

# Synthesis, Characterization, and Study of various physical properties of Poly cyanurates containing BisPhenol-A

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## ABSTRACT

One of the most successful accomplishments is invention polycyanurates containing the heterocyclic ring, an ether link and an aliphatic linkage which imparts flexibility and solubility along with high thermal stability. Polymers containing s-triazine ring have become a subject of major interest due to its high resistance to heat, chemical attack, good mechanical and dielectric properties. In our present investigation, various disperse azo dyes were synthesized using different aromatic amines and couplers. These dyes were further reacted with cyanuric chloride to obtain DCTACNPDANSA which was polycondensed with Bisphenol-A(BPA). All the synthesized polycyanurates were characterized by spectral studies like IR, NMR, elemental analysis, thermal studies like TGA and physical properties viz. solubility, density, solution viscosity

**Keywords:** s-triazine, disperse dyes, polycyanurates, viscosity

## 1. INTRODUCTION

The development of structural and functional materials with an essentially improved set of properties is a topical problem and key avenue in many branches of industry [1-4]. Polymer chemistry plays a vital role in nearly all areas of our daily life and therefore it is one of the most important rapidly growing unique branches of science. The introduction of the new polymer has revolutionized the plastics, textiles, rubber, optical fibers, computer composites, automotive, marine, and aerospace industries. Polymers also have important applications in medicine including use in controlled drug delivery, artificial organs and drug delivery. 1,3,5-triazines are the oldest known organic compounds. Thermoset resins containing s-triazine ring in the main chain are useful in laminated products, moulding compounds, preparation of foams, films, sheets, paper impregnated and electrical appliances [5]. Cyanate ester (CE) resins were proposed as alternative materials for the once epoxy-dominated aerospace industry resins were proposed as alternative materials for the once epoxy-dominated aerospace industry [6]. This new-generation thermoset resin combines the processability of epoxy resins, the thermal characteristics of bismaleimides and the heat and fire resistance of phenolic resins [7]. Polycyanurates are a family of thermosetting resins that are used within a variety of electronic and microelectronic applications. The monomers offer low toxicity, ease of processing and versatile cure and blending options and undergo cure via the formation of cyanurate rings [8].

Cyanuric chloride or 2,4,6-trichloro-s-triazine is the most important and widely used compound. Polymers containing s-triazine ring have become a subject of major interest. These polymers are noted for their high resistance to heat and chemical attack, good mechanical, thermal and dielectric properties [9-14]. Yu.A. Murav'ev et al reported the synthesis of poly(phenylene-s-triazine ethers) by high-temperature polycondensation of cyanuric chloride with bisphenols. Symmetric triazine containing carboxylic bisphenol-A possess good stability, elasticity, maximum impact toughness and bending strength [15]. Bisphenols are used in the manufacture of a large fraction of the world plastic pool. For example, bisphenol (2,2-bis(4-hydroxyphenyl) propane), which is primarily converted into epoxy and polycarbonate, had a total global production of 5.2 million metric tons in 2008 [16,17]. In the present study we have discussed synthesis, characterization and physical property evaluation of derivatives of polycyanurate containing Bis Phenol-A polymers.

## 2. EXPERIMENTAL

### 2.1. Materials

All reagents, solvents, and catalyst were of analytical grade and used directly. All aromatic amines (Analytical grade) were purchased from Spectrum dyes Pvt.Ltd., Palsana and couplers from Atul Ltd., Atul. All the melting points were determined in open capillaries and are uncorrected. The purity of the compounds was checked by TLC (0.5mm) thickness using silica gel-G coated Al-plates (Merck) and spots were visualized by exposing the dry plates in iodine vapors.

## 2.2. Instrumentations

Fourier transform infrared (FT-IR) spectra were recorded on SHIMADZU- FT-IR 8400-Spectrophotometer. The <sup>1</sup>H-NMR spectra were recorded on AvanceBruker(II) 400 MHz NMR spectrometer. Elemental analysis was performed using Carlo ErbaEA1108 elemental analyzer. Samples were prepared in CDCl<sub>3</sub> and DMSO and the spectra were acquired using tetramethylsilane (TMS) as the internal standard. Elemental analysis was performed using Carlo Erba EA 1108 elemental analyzer. Thermogravimetric analysis (TGA) was performed using Pyris-6 TGA instrument heating rate of 10°C/min under flowing N<sub>2</sub> between 30° to 900°C. A suspended level kinematic viscometer developed by Ubbelohde Method was used for the measurement of viscosity. Densities of polycyanurates have been determined at 25 ± 3<sup>0</sup> C by use of powdered sample by pycnometer method. The estimation of the molecular weight of polycyanurates was carried out by Vapour Pressure Osmometry method on a Hewlett Packard-Vapour pressure osmometer by using DMF as a solvent. Dynamic TGA Thermograms obtained were analyzed using the graphical method proposed by Broido and Horowitz & Metzger for measuring the rate of thermal degradation and Activation Energy.

