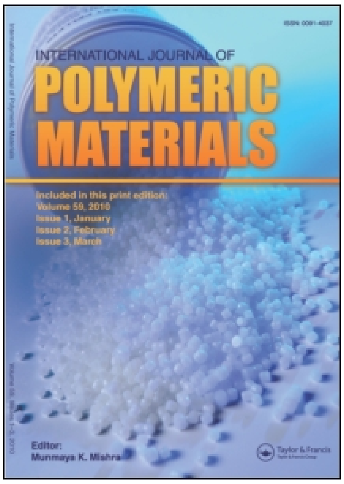

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Novel Copolyesters Based on *s*-Triazine Derivatives

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Ten copolyesters have been synthesized by polycondensation of MPNCCT and different mixtures of diols, such as: BPA + BPC, BPA + BPS, BPA + 1,4-DHA, BPA + Ph, BPA + C, BPA + R, BPA + EG, BPC + Ph, BPC + R, and Ph + 1,4-DHA. DHA. All the copolyesters have been characterized by IR spectra, NMR spectra, solubility, density and viscosity measurements and thermogravimetric analysis, TGA.

Keywords: copolyester, polycondensation, *s*-triazine, solubility, thermogravimetric analysis

SYMBOLS AND ABBREVIATIONS

η_{sp}/c	Reduced viscosity
D	Density
E_A	Activation energy
T_0	Initial decomposition temperature
T_{10}	Temperature for 10% weight loss
T_{max}	Maximum rate of decomposition temperature
T_s	Temperature for 50% weight loss
MPNCCT	2-(N-methylpiperazine)-4,6-bis(2-naphthoxy-6-carbonyl chloride)-s-triazine

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PE(BPA + BPC)	Copolyester of (Bisphenol-A + Bisphenol-C)
PE(BPA + Ph)	Copolyester of (Bisphenol-A + Phenolphthalein)
PE(BPA + 1,4-DHA)	Copolyester of (Bisphenol-A + 1,4-dihydroxy anthraquinone)
PE(BPA + C)	Copolyester of (Bisphenol-A + catechol)
PE(BPC + R)	Copolyester of (Bisphenol-C + resorcinol)
PE(BPC + 1,5-DHA)	Copolyester of (Bisphenol-C + 1,5-dihydroxy anthraquinone)
PE(Ph + 1,4-DHA)	Copolyester of (Phenolphthalein + 1,4-dihydroxy anthraquinone)

INTRODUCTION

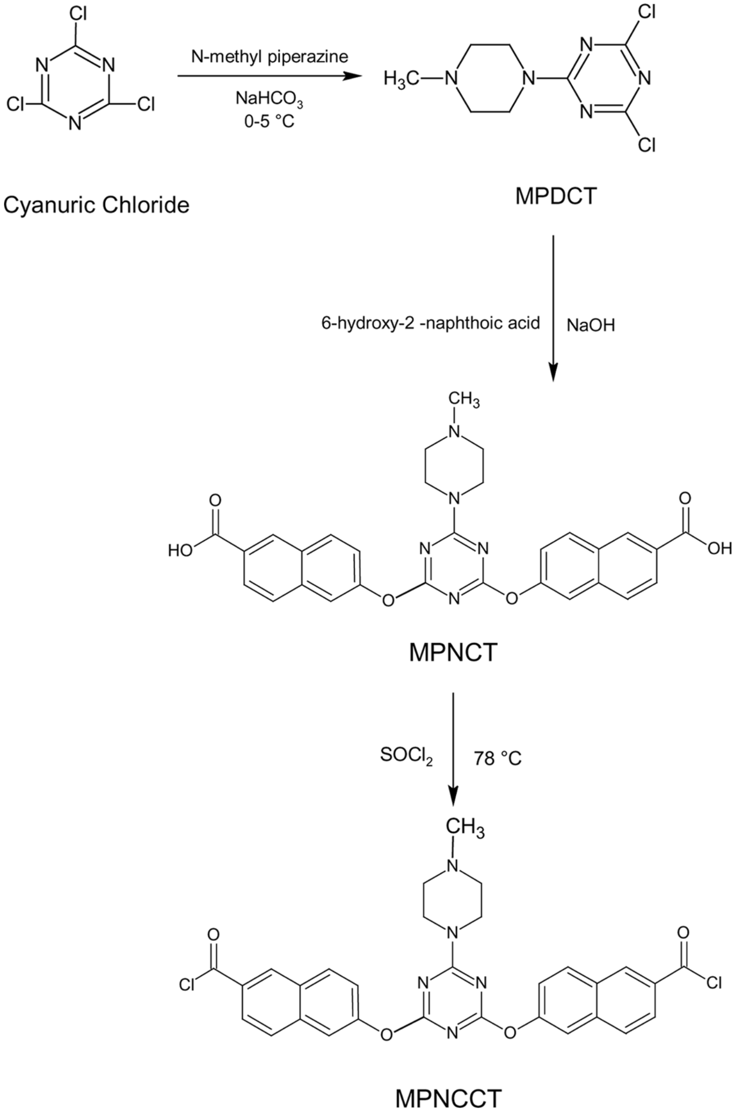
A survey of the literature reveals that polymers based on s-triazine and their derivatives have been extensively studied and utilized since the 1950 s. During the last 30 years, polymer chemists and chemical engineers have greatly improved their ability to make broad ranges of polymer structures. Great attention has recently been paid to synthesize high-performance polymers with increased thermal stability and heat resistance with good mechanical properties, as they are required in modern technological applications [1,2]. A major part of the synthetic polymers used in various fields today are copolymers and a lot of research work in this field is being carried out, too.

