperature for 10% weight loss,  $T_{10}$ , temperature for maximum rate of decomposition,  $T_{max}$ , and temperature for half volatilization,  $T_f$  are presented in Table 4. The higher the value of  $T_{10}$  [22], the greater is the thermal stability of a polymer. The thermal stability order of copolyesters on the basis of initial decomposition temperature  $T_0$  is as follows:

PE(BPA+DHA-1,4) > PE(BPC+R) > PE(BPA+C) > PE(BPA+BPC) = PE(BPA+DHA-1,8) > PE(BPC+Ph) = PE(Ph+DHA-1,8) > PE(BPA+BPS)

A comparison of  $T_{10}$  for copolyesters indicates the

following decreasing order of stability:

PE(BPA+DHA-1,4) > PE(BPA+C) > PE(BPC+R) > PE(BPA+BPC) > PE(BPA+DHA-1,8) > PE(BPC+Ph) > PE(BPA+BPS) > PE(Ph+DHA-1,4)

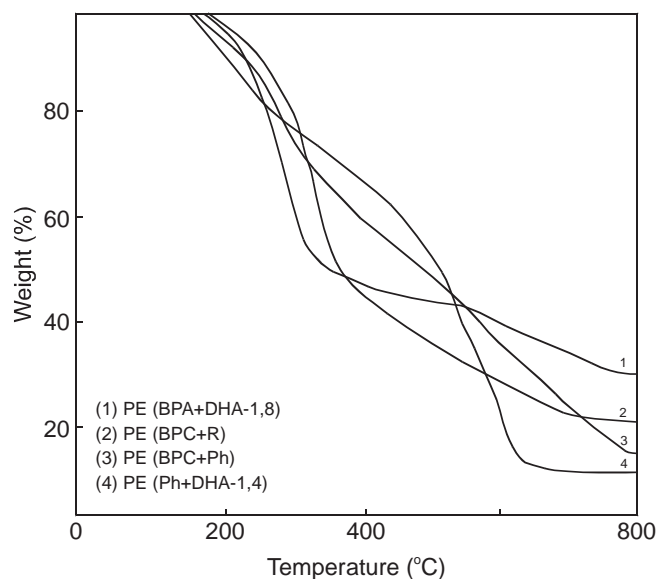
A comparison based on  $T_{max}$  for copolyesters indicates the following order:

PE(BPC+R) > PE(BPA+BPC) > PE(BPA+BPS) = PE(BPA+C) > PE(BPA+DHA-1,4) > PE(BPC+DHA-1,8) > PE(BPC+Ph)

The observations of above thermal stability trends shows that the copolyesters involving resorcinol, catechol, and DHA-1,4 have higher stability than copolyester involving phenolphthalein.

Values of activation energy ( $E_a$ ) were calculated according to the Broido's method [23], which range between 4.98 and 17.75 kcal/mol for the thermal decomposition of copolyesters.

These studies reveal that, the thermal stability of copolyester is significantly related to the aromatic diol component in the molecular chain. They are directly proportional to the molecular size of the diol component.

**Figure 3.** Thermal gravimetric analysis.

**Table 4.** Physical Characteristics of copolyesters.

Polymer	$T_0$ (°C)	$T_{10}$ (°C)	$T_{max}$ (°C)		$T_s$ (°C)	Activation energy (kcal/mol)	
			Step I	Step II		Step I	Step II
PE(BPA+BPC)	190	260	305	570	510	11.31	9.31
PE(BPA+BPS)	150	240	300	470	480	9.59	6.53
PE(BPA+DHA-1,4)	210	280	295	675	630	13.18	11.75
PE(BPA+DHA-1,8)	190	250	285	670	340	17.75	6.90
PE(BPA+C)	200	275	300	705	640	12.59	11.28
PE(BPC+Ph)	180	245	275	-	485	14.00	-
PE(BPC+R)	205	270	320	605	360	15.80	10.09
PE(Ph+DHA-1,4)	180	220	280	550	510	4.98	13.20

## CONCLUSION

Introduction of s-triazine rings and the flexibilizing linkages into the backbone of wholly aromatic copolyesters affords soluble polymers with high thermal stability. Thus, these polymers can be considered as promising, processable, high-temperature resistant polymeric materials.

## ACKNOWLEDGMENTS

The authors express their sincere gratitude to the Head of the Department of Chemistry, Veer Narmad South Gujarat University, Surat for providing necessary research facilities.

## SYMBOLS AND ABBREVIATIONS

$\eta_{sp}/c$	Reduced viscosity
D	Density
$E_a$	Activation energy
$T_0$	Initial decomposition temperature
$T_{10}$	Temperature at 10% weight loss
$T_{max}$	Maximum rate of decomposition temperature
$T_s$	Temperature at 50% weight loss
BPA	Bisphenol-A
BPC	Bisphenol-C
BPS	Bisphenol-S
Ph	Phenolphthalein

R	Resorcinol
C	Catechol
DMF	Dimethylformamide
IR	Infrared
NMR	Nuclear Magnetic Resonance
TGA	Thermogravimetric Analysis
DHA-1,8	1,8-dihydroxy Anthraquinone
DHA-1,4	1,4-dihydroxy Anthraquinone
EAPCCT	2-( <i>N</i> -ethyl anilino)-4,6-bis(phenoxy-2-carbonyl chloride)-s-triazine
EADCT	2-( <i>N</i> -ethyl anilino)-4,6-dichloro-s-triazine
EACPT	2-( <i>N</i> -ethyl anilino)-4,6-bis(2-carbophenoxy)-s-triazine
PE(BPA+BPS)	Copolyester of (Bisphenol-A+Bisphenol-S)
PE(BPA+BPC)	Copolyester of (Bisphenol-A+Bisphenol-C)
PE(BPA+Ph)	Copolyester of (Bisphenol-A+Phenolphthalein)
PE(BPA+C)	Copolyester of (Bisphenol-A+Catechol)
PE(BPA+R)	Copolyester of (Bisphenol-A+Resorcinol)
PE(BPC+Ph)	Copolyester of (Bisphenol-C+Phenolphthalein)
PE(BPC+R)	Copolyester of (Bisphenol-C+Resorcinol)
PE(Ph+DHA-1,4)	Copolyester of (Phenolphthalein+1,4-dihydroxyanthraquinone)
PE(BPA+DHA-1,8)	Copolyester of (Bisphenol-A+1,8-dihydroxy anthraquinone)
PE(BPA+DHA-1,4)	Copolyester of (Bisphenol-A+1,4-dihydroxy anthraquinone)



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