## 2.3. Synthesis of azo dyes

A general method for preparation of azo compound based on 1-Naphthyl amine 6, 8 substituted sulfonic acid. Primary aromatic amine (0.01 mole) was suspended in water (36 ml) Hydrochloric acid (1.08g, 0.03 mole) was added dropwise to this well-stirred suspension. The solution of sodium nitrite (0.7 g, 0.01 mole) was added to previously cooled water at 0°C. The reaction mass was stirred until the positive test for nitrous acid on starch-iodide paper. Excess of nitrous acid was neutralized by adding required amount of sulphamic acid. The resulting diazo solution was used for the subsequent coupling reaction, acid was neutralizing by adding required amount of sulphamic acid. 1-naphthylamine, 8-sulfonic acid was dissolved in acetone and the solution was cooled to 0-5°C in an ice-bath. To this well-stirred solution, the above mentioned diazo solution was gradually added in an hour a 0-5°C, maintaining the pH slightly acidic. The precipitates obtained was filtered and washed with hot water. The yield was between 75-85%. All the azo dyes were synthesized in a similar manner. Scheme 1

### 2.3. Synthesis of cyanurate azo dyes (monomer) DCTACNPDANSA

9.22 g (0.05 mole) of cyanuric chloride in 40 ml of acetone was dissolved with stirring and maintaining the temperature 0-5°C. A dye solution, 40.65 g (0.1 moles) of 8-amino-5-(2-chloro-4-nitrophenyl) diazenyl naphthalene-1-sulfonic acid in 60 ml of acetone was added to the slurry of cyanuric chloride. The mixture was stirred for 2 hours at 5°C and sodium carbonate (5.3 g) dissolved in 80 ml in water was added to maintain pH nearly 7.0. The violet colored product was filtered, washed with cold water and finally dried at 50°C in an oven. The yield was 70-80%. The product was further purified by re-crystallization using DMF. Various monomers were synthesized in a similar manner Scheme.2

### 2.4. Synthesis of Homopolymeric cyanurate azo dyes using Bisphenol A

All the polymers reported in the present investigation have been synthesized by interfacial polycondensation technique with stirring above 50°C. A typical interfacial polycondensation is exemplified below for the synthesis of polycyanurate from {8-[4',6'-dichloro-1',3',5'-triazine-2-yl) amino-5-(2-chloro-4-nitrophenyl) diazenyl]} naphthalene-1-sulfonic acid (DCTACNPDANSA) and bisphenol A. A mixture of 1.98 g (0.01 mole) of 4,4'-diamino-diphenyl methane (DDM), 0.8 g (0.02 mole) of sodium hydroxide and 0.250 g of cetyltrimethyl ammonium bromide in 50 ml of double distilled water was stirred vigorously above 50°C in a 250 ml three-necked flasks equipped with a mechanical stirrer. A solution of 2.77 g (0.005 mole) of DCTACNPDANSA in 25 ml chloroform was rapidly added to the aqueous solution and the emulsion was stirred vigorously for 5 hours above 50°C. The contents were transferred to separating funnel and the aqueous layer was removed. The chloroform layer was washed with water and finally excess of methanol was added for precipitation of the polymer. The polymer was filtered through Buchner funnel, washed thoroughly with water and finally with methanol and dried at 50°C. The dried polymer was further purified by dissolving it in chloroform and precipitating with methanol or petroleum ether. (Yield :75%) Scheme.3

**PCBPA-1:** FT-IR (KBr, cm<sup>-1</sup>): Aromatic: 829(C-H) bend, 3028(C-H) str., 1296(C-N) str., Azo: 1598(N=N) str., Sulphonic 1177(-SO<sub>3</sub>H), Ether 1239 (-C-O-C-), Aliphatic: 2967, 2928(C-H) str., <sup>1</sup>H NMR (CDCl<sub>3</sub>, ppm): 1.56 (6H, s, CH<sub>3</sub>-C-CH<sub>3</sub>), 3.41 (1H, s, -NH), 6.63-7 (17, m, Ar-H at phenyl and naphthyl)

**PCBPA-2:** FT-IR (KBr, cm<sup>-1</sup>): Aromatic: 820(C-H) bend, 3039(C-H) str., 1284(C-N) str., Azo: 1588(N=N) str., Sulphonic 1174(-SO<sub>3</sub>H), Ether 1230 (-C-O-C-), Aliphatic: 2965, 2922(C-H) str., <sup>1</sup>H NMR (CDCl<sub>3</sub>, ppm): 1.56 (6H, s, CH<sub>3</sub>-C-CH<sub>3</sub>), 3.41 (1H, s, -NH), 6.63-7 (17, m, Ar-H at phenyl and naphthyl)