A large amount of research work and activities has been undertaken to synthesize new copolyesters and to modify them for desired properties and applications [3–7].

Aromatic polyesters containing s-triazine ring in the main chain are known for their excellent high-temperature properties and several are commercially available as high-performance engineering plastic materials [8–11]. Hence, it was thought interesting to synthesize and characterize some copolyesters based on *s*-triazine. As an approach to improve the stability and processability of heterocyclic polymers while marinating thermal stability, a number of published reports from our laboratory have described the synthesis and properties of *s*-triazine ring containing polymers, e.g., polycyanurates such as polyesters [12–16], polyamides [17–19], and, others. To continue our work, the present investigation describes the synthesis of some novel aromatic copolyesters by condensation of diacid chloride, 2-(N-methyl piperazine)-4, 6-bis(2-naphthoxy-6-carbonyl chloride)-s-triazine [MPNCCT], with various equimolar mixtures of aromatic diols.

MATERIALS

The monomer, 2-(N-methyl piperazine)-4,6-bis(2-naphthoxy-6-carbonyl chloride)-s-triazine [MPNCCT], was prepared as per the reaction Scheme



SCHEME 1 Synthesis of monomer 2-(N-methyl piperazine)-4,6-bis(2-naphthoxy-6-carbonyl chloride)-*s*-triazine [MPNCCT].

in Scheme 1. N-methyl piperazine of purity greater than 99% was received from Enzal Chemicals Ltd., Panoli, Ankleshwar. 6-hydroxy-2-naphthoic acid of purity greater than 99% was received from Atul Ltd., Atul, Valsad. All other chemicals, such as thionyl chloride, sodium bicarbonate and sodium hydroxide pellets, were used as received.

EXPERIMENTAL

Synthesis of Monomer

The monomer, 2-(N-methyl piperazine)-4,6-bis(2-naphthoxy-6-carbonyl chloride)-s-triazine [MPNCCT], was prepared as per the reaction Scheme 1. The procedure adopted for synthesis is described below.

(1) 2-(N-methyl piperazine)-4,6-dichloro-s-triazine [MPDCT]

A solution of cyanuric chloride (18.44 g, 0.1 mol) in 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 cyanuric chloride slurry. A solution of N-methyl piperazine (11.1 mL, 0.1 mol) in 10 mL acetone was added to this cold slurry with continuous stirring. After addition was completed, the reaction mixture was stirred for two hours at 0–5°C. The white-colored product was filtered and recrystallized from ethanol and dried in a vacuum desiccator (yield 90% and M.P. 125°C).

If the reaction is carried out in water, the reaction is left incompleted 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 [20].

