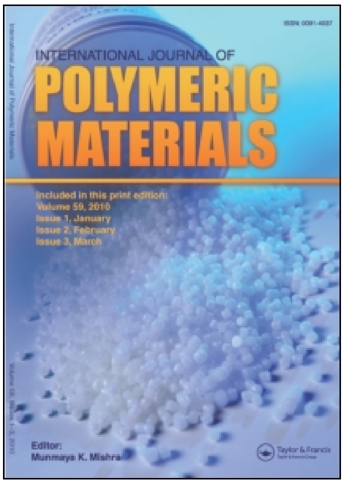

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Synthesis and Physicochemical Studies of Some Novel Homopolyesters Based on *s*-Triazine

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Synthesis and Physicochemical Studies of Some Novel Homopolyesters Based on *s*-Triazine

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The diacid chloride 2-(N-methyl piperazine)-4,6-bis(2-naphthoxy-6-carbonyl chloride) [MPNCCT] has been synthesized, from which different homopolyesters have been made by polycondensation with the following diols: bisphenol-A [BPA], bisphenol-C [BPC], bisphenol-S [BPS], 1,4-dihydroxy anthraquinone [1,4-DHA], 1,8-dihydroxy anthraquinone [1,8-DHA], catechol [C], resorcinol [R], phenolphthalein [Ph], ethylene glycol [EG] and diethylene glycol [DEG]. All the synthesized polyesters were characterized by solubility, density, viscosity measurements, IR spectra, NMR spectra and thermogravimetric analysis [TGA].

Keywords density, homopolyesters, physicochemical, solubility, synthesis, viscosity

INTRODUCTION

In our previous work we have synthesized polyesters based on *s*-triazine derivatives and studied their various physicochemical properties [1–5]. The aim of the present work is to extend this work.

A great deal of research has been carried out on thermally stable polymers in which aromatic and heterocyclic rings are linked together in the main chain. Aromatic polyesters containing the *s*-triazine ring in the main chain

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are known for their excellent high temperature properties, and several were commercialized as high-performance engineering plastic materials [6,7]. Today, they are one of the most important classes of polymer as they are important in applications such as laminating resins, molding composites, fibers, films, surface coatings resins, rubbers and plasticizers. The linear polyesters of high molecular weight make synthetic fibers of excellent properties [8]. Many aromatic polyesters [9–11] have been synthesized from various types of bisphenols, and polyesters derived from bisphenol-A and isophthaloyl chloride (IPCl) or terphthaloyl chloride (TPCl) have been commercialized.

MATERIALS

Benzene, acetone, dimethylformamide (DMF) and solvents like triethylamine, petroleum-ether, carbon tetrachloride, methanol, ethanol, N-methyl-2-pyrrolidone (NMP), N,N′-dimethylacetamide (DMAC), dimethyl sulphoxide (DMSOL), m-cresol, sulphuric acid, and hexane were all laboratory reagents and were used as received from Merck. Double-distilled water was used for the preparation of solutions. *N*-methyl piperazine of purity greater than 99% was received from Enzal Chemicals Ltd., Panoli, Ankleshwar. Cyanuric chloride (Fluka) was purified by repeated crystallization from pure benzene. 6-hydroxy-2-naphthoic acid of purity greater than 99% was received from Atul Ltd., Valsad. Resin-grade bisphenol-A (Atul Ltd., Valsad) was repeatedly crystallized from 50% aqueous acetic acid and finally purified by recrystallization from benzene. Resorcinol (MERCK), catechol (Alfa Aesar), phenolphthalein (BDH), 1,4-dihydroxy anthraquinone (MERCK) and 1,8-dihydroxy anthraquinone (Merck) were used as received. Chemicals such as thionyl chloride, sodium bicarbonate, sodium hydroxide were used as received from Fluka.