**PCBPA-3:** FT-IR (KBr, cm<sup>-1</sup>): Aromatic: 825(C-H) bend, 3026(C-H) str., 1292(C-N) str., Azo: 1599(N=N) str., Sulphonic 1175(-SO<sub>3</sub>H), Ether 1237 (-C-O-C-), Aliphatic: 2969, 2925(C-H) str., <sup>1</sup>H NMR (CDCl<sub>3</sub>, ppm): 1.56 (6H, s, CH<sub>3</sub>-C-CH<sub>3</sub>), 3.41 (1H, s, -NH), 6.63-7 (17, m, Ar-H at phenyl and naphthyl)

**PCBPA-4:** FT-IR (KBr, cm<sup>-1</sup>): Aromatic: 822(C-H) bend, 3024(C-H) str., 1299(C-N) str., Azo: 1591(N=N) str., Sulphonic 1182(-SO<sub>3</sub>H), Ether 1244 (-C-O-C-), Aliphatic: 2959, 2933(C-H) str., <sup>1</sup>H NMR (CDCl<sub>3</sub>, ppm): 1.56 (6H, s, CH<sub>3</sub>-C-CH<sub>3</sub>), 3.41 (1H, s, -NH), 6.63-7 (17, m, Ar-H at phenyl and naphthyl)

**PCBPA-5:** FT-IR (KBr,  $\text{cm}^{-1}$ ): Aromatic: 819(C-H) bend, 3042(C-H) str., 1282(C-N) str., Azo: 1585(N=N) str., Sulphonic 1179(-SO<sub>3</sub>H), Ether 1244 (-C-O-C-), Aliphatic:2960,2935(C-H) str., <sup>1</sup>HNMR (CDCl<sub>3</sub>, ppm): 1.56 (6H, s, CH<sub>3</sub>-C-CH<sub>3</sub>), 3.41 (1H, s, -NH), 6.63-7 (17, m, Ar-H at phenyl and naphthyl)

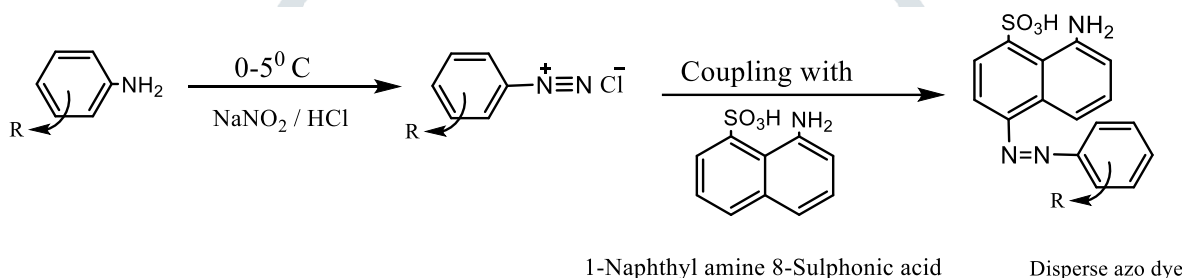
**PCBPA-6:** FT-IR (KBr,  $\text{cm}^{-1}$ ): Aromatic: 815(C-H) bend, 3048(C-H) str., 1296(C-N) str., Azo: 1594(N=N) str., Sulphonic 1171(-SO<sub>3</sub>H), Ether 1232 (-C-O-C-), Aliphatic:2965,2925(C-H) str., <sup>1</sup>HNMR (CDCl<sub>3</sub>, ppm): 1.56 (6H, s, CH<sub>3</sub>-C-CH<sub>3</sub>), 3.41 (1H, s, -NH), 6.63-7 (17, m, Ar-H at phenyl and naphthyl)

**PCBPA-7:** FT-IR (KBr,  $\text{cm}^{-1}$ ): Aromatic: 834(C-H) bend, 3045(C-H) str., 1289(C-N) str., Azo: 1596(N=N) str., Sulphonic 1175(-SO<sub>3</sub>H), Ether 1235 (-C-O-C-), Aliphatic:2968,2924(C-H) str., <sup>1</sup>HNMR (CDCl<sub>3</sub>, ppm): 1.56 (6H, s, CH<sub>3</sub>-C-CH<sub>3</sub>), 3.41 (1H, s, -NH), 6.63-7 (17, m, Ar-H at phenyl and naphthyl)

**PCBPA-8:** FT-IR (KBr,  $\text{cm}^{-1}$ ): Aromatic: 826(C-H) bend, 3042(C-H) str., 1295(C-N) str., Azo: 1586(N=N) str., Sulphonic 1171(-SO<sub>3</sub>H), Ether 1242 (-C-O-C-), Aliphatic:2962,2920(C-H) str., <sup>1</sup>HNMR (CDCl<sub>3</sub>, ppm): 1.56 (6H, s, CH<sub>3</sub>-C-CH<sub>3</sub>), 3.41 (1H, s, -NH), 6.63-7 (17, m, Ar-H at phenyl and naphthyl)