(2) 2-(N-methyl piperazine)-4,6-bis(2-naphthoxy-6-carboxy)-s-triazine [MPNCT]

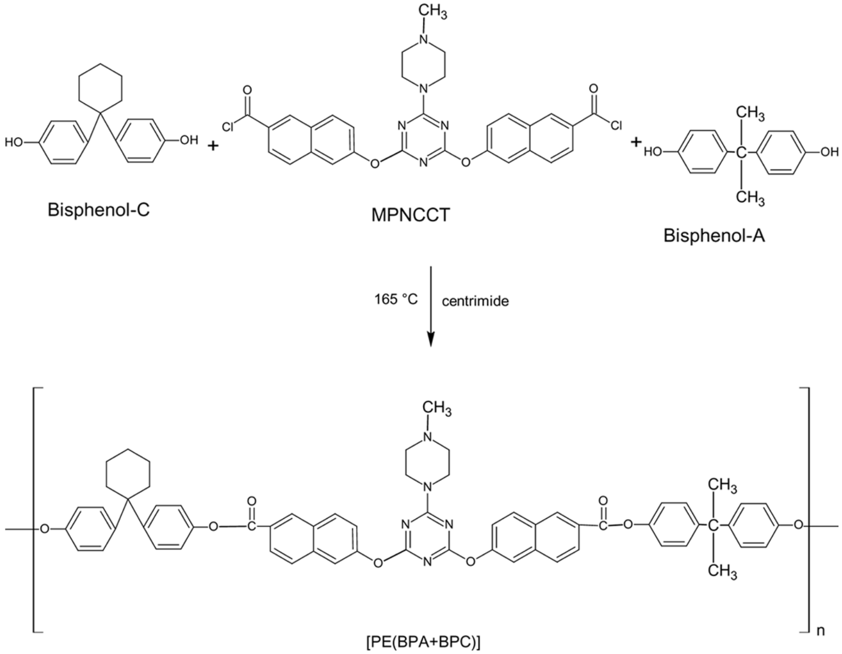
To 2-(N-methyl piperazine)-4,6-dichloro-s-triazine (MPDCT) (24.8 g, 0.1 mol) in 40 mL of acetone, a mixture of NaOH (16.0 g, 0.4 mol) and 6-hydroxy-2-naphthoic acid (37.6 g, 0.2 mol) in 80 mL double-distilled water was added slowly at room temperature with constant stirring. Reaction was continued for 4 h: First 2 h at room temperature and then another 2 h at 80°C. At the end of the reaction, a white solid separated from the reaction mixture that 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 85% with M.P. 220°C.

(3) 2-(N-methyl piperazine)-4,6-bis(2-naphthoxy-6-carbonyl chloride)-s-triazine [MPNCCT]

Thionyl chloride (11.9 mL, 0.1 mol) was added to 2-(N-methyl piperazine)-4,6-bis(6-naphthoxy-2-carboxy)-s-triazine (MPNCT) (5.51 g, 0.01 mol) and the reaction mixture was refluxed at 78°C for 2 h. At the end of the reaction the excess thionyl chloride was distilled off and dry product was recovered. The yield was about 65% and recrystallized from dimethylformamide (M.P. 180°C).

Synthesis of Copolyesters

A mixture of MPNCCT (5.88 g, 0.01 mol) in minimum quantity of DMF (~10 mL) was heated to about 150°C. Then cetrimide (0.25 g) and a mixture of diols [bisphenol-A (1.14 g, 0.005 mol) + bisphenol-C (1.34 g, 0.005 mol)] were added to it. The reaction temperature was raised to 165°C and heated for 8 h. The reaction mixture was cooled and poured with constant stirring in 50 mL of ice-cold water. The solid was filtered, washed with hot water and then with methanol to remove unreacted monomer, and dried. The yield was about 80%.



SCHEME 2 Synthesis of copolyester of (Bisphenol-A + Bisphenol-C).