EXPERIMENTAL

Synthesis of 2-(*N*-methylpiperazine)-4,6-bis(2-naphthoxycarbonylchloride)-*s*-triazine (MPNCCT)

*2-(*N*-methylpiperazine)-4,6-dichloro-*s*-triazine [MPDCT]*

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 (9.40 g, 0.1 mol) in 100 mL of distilled water. This resulted in the formation of a slurry of cyanuric chloride. To this, a solution of N-methyl piperazine (11.10 mL, 0.1 mol) in 10 mL acetone was added with continuous stirring. The reaction mixture was stirred for 2 h at 0–5°C. The white-colored product was filtered, recrystallized from ethanol and dried. The yield was 90%, MP 125–127°C.

*2-(*N*-methylpiperazine)-4,6-bis(2-naphthoxy-Carboxy)-*s*-triazine [MPNCT]*

To 24.8 g of MPDCT (0.1 mol) in 40 mL of acetone, a mixture of sodium hydroxide (16.0 g, 0.4 mol) and 6-hydroxy-2-naphthoic acid (37.60 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 a further 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. It was recrystallized from ethanol. Yield was 85%. MP 220–222°C.

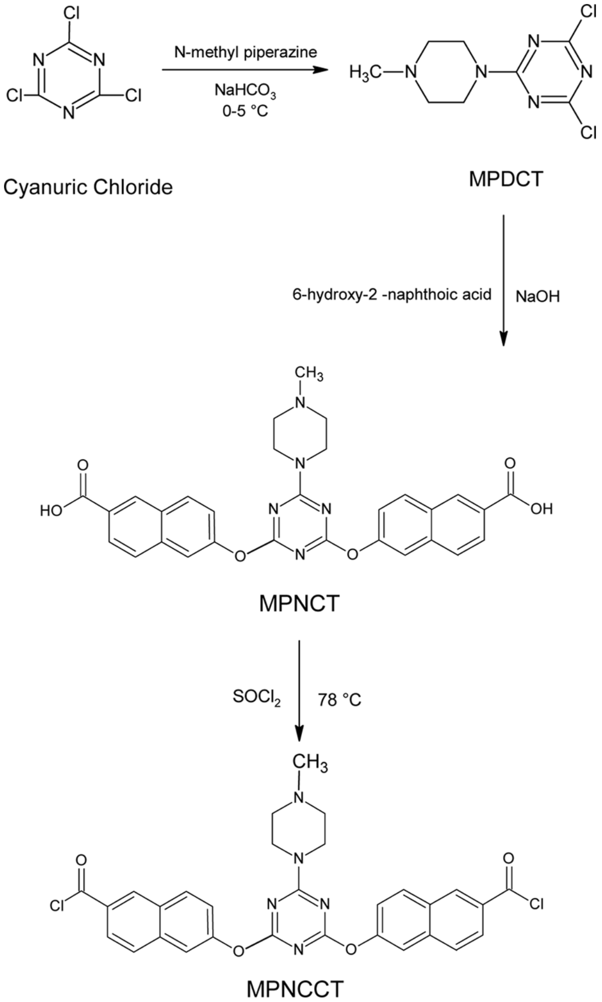

Figure 1: Synthesis of monomer 2-(*N*-methyl piperazine)-4,6-bis(2-naphthoxy-6-carbonyl chloride)-*s*-triazine (MPNCCT).

*2-(*N*-methylpiperazine)-4,6-bis(2-naphthoxy-Carbonylchloride)-*s*-triazine [MPNCCT]*

Thionyl chloride (11.90 mL, 0.1 mol) was added to 5.51 g MPNCT (0.01 mol) in a dry round-bottom flask, from 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 thionyl chloride was distilled off and the dry product was recovered. Yield was about 65%, MP 180–182°C (Figure 1).