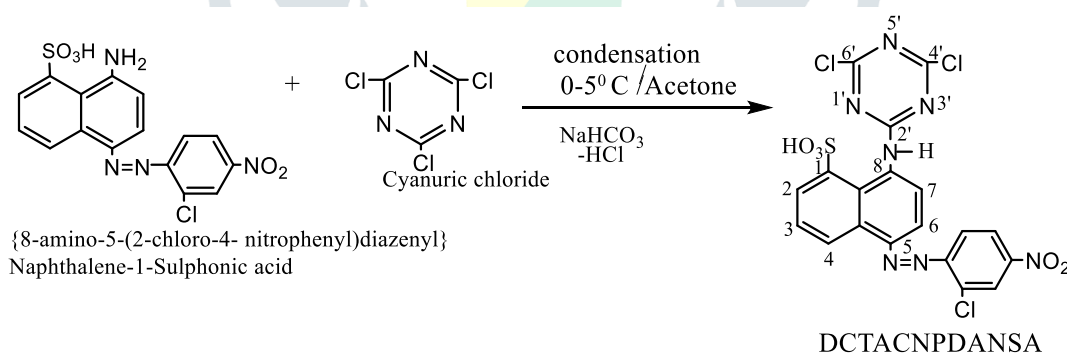
**PCBPA-9:** FT-IR (KBr,  $\text{cm}^{-1}$ ): Aromatic: 828(C-H) bend, 3035(C-H) str., 1288(C-N) str., Azo: 1595(N=N) str., Sulphonic 1179(-SO<sub>3</sub>H), Ether 1232 (-C-O-C-), Aliphatic:2959,2933(C-H) str., <sup>1</sup>HNMR (CDCl<sub>3</sub>, ppm): 1.56 (6H, s, CH<sub>3</sub>-C-CH<sub>3</sub>), 3.41 (1H, s, -NH), 6.63-7 (17, m, Ar-H at phenyl and naphthyl)

**PCBPA-10:** FT-IR (KBr,  $\text{cm}^{-1}$ ): Aromatic: 821(C-H) bend, 3025(C-H) str., 1285(C-N) str., Azo: 1599(N=N) str., Sulphonic 1170(-SO<sub>3</sub>H), Ether 1240 (-C-O-C-), Aliphatic:2965,2932(C-H) str., <sup>1</sup>HNMR (CDCl<sub>3</sub>, ppm): 1.56 (6H, s, CH<sub>3</sub>-C-CH<sub>3</sub>), 3.41 (1H, s, -NH), 6.63-7 (17, m, Ar-H at phenyl and naphthyl)

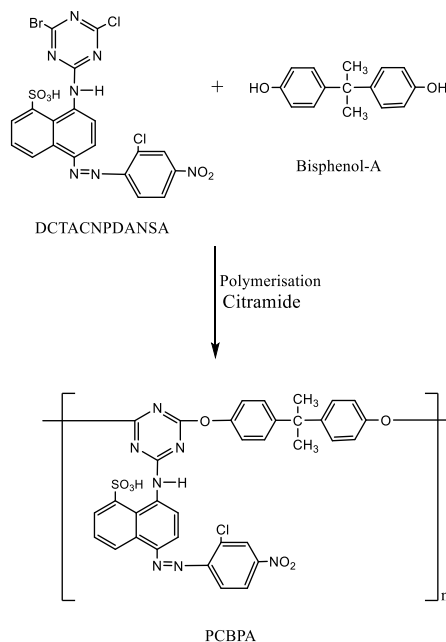


**R=** 2- Cl, 4- NO<sub>2</sub>; 2,6 dibromo 4-NO<sub>2</sub>; 2-CH<sub>3</sub>; 2-CN, 4-NO<sub>2</sub>; 2-OCH<sub>3</sub>; 2,6dichloro 4-NO<sub>2</sub>; 3-CH<sub>3</sub>; 2- Cl; 3-NO<sub>2</sub>; 4-Cl.