The other copolyesters from MPNCCT (5.88 g, 0.01 mol) and various diol mixtures such as bisphenol-A(BPA) + bisphenol-S(BPS), BPA + 1,4-dihydroxy anthraquinone, BPA + 1,8-dihydroxy anthraquinone, BPA + phenolphthalein, BPA + catechol, BPA + resorcinol, BPA + ethylene glycol, bisphenol-C(BPC) + resorcinol, BPC + phenolphthalein and phenolphthalein + 1,4-dihydroxy anthraquinone, were synthesized by the same method as shown in the scheme in Scheme 2.

RESULTS AND DISCUSSION

The yield of copolyesters varies from 65–90% depending upon the reactivity of the diol components. Most of the copolyesters are gray to black. All the copolyesters were obtained in powdery form. The yield and color of copolyesters depend upon the color, nature and structure of the diol component. The color and yield and density are presented in Table 1.

Solubility Characteristics

30 to 50 mg sample of finely ground polymer was placed into a small test tube and 1 mL of solvent was added to each. The mixtures were stored at 25°C for some time with occasional shaking. Formation of streaks while shaking indicated dissolution. Polymer samples that swelled without dissolving at 25°C were heated to 50°C in order to affect the process of dissolution. Several solvents were tested for this purpose as shown in Table 2. The presence of solubility can give much information about the polymer [21–23].

TABLE 1 Yield, Color and Density of Copolyesters

Polymer	Yield %	Color	Density (g/cm^3)
PE(BPA + BPC)	85	Gray	1.227
PE(BPA + BPS)	65	light brown	1.165
PE(BPA + DHA-1,4)	70	light brown	1.211
PE(BPA + Ph)	75	brown	1.195
PE(BPA + C)	90	black	1.189
PE(BPA + R)	90	brown	1.204
PE(BPA + EG)	85	gray	1.179
PE(BPC + Ph)	70	brown	1.220
PE(BPC + R)	75	brown	1.171
PE(Ph + DHA-1,4)	70	gray	1.185

TABLE 2 Solubility of Copolyesters in Various Solvents

Solvent	PE(BPA + BPC)	PE(BPA + BPS)	PE(BPS + DHA-1,4)	PE(BPA + Ph)	PE (BPA + C)	PE (BPA + R)	PE(BPA + EG)	PE(BPC + Ph)	PE (BPC + R)	PE(Ph + DHA-1,4)
$(CH_3)_2CO$	−±	−−	−−	−±	−−	−±	−±	−−	−±	−−
C_6H_6	−−	−−	−−	−−	−−	−−	−−	−−	−−	−−
C_6H_5Cl	−−	−−	−−	−−	−−	−−	−−	−−	−−	−−
$CHCl_3$	−−	−−	−−	−−	−−	−−	−−	−−	−−	−−
CCl_4	−−	−−	−−	−−	−−	−−	−−	−−	−−	−−
DMAc	++	++	±+	±+	±+	++	±+	±+	++	++
DMF	++	++	++	++	++	++	++	++	++	++
CH_3COOEt	±+	++	±+	±+	±+	++	−±	±+	±+	−±
1,4-Dioxane	±+	−±	±+	++	±+	±+	±+	±+	±+	±+
Petro. ether	−±	−±	−−	−−	−−	−−	−−	−−	−−	−−
Methanol	−−	−−	−−	−−	−−	−−	−±	−±	−−	−−
Ethanol	−−	−−	−±	−−	−−	−±	−±	−±	−−	−−

The first and second symbol indicates the solubility of polymer at R.T. and 50°C, respectively.
+ = Soluble, − = Insoluble, ± = Partly soluble.

Density Measurements

Density of polyesters have been determined at 25 ± 3°C by use of powdered sample [24]. Density of each of the ten copolyesters was determined at 25 ± 1°C by suspending each of the copolyesters in a liquid mixture of carbon tetrachloride and petroleum ether and subsequently measuring the density of the liquid mixture by a pyknometer, are presented in Table 1. This liquid system was found to be inert to all the copolyesters since they remained in a state of suspension even for a prolonged time. Estimated accuracy of density, is $\pm 0.001\,\mathrm{g/cm^3}$. Density of all the copolyesters varies from 1.227 to $1.165\,\mathrm{g/cm^3}$. The density of copolyesters varies with the different diols used, and decreased in the following order:

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

Viscosity Measurements

Dilute solution viscosity measurements were carried out using Ubbelohde suspended level kinemetic viscometer. The polymer solutions were prepared in dimethylformamide and were filtered through G-3 sintered glass funnel prior to flow time measurements. Intrinsic, reduced and inherent viscosity along with Huggins and Kraemer constants for 1% solution, for all the copolyesters are given in Table 3.