Synthesis of Polyester of Bisphenol-A (PEBA)

A mixture of MPNCCT (5.88 g, 0.01 mol) in minimum quantity of DMF (~10 mL) was heated to about 150°C. Cetrimide (0.25 g) and then bisphenol-A (2.28 g, 0.01 mol) were added. The temperature was raised to 165°C and maintained there for 8–10 h. The reaction mixture was cooled and poured with constant stirring into ice-cold water. The solid was filtered, washed with hot water and then methanol and then dried. The yield was about 70%. Similarly, other polyesters from MPNCCT were synthesized following the same procedure using equimolar amounts of the diols BPC, BPS, hydroquinone, catechol, phenolphthalein, resorcinol, 1,4-DHA, 1,8-DHA, ethylene glycol and diethylene glycol (Figure 2).

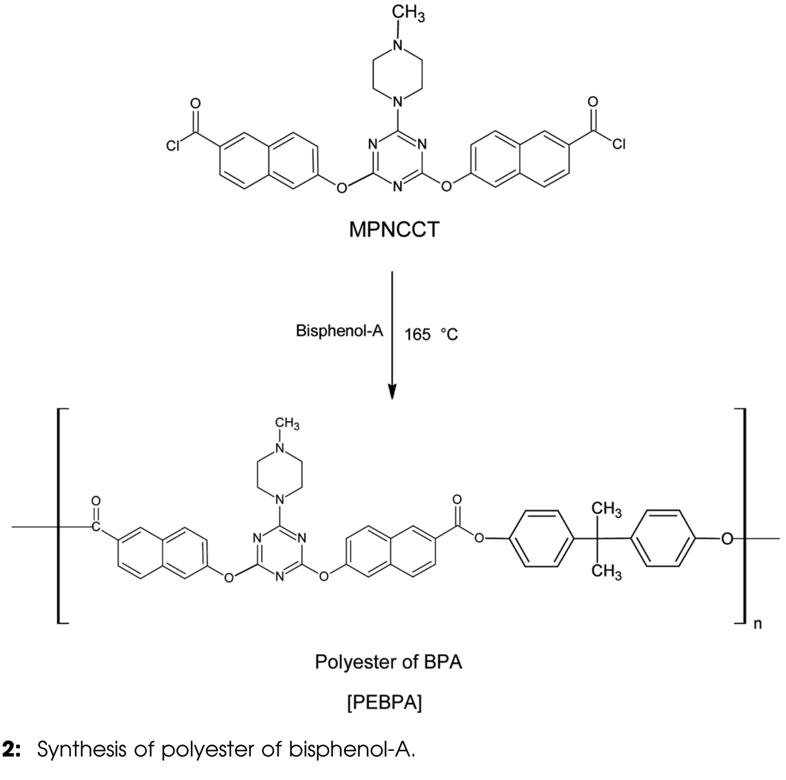

Figure 2: Synthesis of polyester of bisphenol-A.

MEASUREMENTS

Solubility tests were conducted with several solvents. Infrared spectra of the synthesized polymers have been measured using KBr pellets on a Perkin-Elmer FTIR Spectrometer Paragon-500. Nuclear magnetic resonance (NMR) spectroscopy of the polymers has been carried out using DMSO as a solvent and TMS as an internal standard. NMR spectra were recorded on a Perkin-Elmer Model-32 ^{1}H-NMR Spectrometer (300 MHz) for the structure elucidation. Densities of polyesters have been determined at $25 \pm 3°C$ by use of a powdered sample [12] and were determined with the aid of a Pyknometer using the suspension method. Viscosities of polyesters were conveniently measured using an Ubbelohde capillary viscometer [13]. The thermograms of polyesters have been obtained on a Mettler TA-4000 system thermobalance at a constant heating rate of 10°C/min in the temperature range from ambient temperature to 800°C.

RESULTS AND DISCUSSION

Various polyesters were synthesized by high temperature polycondensation of MPNCCT with different diols. The effects of variables such as temperature, the nature of organic solvents and reaction time were examined. The rest of the variables were maintained nearly constant for all the experiments.