### Scheme 1 Synthesis of azo dye



### Scheme 2 Synthesis of cyanurate azo dyes(monomer) DCTACNPDANSA



Scheme 3 Synthesis of Homopolymericcyanurate azo dyes using Bisphenol A

### 3. RESULTS AND DISCUSSION

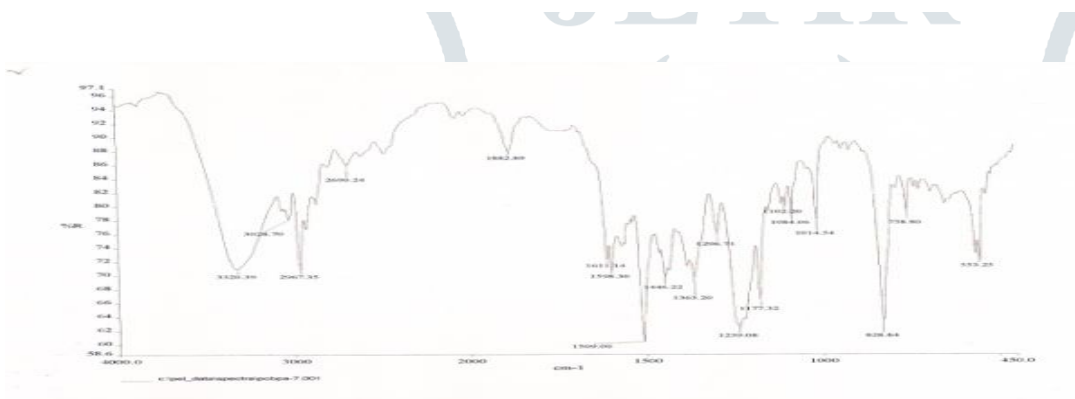


Figure 1 FT-IR spectra of PCBPA-7

Figure 1 shows the FT-IR spectra of PCBPA-7 in which the absence of a band at  $3,400\text{--}3,500\text{ cm}^{-1}$  for  $\text{--OH}$  group indicates the completion of the reaction.

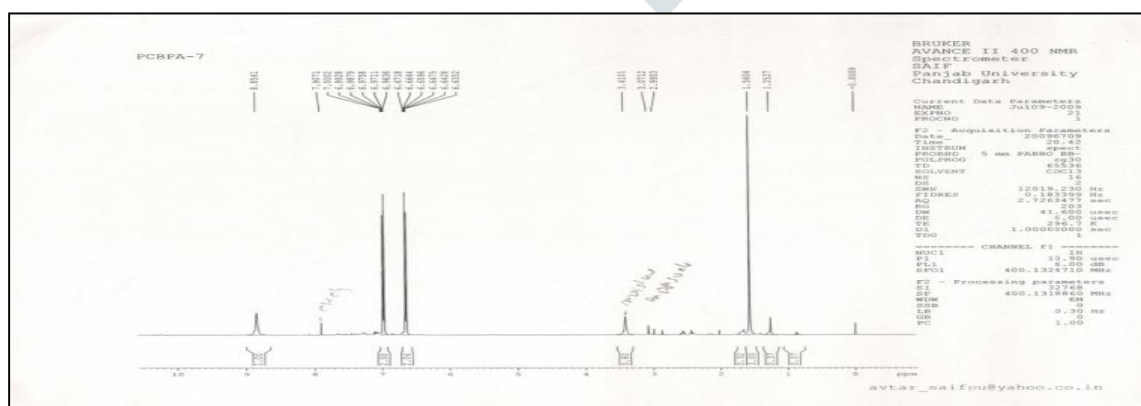
Figure 2  $^1\text{H}$ NMR spectra of PCBPA-7

Figure 2 shows the  $^1\text{H}$  NMR of homopolymericcyanurate azo dyes (PCBPA-7). The methyl protons appear as a singlet at 1.56 ppm. The N-H proton appears as a singlet at 3.41 ppm. The Aromatic and naphthyl protons appear in the range of 6.63–7.00 ppm. The absence of hydroxyl protons at 9–10.5 ppm indicates the formation of diaryl ether.

Table 1 Yield and elemental analysis data of PCBPA 1-10

Polymers	M. F.	M.W.	C %		H %		N %	
			Cal.	Obs.	Cal.	Obs.	Cal.	Obs.
PCBPA-1	$C_{34}H_{24}O_7N_7SCl$	709.5	57.50	57.45	3.38	3.32	13.81	13.86
PCBPA-2	$C_{34}H_{23}O_7N_7SBr_2$	833.0	48.97	49.10	2.76	2.82	11.76	11.70
PCBPA-3	$C_{35}H_{28}O_5N_6S$	644.0	65.21	65.27	4.34	4.28	13.04	13.11
PCBPA-4	$C_{35}H_{24}O_7N_8S$	700.0	60.00	59.91	3.42	3.38	14.00	13.92
PCBPA-5	$C_{35}H_{28}O_6N_6S$	660.0	63.63	63.69	4.24	4.29	12.72	12.79
PCBPA-6	$C_{34}H_{23}O_7N_7SCl_2$	744.0	54.83	54.78	3.09	2.98	13.17	13.11
PCBPA-7	$C_{35}H_{28}O_5N_6S$	644.0	65.21	65.29	4.34	4.26	13.04	13.14
PCBPA-8	$C_{34}H_{25}O_5N_6SCl$	664.5	61.39	61.44	3.76	3.82	12.64	12.58
PCBPA-9	$C_{34}H_{25}O_7N_7S$	675.0	60.44	60.37	3.70	3.74	14.51	14.56
PCBPA-10	$C_{34}H_{25}O_5N_6SCl$	664.5	61.39	61.32	3.76	3.81	12.64	12.70