TABLE 3 Various Viscosity Values and Constants of Huggins and Kraemer Equation for Polyesters

Polymer	Intrinsic viscosity η	Reduced viscosity η_{sp}/C (dl/g)	Inherent viscosity $\ln \eta_{rel}/C$	Huggins constant K	Kraemer constant β
PE(BPA + Ph)	0.570	0.672	0.514	0.302	0.180
PE(BPA + BPC)	0.567	0.665	0.510	0.294	0.186
PE(BPC + R)	0.551	0.651	0.501	0.329	0.164
PE(BPA + DHA-1,4)	0.448	0.521	0.448	0.363	0.144
PE(BPA + C)	0.534	0.632	0.490	0.344	0.154
PE(BPA + R)	0.474	0.611	0.477	0.327	0.165
PE(BPA + EG)	0.442	0.510	0.412	0.348	0.153
PE(BPC + Ph)	0.502	0.591	0.464	0.353	0.151
PE(BPA + BPS)	0.471	0.552	0.439	0.347	0.158
PE(Ph + DHA-1,4)	0.515	0.611	0.477	0.362	0.143

Conc. of polymer solution is 1.0 g/dl.

The intrinsic viscosity of the copolyesters obtained from MPNCCT and various diol mixtures follows the sequence given below:

$$\begin{gathered}\mathrm{PE(BPA+Ph)} > \mathrm{PE(BPA+BPC)} > \mathrm{PE(BPC+R)} > \mathrm{PE(BPA+C)} > \\ \mathrm{PE(Ph+DHA\text{-}1,4)} > \mathrm{PE(BPC+Ph)} > \mathrm{PE(BPA+R)} > \\ \mathrm{PE(BPA+BPS)} > \mathrm{PE(BPA+DHA\text{-}1,4)} > \mathrm{PE(BPA+EG)}\end{gathered}$$

Infrared Spectroscopy

The expected spectral features of copolyesters synthesized from MPNCCT and diol mixtures have been assigned. An inflection at 3050–3085 cm^{-1} is attributed to aromatic nucleus C−H stretching. IR spectra of all the copolyesters exhibit a band at 720–795 cm^{-1}, 840–890 cm^{-1} may be attributed to the bending vibration of the C−H bonds of the aromatic ring. Several bands at 800–840 cm^{-1} may be due to the out of plane vibration of the s-triazine ring. The presence of s-triazine ring is further supported by the appearance of a band at 1440–1510 cm^{-1}, which is due to the in-plane bending vibration of the s-triazine ring. The bands observed around 1020–1050 cm^{-1} are attributed to the vibration of aryl-ether linkage and 1080–1110 cm^{-1} may be due to in-plane bending vibration of aromatic C−H. The presence of aryl-ether linkage is further supported by the appearance of a band at 1220–1240 cm^{-1}. The presence of a band due to the stretching of the C−O−C system of ester and –COOH group is anticipated in the region 1300 cm^{-1} to 1100 cm^{-1}, depending upon the structure. In the spectra of all the copolyesters, two bands are observed, one at 1140–1160 cm^{-1} and another at 1260–1300 cm^{-1}, which is attributed to symmetric and asymmetric stretching vibration of C−O−C group. It is not possible to assign these bands to C−O−C of acid or ester group. In light of literature reports the above positions of the bands are attributed to C−O−C stretching. A band at 1340–1380 cm^{-1} may be due to aromatic C−N stretching linkage (N-containing six-member ring). The presence of bands around 1700–1740 cm^{-1} indicates the presence of the ester carboxyl group. Such a band is observed in all the copolyester samples around 1720–1740 cm^{-1}, which confirms the presence of the ester carboxyl group. The bands observed at 2845–2850 cm^{-1}(symmetric) and 2915–2925 cm^{-1} (asymmetric) are due to C−H stretching vibration of the –CH_2 group. The presence of a band around 3050–3085 cm^{-1} indicates the presence of aromatic C−H stretching vibration and a band around 3380–3450 cm^{-1} is due to stretching vibration of O−H (bonded). IR spectra of copolyesters involving BPA exhibit a distinct band at 550–580 cm^{-1} confirming the presence of C–C deformation vibration of propyl link due to bisphenol-A, while copolyesters involving BPC exhibit two bands at 540 cm^{-1} and around 960–965 cm^{-1} due to deformation vibration of cyclohexane ring of bisphenol-C. The spectra of PE(BPA + Ph), PE(BPC + Ph), and PE(Ph + DHA-1,4) exhibit a strong band at 1720–1750 cm^{-1} due to the C–O stretching vibration of lactone ring, of phenolphthalein in polymer chain. Spectral distinction between other polymers like PE(BPA + Ph), PE(BPA + C), PE(BPC + Ph), and PE(Ph + DHA-1,4) exhibit a band at 700–730 cm^{-1}, confirming the presence of o-disubstituted phenyl ring, and the polymer PE(BPC + R) exhibits a band at 800 cm^{-1} due to m-disubstituted phenyl ring. All the IR spectral characteristics of different copolyester samples are in good agreement with expected IR frequencies of the proposed structures.