Yield and Color

The yield of the polyesters varied from 65–85%. Highest yield of 85% was obtained for PEBPA and lowest 65% for PEBPS. All the polyesters were obtained in powdery form. The yields of the polyesters depend upon the reactivity and structure of the diol component used. The approximate yields and colors of the synthesized polyesters are presented in Table 1.

Table 1: Yield, color and density of polyesters.

Polymer	Yield %	Color	Density (g/cm^3)
PEBPA	85	brown	1.144
PEBPC	70	light yellow	1.135
PEBPS	65	light green	1.195
PEDHA-1,8	75	brown	1.188
PEDHA-1,4	70	brown	1.184
PEPh	70	black	1.204
PER	80	brown	1.189
PEC	75	black	1.191
PEEG	70	brown	1.124
PEDEG	70	brown	1.127

Density

The density of the polyesters was determined at 25 ± 3°C by suspending each in a liquid mixture of carbon tetrachloride and petroleum ether and subsequently measuring the density of the liquid mixture by a pyknometer. This liquid system was found to be inert to all polyesters since they remained in a state of suspension even for a prolonged time. Estimated accuracy of density is $\pm 0.001\,\mathrm{g/cm^3}$.

The sequence of density of polyesters is as follow:

$$\mathrm{PEPh} > \mathrm{PEBPS} > \mathrm{PEC} > \mathrm{PER} > \mathrm{PEDHA\text{-}1,8} > \mathrm{PEDHA\text{-}1,4}$$
$$> \mathrm{PEBPA} > \mathrm{PEBPC} > \mathrm{PEDEG} > \mathrm{PEEG}$$

Densities of the polyesters are presented in Table 1.

Solubility

Solubility results show that the methylated solvents are good solvents for all the polyesters. It was also noticed that solubility increases with the temperature and those polymers that have partial solubility at 25°C get dissolved at higher temperatures. Among these methylated solvents, DMF was found to be the best solvent, since under given conditions polyesters dissolved rapidly in dimethylformamide compared to other similar solvents. Aliphatic or aromatic chlorinated solvents are nonsolvents for polyesters. Solubility of polyesters in various solvents is shown in Table 2.

Viscosity

Typical Huggins [14] and Kraemer [15] plots were used to obtain intrinsic viscosity for the homopolyesters. Solution viscosity of PEBPA at different concentration is shown in Table 3. Flow time (t_0) for DMF solvent was found to be 127 sec.

Intrinsic, reduced and inherent viscosity along with Huggins and Kraemer constants for 1% solution for all the polyester reveal that PEBPA has the highest solution viscosity and probably, the highest molecular weight amongst the polyesters, whereas the PEEG has the lowest. Examination of reduced viscosity values reveals that, among DMF soluble polycyanurates, PEBPA has the highest whereas PEBPS has the lowest solution viscosity. The trend of these values reflects the order of relative reactivity of bisphenols. BPA is the least acidic, and hence most reactive towards nucleophilic displacement reaction, and hence leads to the formation of a comparatively higher molecular weight polymer, while, due to electronic effects, BPS is highly acidic and hence least reactive and leads to the formation of a lower molecular weight polymer.

Table 2: Solubility of polyesters in various solvents.

Solvent	PEBPA	PEBPC	PEBPS	PEDHA-1,8	PEDHA-1,4	PEPh	PER	PEC	PEEG	PEDEG
$(CH_3)_2CO$	− ±	− ±	− −	− ±	− ±	− −	− −	− ±	− ±	− −
C_6H_6	− −	− −	− −	− −	− −	− −	− −	− −	− −	− −
C_6H_5Cl	− −	− −	− −	− −	− −	− −	− −	− −	− −	− −
$CHCl_3$	− −	− −	− −	− −	− −	− −	− −	− −	− −	− −
CCl_4	− −	− −	− −	− −	− −	− −	− −	− −	− −	− −
DMAc	± +	± +	+ +	+ ±	± +	± +	+ +	± +	+ +	+ +
DMF	+ +	+ +	+ +	+ +	+ +	+ +	+ +	+ +	+ +	+ +
CH_3COOEt	+ +	+ +	+ +	± +	± +	± +	+ +	+ +	± ±	± ±
1,4-Dioxan	± +	+ +	± +	+ +	± +	+ +	− ±	− ±	± +	± +
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.