### Thermogravimetric Analysis

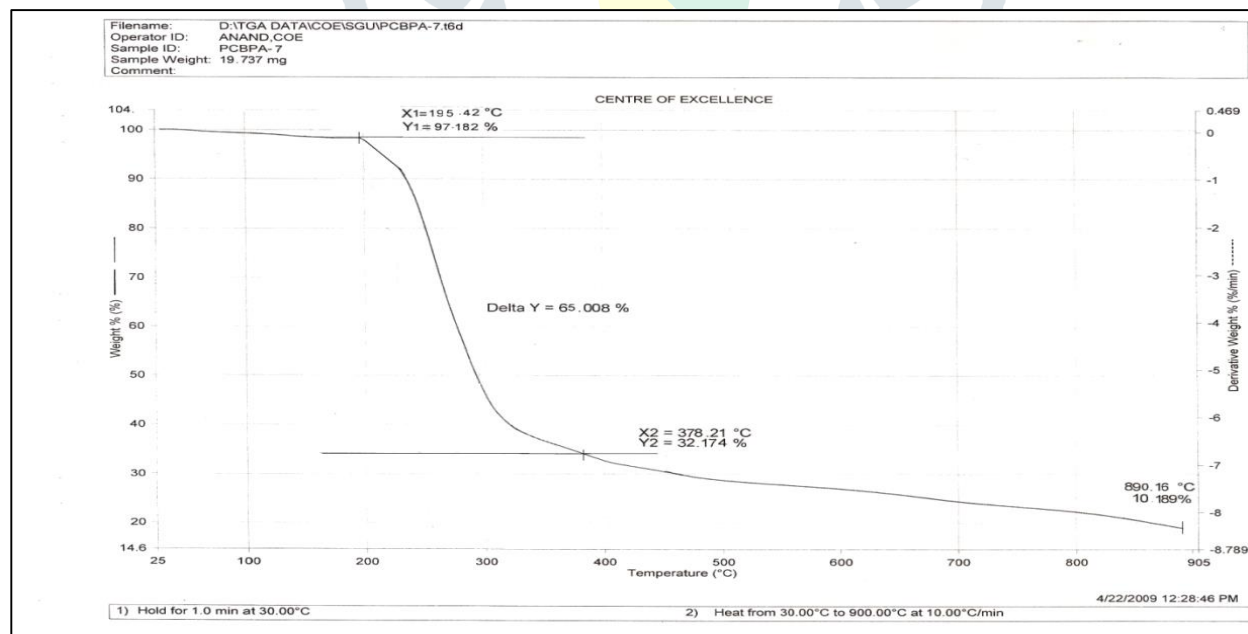


Figure 3 TGA thermogram of PCBPA- 7

The thermal degradation of homopolycyanurate azo dye PCBPA-7 was studied by TGA and the thermogram is shown in figure 3. Table 2 gives initial decomposition ( $T_i$ ), 10% weight loss ( $T_{10}$ ) and char yield. The polymer begins to decompose at

about 195°C. The decomposition is marked with a rapid weight loss in the temperature range 195-378°C during which polymer loses 65.008% of its weight. The maximum weight loss occurs at 300°C. After 500°C the sample decomposes slowly leaving about 10.189% char yield at 900°C

**Table 2.** Densities of homopolycyanurates azo dyes

POLYMER	DENSITY (g/cm <sup>3</sup> )	POLYMER	DENSITY (g/cm <sup>3</sup> )
PCBPA-1	1.287	PCBPA-6	1.283
PCBPA-2	1.289	PCBPA-7	1.277
PCBPA-3	1.280	PCBPA-8	1.279
PCBPA-4	1.282	PCBPA-9	1.285
PCBPA-5	1.286	PCBPA-10	1.278

### Density measurements

The densities were determined at 25±5°C by use of the powdered sample. Powdered sample was placed in a liquid of lower density held in stopper tube. To this liquid of lower density held in a stopper tube. To this, a liquid of higher density was added drop wise with constant stirring from a micro burette. Addition of higher density liquid was continued until a state of suspension; the density of the liquid mixture is identical with that sample. The density of the liquid mixture was then determined with the aid of Pyknometer (15 volume)

### Solubility

30 to 50 mg of finely grounded homopolycyanurate azo dye was placed into a small test tube and 1 ml solvent was added. The mixtures were stored at 30°C for few seconds with occasional shaking. Formation of streaks while shaking indicated dissolution. The sample which swelled without dissolving at 30°C was heated to 50°C in order to affect the process of dissolution

**Table 3. Relative solubility of homopolycyanurate azo dye in various solvents**

+ = Soluble, - = Insoluble, ± = partly soluble

Symbol- A indicates solubility at room temperature and symbol- B at 50°C.