NMR Spectroscopy

High-resolution (300 MHz) ^{1}H-NMR spectra of solution of representative copolyester sample were measured in deuterated dimethylsulphoxide (DMSO-d_6) using TMS as an internal reference. The ^{1}H-NMR spectra of PE(BPA + BPS) shows chemical shift δ at 6.40–8.90 ppm that may be due to the presence of aromatic protons, 8 H of methylene groups of piperazine ring produced multiplet at δ 2.40–3.35 ppm, 3 H of –NCH_3 of piperazine produced singlet at δ 2.25 ppm, and 6 H due to isopropyl group the produced a singlet at δ 1.50 ppm.

Thermogravimetric Analysis

The thermal degradation of polymer depends upon various variables. Important variables include decomposition temperature, heating rate, rate of removal of volatile products from the reaction zone, softening and melting points of the polymer, sample size and dimensions, presence of oxygen in the atmosphere or absorbed in the sample or other occluded impurities, the initiators used and the mechanism of termination, extraneous units copolymerized into the polymer and many other factors. In order to identify the generalized mechanism, therefore, thermal decomposition was carried out under inert atmosphere with carefully purified polymers in the form of finely divided powder.

Qualitative Estimation of Thermal Stability

Several temperature characteristics used for the qualitative assessment of relative thermal stability of polymers are initial decomposition temperature (T_0), for 10% weight loss (T_{10}), temperature for maximum

TABLE 4 Thermal Characteristics of Various Polyesters

Polymer	T_0	T_{10}	Tmax Step I	 Step II	T_s
PE(BPA + BPC)	235	265	290	–	335
PE(BPA + EG)	195	220	280	–	345
PE(BPC + Ph)	185	305	350	–	465
PE(Ph + DHA-1,4)	240	290	315	460	500

rate of decomposition (T_{max}) and temperature from half volatilization (T_s). These characteristics are presented in Table 4.

Thermal stability of the copolyesters on the basis of initial decomposition temperature T_0 is as follows:

$$\mathrm{PE(Ph + DHA\text{-}1,4) > PE(BPA + BPC) > PE(BPA + EG) > PE(BPC + Ph)}$$

The thermal degradation of PE(BPA + BPC) involves one-step decomposition. The copolyester begins to decompose at 235°C. The decomposition is marked with rapid weight loss in the temperature range 240–350°C. The maximum rate of weight loss occurs at 335°C. After 500°C, the sample degrades slowly up to the end temperature and leaves about 3.5% residue at 800°C.

The overall degradation of copolyester PE(BPA + EG) involves one-step decomposition. The decomposition begins at about 195°C. The maximum rate of weight loss occurs at 280°C. After 400°C, the sample degrades slowly up to the end temperature and leaves about 5% residue.

The overall degradation of copolyester PE(BPC + Ph) involves single-step decomposition. The decomposition is marked with rapid weight loss in the temperature range 330–425°C, involving 32% weight loss, with a maximum rate of weight loss at 350°C. The residue left is about 25% at 800°C.