Table 3: Solution viscosity of polyester PEBPA.

Conc. (C) g/dl	Flow time t sec	$\eta_{rel} = t/t_o$	$\eta_{sp} = \eta_{rel} - 1$	η_{sp}/C	$\ln \eta_{rel}/C$
0.2	143.5	1.130	0.130	0.650	0.611
0.4	161	1.268	0.268	0.670	0.594
0.6	180	1.421	0.421	0.701	0.585
0.8	202	1.590	0.590	0.735	0.580
1.0	223	1.756	0.756	0.756	0.563

The intrinsic viscosity of the homopolyesters synthesized from MPNCCT and various diols, follows the sequence given below:

$$\text{PEBPA} > \text{PEBPC} > \text{PEPh} > \text{PEDHA-1,8} > \text{PEDHA-1,4} > \text{PER} > \text{PEC} > \text{PEBPS} > \text{PEEG} > \text{PEDEG}$$

Within a given reaction time, a highly active aromatic diol is expected to yield a higher molecular weight polymer than the least active one. Thus the reduced viscosity values reflect the order of relative reactivity in nucleophilic substitution reaction of these bisphenols. Intrinsic, reduced and inherent viscosities of all the polyesters and constants of the Huggins and Kraemer equation are shown in Table 4.

IR Spectroscopy

The IR spectra of all the synthesized polyesters exhibited several common characteristic absorption frequencies. Formation of polyesters was confirmed from the typical characteristic FTIR bands observed around 1700–1740 cm^{-1} ($>$C=O stretch in ester) [16]. In spectra of the polyesters the presence of the band around 3050–3085 cm^{-1} indicates the presence of aromatic C−H stretching vibration and a band around 3350–3450 cm^{-1} is due to the stretching

Table 4: Various viscosity values and constants of the 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
PEBPA	0.619	0.756	0.563	0.358	0.146
PEBPC	0.606	0.709	0.536	0.338	0.158
PEBPS	0.416	0.475	0.389	0.341	0.156
PEDHA-1,8	0.489	0.595	0.467	0.348	0.152
PEDHA-1,4	0.480	0.550	0.438	0.325	0.167
PEPh	0.561	0.693	0.526	0.362	0.128
PER	0.451	0.520	0.419	0.340	0.158
PEC	0.424	0.488	0.397	0.355	0.150
PEEG	0.346	0.375	0.318	0.333	0.168
PEDEG	0.330	0.356	0.304	0.327	0.183

*Concentration of solution is 1 g/dl for all polymers.

vibration of O–H (bonded). Strong or moderately strong absorptions at frequencies of 810–840 cm^{-1} and 1440–1510 cm^{-1} are attributed to out-of-plane and in-plane vibration of the *s*-triazine ring, respectively. A band at 1210–1240 cm^{-1} is attributed to vibration involving the aryl-ether linkage [17]. In addition to this, the spectra exhibited few other IR absorption frequencies, based on which these polyesters can be distinguished from each other.

In the spectrum of PEBPA, the band appearing at about 550 cm^{-1} is attributed to C–C deformation vibration of the propyl link, while in the spectrum of PEBPC the bands observed at 560 cm^{-1} and 970 cm^{-1} the attributed to C–C deformation vibration of the cyclohexane ring. In spectrum of PEBPS the band observed at 1150 cm^{-1} and 1352 cm^{-1} is attributed to stretching of $-SO_2$ group in the chain [18]. In spectra of PEDHA-1,8 and PEDHA-1,4, the band at 1650 cm^{-1} is attributed to stretching vibration of the quinone groups. All these IR spectral features seem to confirm the assumed chemical structure of the polyesters.