Solvent	PCBPA 1		PCBPA 2		PCBPA 3		PCBPA 4		PCBPA 5		PCBPA 6		PCBPA 7		PCBPA 8		PCBPA 9		PCBPA 10	
	A	B	A	B	A	B	A	B	A	B	A	B	A	B	A	B	A	B	A	B
	Chloro benzene	-	±	-	-	-	±	-	-	-	±	-	-	-	-	-	±	-	-	-
Chloroform	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
Dichloro ethane	±	+	±	+	±	+	±	+	±	+	±	+	±	+	±	+	±	+	±	+
Dimethyl formamide	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
Dioxane	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
Xylene	-	-	-	-	-	±	-	-	-	±	-	-	-	-	-	±	-	-	-	-
Dimethyl sulfoxide	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
Methanol	+	+	+	+	±	+	±	+	-	±	+	+	±	+	-	±	±	+	+	+

Acetone	±	+	±	+	±	+	±	+	±	+	±	+	±	+	±	+	±	+	±	+
Carbon tetrachloride	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Ethyl acetate	±	+	±	+	-	±	-	±	±	+	-	±	-	±	±	+	±	+	-	±
Toluene	-	-	-	±	-	-	-	-	-	-	-	-	-	±	-	-	-	-	-	-

**Solution Viscosity Measurements**

Measurement of solution kinematic viscosity is generally made by comparing the efflux time(t) required for the specified volume of the polymer solution to flow through a capillary with the corresponding efflux time(t<sub>0</sub>) of the solvent. From t, t<sub>0</sub> and solute concentration, the relative viscosity (η<sub>rel</sub>), specific viscosity(η<sub>sp</sub>), reduced specific viscosity(η<sub>red</sub>), inherent viscosity(η<sub>in</sub>) and intrinsic viscosity [η] were evaluated.

**Table 4 The solution viscosity of homopolycyanurates azo dyes**

**PCBPA 3** Solvent: Chloroform t<sub>0</sub> = 86 Sec.

Conc. (C) g/dl	Flow time t sec.	η <sub>rel</sub> = t/t <sub>0</sub>	η <sub>sp</sub> = η <sub>rel</sub> -1	η <sub>sp</sub> /C	ln η <sub>rel</sub> /C
0.2	95.50	1.1104	0.1104	0.5520	0.5236
0.4	105.50	1.2267	0.2267	0.5657	0.5150
0.6	116.10	1.3500	0.3500	0.5833	0.5000
0.8	127.58	1.4834	0.4834	0.6043	0.4929
1.0	139.30	1.6197	0.6197	0.6197	0.4822

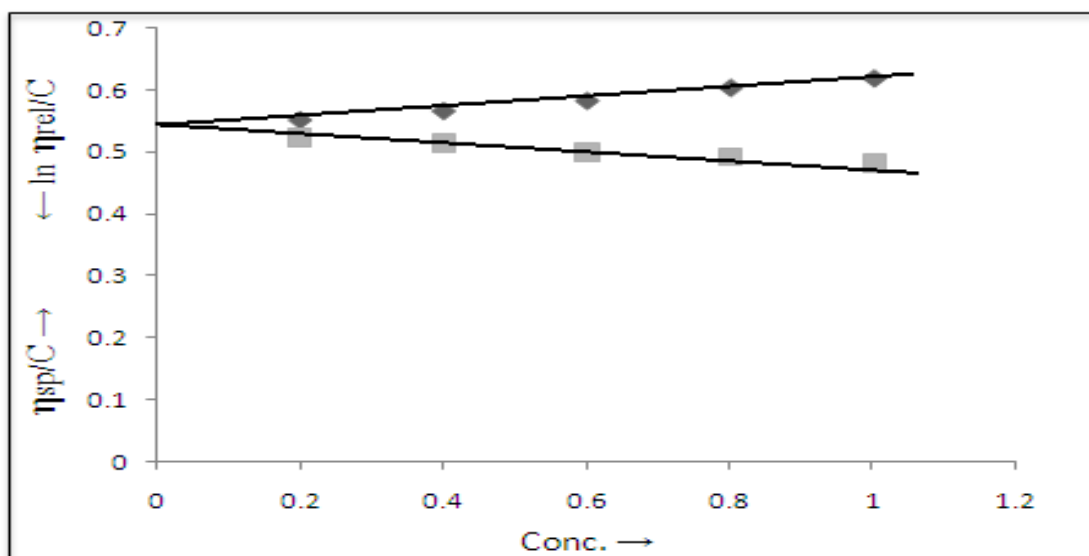


Figure 4 Typical Huggins and Krammer plots for intrinsic viscosity of PCBPA-3

Table 5 Intrinsic and reduced viscosities of homopolycyanurates and constant of Huggins and Krammer Equations:

polymer	Intrinsic Viscosity $\eta$	Reduced Viscosity of 1% solution $\eta_{sp}/C$	Inherent Viscosity $\ln \eta_{rel}/C$	K	$\beta$
PCBPA-1	0.492	0.552	0.424	0.247	0.280
PCBPA-2	0.514	0.584	0.445	0.264	0.261
PCBPA-3	0.520	0.619	0.482	0.407	0.156
PCBPA-4	0.509	0.574	0.439	0.250	0.270
PCBPA-5	0.510	0.603	0.458	0.319	0.199
PCBPA-6	0.810	0.991	0.688	0.425	0.134
PCBPA-7	0.517	0.590	0.464	0.269	0.245
PCBPA-8	0.634	0.741	0.554	0.327	0.182
PCBPA-9	0.733	0.906	0.645	0.415	0.138
PCBPA-10	0.667	0.781	0.577	0.337	0.167