Thermal degradation of PE(Ph + DHA-1,4) occurs in two distinct steps. The copolyester begins to decompose at 240°C. A rapid weight loss occurs in the temperature range 300–450°C, involving about 45% weight loss. The maximum rate of weight loss occurs at 285°C. The second decomposition step begins at 450°C and extends to about 800°C, with maximum rate of weight loss at 500°C. After 600°C, the sample degrades slowly up to the end temperature and leaves about 28% residue.

TABLE 5 Application of Broido Method to Thermogram of PE(BPA + BPC)

Temp.°C	% Wt.	$y = \frac{W_t - W_\alpha}{W_t - W_o}$	1/y	ln ln 1/y	T °K	$1/T \times {}^\circ K^{-1}$
250	96.00	0.9593	1.0424	−3.1810	523	1.9120
260	94.00	0.9386	1.0654	−2.7590	533	1.8761
270	90.00	0.8972	1.1351	−2.0657	543	1.8416
280	86.50	0.8610	1.1614	−1.8996	553	1.8083
290	81.00	0.8041	1.2436	−1.5232	563	1.7762
300	78.50	0.7782	1.2850	−1.3832	573	1.7452
310	73.00	0.7213	1.3864	−1.1187	583	1.7152
320	64.00	0.6282	1.5918	−0.7660	593	1.6863
330	49.75	0.4808	2.0799	−0.3115	603	1.6584

Evaluation of Kinetic Parameters

Dynamic TGA thermograms obtained at heating rate 10°C/min have been analyzed as per the graphical methods, proposed by Broido and Horowitz-Metzger. A typical application of Broido method [25] and Horowitz and Metzger method [26] are exemplified for PE(BPA + BPC) in Tables 5 and 6 respectively.

Since the magnitude of an apparent activation energy for degradation reaction reflects the ease with which it can proceed, it may be used to approximate relative ease of thermal degradation of copolyesters and hence, their relative thermal stability.

The thermograms of other copolyesters were also analyzed in the same graphical method. The values of apparent activation energy corresponding to different decomposition steps involved were evaluated from the slope of the least square plots of the relevant data and are presented in Table 6.

Examination of the data presented therein reveals that both methods give comparable values for activation energy. The experimental points corresponding to initial stage (10% loss) of thermal degradation

TABLE 6 Activation Energy (E) Calculated by Broido and Horowitz and Metzger Method

	Activation energy E(K.Cal/mol)			
	Broido		Horowitz-Metzger	
Polymer	Step I	Step II	Step I	Step II
PE(BPA + BPC)	20.75	–	21.27	–
PE(BPA + EG)	16.45	–	15.64	–
PE(BPC + Ph)	12.54	–	11.47	–
PE(Ph + DHA-1,4)	22.81	17.80	23.31	16.66

tended to deviate from linearity. This deviation may be due to the fact that the decomposition of solids does not obey first order kinetics in the initial stages.

Between the two methods, the Broido method is expected to provide reliable estimates of E, since no other temperature characteristics are involved. The values of activation energy (E_a) vary from 12.54 to 20.75 KCal/mole according to Broido method. The values of activation energy calculated according to the Horowitz and Metzger method are in good agreement with these values. The activation energy of PE(BPA + BPC) is lower than the other copolyesters. Further, this trend of relative stability indicates that the initiation of site of degradation may lie in the nature of the bridge X in the aromatic diol component of the molecular chain. Thus, results based on qualitative and semiquantitative treatment of dynamic thermogravimetric data of copolyesters point out that inclusion of bisphenol in a copolyester backbone is advantageous, so far as the thermal stability requirement is concerned. Moreover, such an inclusion does not adversely affect the stability of the resultant copolyesters.

CONCLUSION

A series of novel copolyesters containing *s*-triazine rings in the main chain was successfully synthesized by high temperature polycondensation. The introduction of *s*-triazine ring into the wholly aromatic polyesters afforded soluble polymers with highly thermal stability. Thus, these polyamides can be considered a promising, processable, highly temperature–resistant polymer materials.

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