NMR Spectroscopy

The ^{1}H-NMR spectrum of PEBPA shows a chemical shift δ at 6.80–8.20 (m) ppm that may be due to the presence of aromatic protons. Six hydrogens due to methyl protons of isopropyl group produce a singlet at δ 1.51 ppm. Three hydrogens of methyl group of piperazine produce a singlet at δ 2.25 ppm and eight hydrogens of methylene groups $[(CH_2)_4]$ of piperazine ring produce a multiplet at δ 2.40–3.35 ppm.

The ^{1}H-NMR spectrum of PEDEG shows a chemical shift δ at 6.60–7.45 ppm due to the presence of aromatic protons. Three hydrogens, due to methyl group of piperazine, produce a singlet at δ 2.27 ppm and a multiplet due to eight hydrogens of methylene groups $[(CH_2)_4]$ of piperazine at δ 2.20–3.35 ppm. Eight hydrogens, due to methylene groups of diethylene glycol, produce a multiplet at δ 4.15 ppm.

Thermogravimetric Analysis

Many polymeric materials are subjected to high temperature during processing and use; therefore, it is necessary for them to have a certain degree of thermal stability. Thermal analysis is used to identify the degree of curing and to study the kinetics of degradation reactions. Such investigations assist in establishing criteria for the selection of materials for specific uses. Such studies have also proved as useful tools to look into the molecular structure of macromolecules, and aid in the determination of the strength of various bonds within the polymer. In conjugation with chromatography or spectroscopy, analytical methods such as thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), and differential thermal analysis (DTA), are valuable tools for the characterization of polymers and reinforced plastics and to determine the thermal stability of polymer samples [19–21].

Thermal stability of a polymer provides an important guideline of its usefulness. The studies of thermal behavior not only explain the behavior at high temperature but also assist in establishing criteria for the selection of materials for specific uses [22,23]. In the present investigation, we have used thermogravimetric analysis to determine the thermal stability of some representative polyesters synthesized in the present work.

Thermograms of Polyesters

The thermogram of PEBPA exhibits two distinct steps of thermal degradation. The polymer begins to decompose at about 215°C temperature. However, the small initial weight loss may be due to the presence of a small amount of solvent or absorbed moisture. The first decomposition step begins around 225°C and extends up to 520°C, which involves loss of about 45% of weight. The maximum rate of weight loss occurs at 365°C. The second decomposition step commences in the temperature range 500–800°C with a maximum rate of decomposition at 650°C, involving about 45% weight loss. At 800°C about 45% residue is left behind.

The thermal degradation of PER involves one-step decomposition. The degradation starts around 240°C and proceeds up to 500°C, involving 42% weight loss, with a maximum rate at 300°C.

The thermogram of PEDHA-1,8 exhibits one-step degradation. The degradation starts around 220°C, extends to about 450°C, and involves about 60% weight loss with a maximum rate at 280°C.

The thermogram of PEEG exhibits one-step degradation. The degradation starts around 190°C and extends to about 400°C involving about 45% weight loss, with a maximum rate at 300°C.

Qualitative Estimation of Thermal Stability

For half volatilization temperature, the thermal stability of the polymers is as follow:

$$\text{PEEG} > \text{PER} > \text{PEBPA} > \text{PEDHA-1,8}$$

The characteristic data are presented in Table 5.

Table 5: Thermal characteristics of various polyesters.

			T_{max}		
Polymer	T_0	T_{10}	**Step I**	**Step II**	T_s
PEBPA	215	300	340	650	680
PER	240	270	320	–	45
PEDHA-1,8	220	285	370	–	390
PEEG	190	250	285	–	410

Table 6: Application of Broido method to thermogram of PER.