The basicity and hence reactivity towards a nucleophilic substitution is higher for less acidic bisphenols and diamines. 4,4'-diamino diphenyl methane is the least acidic and hence, most reactive amongst all the bisphenols & diamines. Within a given time of reaction highly active aromatic diols and diamines are expected to yield higher molecular weight polymer than the less active one.

#### The molecular weight ( $M_n$ ) determination by Vapour Pressure Osmometry Method (VPO):

The estimation of  $M_n$  by VPO method was carried out on a Hewlett Packard Vaporpressure osmometer by using DMF as a solvent. The constant 'K' was estimated by using polystyrene of known molecular weight. Solutions four different concentrations of polystyrene were prepared in DMF. The concentrations were 5, 10, 15 and 20 g/kg respectively. The experiment was carried out following the instructions in the instrument manual. The bridge output voltage; in millivolts (mV) which is proportional to the vapor pressure lowering was measured for each concentration.

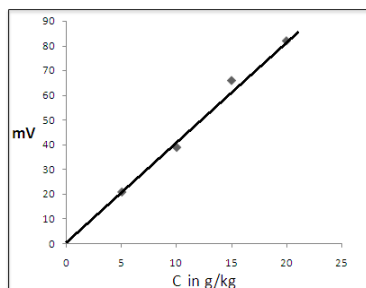
Their data are furnished in Table 5. The plot of  $mV \rightarrow C$  was plotted and was found to be linear passing through the origin. The slope was measured and hence 'K' =  $1.6279 \times 10^4$  mV/kg/mole was calculated



**Table 6 Estimation of K employing polystyrene**

Sample: Polystyrene (Mn = 3876)  
 Solvent: Dimethyl formamide(DMF)  
 Temperature: 60°C  
 Time interval 15 minutes (For steady state)

C in g/kg	5	10	15	20
mV	21	39	66	82



Slope = 4.2,  $K = M_n \times \text{Slope} = 3876 \times 4.2$

**Figure 6** Plot of mV → C of polystyrene =  $1.6279 \times 10^4$  mV kg / mol

### Estimation of Mn of polycyanurates:

Solutions of four different concentrations of 1.4, 2.8, 3.5 and 5.4 g/kg of each polycyanurates were prepared in DMF. The VPO measurements for all the polycyanurates for each concentration were carried out and for each concentration, their corresponding bridge output voltage(mV) were noted. The plots of mV → C were plotted for all the polycyanurates and found to be linear passing through the origin. From the plots, slopes were determined for each of the polycyanurates. From the values of the slope and 'K', the Mn of all polycyanurates were estimated.

**Table 7 Molecular weight determination of polycyanurates by VPO method**

Solvent:DMF Temperature:90°C Calibrant: Polystyrene  
 The time interval for the steady state: 15 minutes

Polymer	Concentration g/kg	mV	Slope	Mn = K / Slope gm/mole
PCBPA-1	1.4	11.6	6.0	2714
	2.8	19.3		
	3.5	23.1		
	5.4	33.3		
PCBPA-2	1.4	9.8	4.38	3716
	2.8	18.5		
	3.5	23.9		
	5.4	33.4		
PCBPA-3	1.4	6.8	7.1	2292
	2.8	15.3		
	3.5	18.8		
	5.4	27.3		
PCBPA-4	1.4	9.8	6.10	2668
	2.8	18.5		
	3.5	22.4		
	5.4	31.5		
PCBPA-5	1.4	7.5	6.70	2429
	2.8	17.4		
	3.5	21.6		
	5.4	30.2		
PCBPA-6	1.4	10.8	4.61	3531
	2.8	19.6		
	3.5	22.8		
	5.4	31.6		

PCBPA-7	1.4	6.5	6.32	2223
	2.8	16.4		
	3.5	20.5		
	5.4	29.8		
PCBPA-8	1.4	8.2	6.40	2543
	2.8	18.4		
	3.5	22.3		
	5.4	31.5		
PCBPA-9	1.4	9.3	6.35	2563
	2.8	18.6		
	3.5	21.6		
	5.4	32.1		
PCBPA-10	1.4	8.3	6.41	2539
	2.8	18.9		
	3.5	22.5		
	5.4	32.8		

### Conclusions

The number average molecular weight of polycyanurates is in the range of 2223-3716gm/mole. The highest intrinsic viscosity is PCBPA-9 is 0.733Hence it is proved that PCBPA-2 has the highest molecular weight and PCBPA-7 has the lowest.

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