Temp. °C	% Wt.	$Y = \frac{W_t - W_a}{W_t - W_0}$	1/y	ln ln 1/y	T °K	1/T × 10^3 °K^{-1}
290	87.25	0.8195	1.2202	−1.450	563	1.7762
300	86.00	0.8018	1.2472	−1.510	573	1.8083
310	84.50	0.7805	1.2812	−1.395	583	1.7153
320	77.50	0.6814	1.4676	−0.958	593	1.6863
330	76.00	0.6602	1.5147	−0.879	603	1.6584
340	74.25	0.6353	1.5741	−0.790	613	1.6313
350	73.50	0.6247	1.6001	−0.755	623	1.6051
360	70.00	0.5752	1.7385	−0.557	633	1.5798
370	67.50	0.5398	1.8525	−0.484	643	1.5552

Thermal stability of the polyesters on the basis of T_{max} for the first step of decomposition decreases in the following order:

$$\text{PEDHA-1,8} > \text{PEBPA} > \text{PER} > \text{PEEG}$$

Evaluation of Kinetic Parameters

Thermogravimetric analysis (TGA) has become a useful tool for evaluating kinetic parameters of various reactions of materials under application of heat. It provides a valuable technique for the evaluation of the potentially unstable nature of materials. TGA methods for calculating kinetic parameters have been proposed, based on the relationship between weight loss and temperature, often utilizing the derivative rather than integral work. The derivation of kinetic data from TGA curves obtained under nonisothermal conditions has received considerable attention and several comprehensive reviews are available.

TGA thermograms obtained at a heating rate of 10°C/min have been analyzed as per the graphical methods proposed by Broido [24] and Horowitz and Metzger [25]. The Broido and Horowitz-Metzger methods are respectively exemplified for PER in Tables 6 and 7.

The thermograms of other homopolyesters were also analyzed in the same manner. The values of apparent activation energy corresponding to different

Table 7: Application of Horowitz-Metzger method to TGA thermogram of PER.

T °C	% Wt	$1 - \alpha$	$1/1 - \alpha$	ln ln $(1/1 - \alpha)$	θ
290	87.25	0.8195	1.2202	−1.41	−30
300	86.00	0.8018	1.2472	−1.51	−20
310	84.50	0.7805	1.2812	−1.395	−10
320	77.50	0.6814	1.4676	−0.958	0
330	76.00	0.6602	1.5147	−0.879	10
340	74.25	0.6353	1.5741	−0.79	20
350	73.50	0.6247	1.6001	−0.755	30

Table 8: Activation energy (E) calculated by Broido and Horowitz and Metzger Method.

Polymer	Activation energy E (K.Cal/mol) Broido Step I	Broido Step II	Horowitz-Metzger Step I	Horowitz-Metzger Step II
PER	10.34	–	12.31	–
PEBPA	8.30	11.84	8.47	10.58
PEDHA	13.27	–	12.46	–
PEEG	16.76	–	15.24	–

decomposition steps involved were evaluated from the slope of the least square plots of the relevant data and are presented in Table 8.

Examination of the data reveals that both methods yield comparable values for activation energy. The experimental points corresponding to initial stage (10% loss) of thermal degradation 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 more reliable estimates of E, since no other temperature characteristics are involved.

CONCLUSIONS

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

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
K	Huggins constant
β	Kraemer constant

MPNCCT	2-(*N*-methylpiperazine)-4,6-bis(2-naphthoxy-6-carbonyl chloride)-*s*-triazine
PEBPA	Polyester of bisphenol-A
PEBPC	Polyester of bisphenol-C
PEBPS	Polyester of bisphenol-S
PEDHA-1,4	Polyester of 1,4-dihydroxy anthraquinone
PEDHA-1,8	Polyester of 1,8-dihydroxy anthraquinone
PEC	Polyester of catechol
PER	Polyester of resorcinol
PEPh	Polyester of phenolphthalein
PEEG	Polyester of ethylene glycol
PEDEG	Polyester of diethylene glycol

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