

Synthesis And Characterization of Novel Processable Poly (Ether-Azomethine)s Containing Naphthyl Moiety

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Abstract: A new diamine 2, 2-Bis (4- phenoxy thiazole-2-amine) -1, 1-binaphthyl was synthesized starting from 2- naphthol and iron (III). New series of poly (ether-azomethine)s were synthesized from 2, 2-Bis (4- phenoxy thiazole-2-amine) -1, 1-binaphthyl with different compositions of dialdehydes such as isophthalaldehyde and terphthalaldehyde in N, N'- dimethyl acetamide (DMAc) with 5 wt% LiCl by the solution polycondensation method. Inherent viscosities of these polymers were in the range 0.30 to 0.49 dL/g, indicating formation of moderate molecular weights. These polymers exhibited good solubility in various polar aprotic solvent such as Nmethyl-2-pyrrolidone (NMP) and H₂SO₄ etc. However some polymers showed partial solubility in DMF, DMAc and THF etc. X-Ray diffraction pattern of polymers showed amorphous nature. Thermal stability was assessed by 10% weight loss temperature and the degradation temperature of the resultant polymers falls in the ranges from 378°C to 421°C in nitrogen. The glass transition temperature was in the range of 224-248°C. The structure-property correlation among these polyazomethines were studied; in view of their potential applications as high performance polymers.

Keywords: 2,2-Bis (4-phenoxy Thiazole 2- amine) -1,1-binaphthyl, processability, Viscosity, Thermal stability.

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I. Introduction

The azomethine bond - CH = N - is formed during polycondensation of aromatic and aliphatic diamines with aromatic and aliphatic dialdehydes. The literature of polyazomethines is extensive [1-4]. Many researchers have tried to synthesize high molecular weight polymers but failed due to poor solubility in common organic solvents. Polyazomethines are of interest because of their high thermal stability, good mechanical properties, which makes them favorable members for aerospace applications (5). In addition, polyazomethines exhibit desirable useful properties such as electronic, optoelectronic (6-7) and liquid crystalline (9-10), fiber forming, ability to form metal chelates (11-13) and for semiconducting properties (14).

The first polyazomethines were reported by Adams and coworkers (15) in 1923. Suematsu and Morgan and coworkers have also reported the synthesis of some soluble, film- and fiber-forming polyazomethine (16). It is established that the presence of ether linkages in the polymer backbone imparts segmental mobility to the polymer which enhances the solubility and lowers the glass transition temperature.

To enhance the processability of aromatic polyazomethines, the present study describes a successful synthesis of new soluble and processable aromatic polyazomethines without much sacrifice of thermal stability by incorporation of aromatic naphthalene moiety and heterocyclic thiazole group into the polymer backbone. For this, a new aromatic diamine monomer having aromatic naphthalene as well as heterocyclic thiazole group was designed and synthesized, which was subjected to polycondensation with various aromatic dialdehydes.

II. Experimental

Measurements

FTIR spectra were recorded on a Perkin-Elmer Spectrum GX spectrophotometer. NMR spectra were recorded on a Bruker 400 MHz spectrometer for ¹H spectrum and 100 MHz for ¹³C spectrum measurements using (d₆-DMSO). Inherent viscosity of polymers was measured with 0.5% (w/v) solution of polymer in N, N-dimethylformamide at 30±0.1°C using an Ubbelohde Suspended Level Viscometer. Solubility of polyamides was determined at concentration of 3% (w/v) in different solvents at room temperature or on heating. Dried polymer powder was used for X- ray measurements. Differential scanning calorimetry (DSC) measurements were made on a Mettler Toledo Instrument at a heating-cooling rate of 10°C /min under nitrogen. The Tg was taken at the middle of the step transition in the second heating run. Thermogravimetry (TGA) was measured on

a Mettler Toledo Instrument. A heating rate of 10°C/min was used for the determination of the decomposition temperature (Td) at a 10% weight loss under nitrogen environment. X-ray diffraction patterns of polymers were obtained on a RigakuDmax 2500 X-ray diffractometer.

Materials

All the solvents/chemicals were purified before use by using standard procedures.

The reagents such as 2-Naphthol, 4-fluoro acetophenone, Thiourea, Iodine, Terephthalaldehyde (TPA), Isophthalaldehyde (IPA) were purchased from Sigma Aldrich (USA) and used as received. DMAc was purified by vacuum distillation from barium oxide. Potassium carbonate (K₂CO₃) was dried under vacuum at 150°C for 6h. Lithium chloride was dried under vacuum at 150°C for 6h.

Monomer Synthesis

Synthesis of 2, 2'-Dihydroxy-1,1-binaphthyl (I):

In a three necked flask equipped with a dropping funnel and reflux condenser, 14.4g (0.1 mol) of 2-naphthol and 600 mL of water were placed and heated to boil. To the boiling liquid containing 2-naphthol in suspension; a solution of 28 g (0.1 mol) crystallized iron (III) chloride in 60 mL water was added. While addition of iron (III) chloride solution, oily drops of 2-naphthol was observed. Further reaction mixture was boiled till oily drops were disappeared and the 2,2'-Dihydroxy-1,1-binaphthyl (I) separates out in flasks boiled for 10 minutes. The hot suspension was filtered through the previously warmed Buckner funnel; the crude product was washed with boiled water and dried well. The 2,2'-Dihydroxy-1,1-binaphthyl was recrystallized from toluene (about 150 mL) to get colorless crystals.

Yield: 13.12 g (92.25 %)

M. P.: 217°C.

Synthesis of 2, 2'-Bis (4-acetophenone)-1, 1-binaphthyl (II):

In a 500 mL three neck round bottom flask equipped with calcium chloride guard tube, thermo well, N₂ gas inlet were placed 14.3g 2,2'-Dihydroxy-1,1-binaphthyl (0.05 mol) and 13.814 g 4-Fluoroacetophenone (0.1 mol) in 125 mL N,N-dimethyl acetamide (DMAc), then 13.821 g of anhydrous K₂CO₃ was added. The resulting reaction mixture was refluxed for 5 h. The progress of reaction was studied by TLC method. After completion, reaction mixture was cooled to room temperature and water was added in it for precipitating the product from solution. Finally the product was isolated by filtration, washed with water and finally dried under vacuum.

Yield: 21.61 g (82.75 %)

M.P.: 130°C

IR: 3033, 2974, 1696, 1594, 1403, 1222, 1071, 1062, 815, 774 cm⁻¹.

Synthesis of 2, 2'-Bis (4-phenoxy Thiazole 2- amine) -1,1-binaphthyl (III):

In a 100 mL three neck round bottom flask added compound (I) 0.4 g (0.001 mol) and iodine 0.252 g (0.003mol), Thiourea 1.2 g (0.006mol) and the mixture was stirred in THF at reflux for 48 h. Then poured the solution with stirring in water, the yellow solid was obtained. Product was recrystallized in ethanol.

Yield: 11.80 g (81.49 %)

M.P.: 165°C.

IR: 3300, 3275, 3091, 3010, 1607, 1509, 1376, 1219, 1157, 1014, 827, 781 cm⁻¹.

¹H NMR (d₆-DMSO): δ = 8.14 (d, 4H), 7.92 (d, 2H), 7.49 (t, 2H), 7.32 (t, 4H), 7.29 (d, 4H), 6.81 (d, 4H), 6.09 (d, 2H), 3.93 (s, 4H).

¹³C NMR (d₆-CDCl₃): δ = 168.95, 151.48, 147.68, 129.32, 125.51, 124.81, 124.21, 123.26, 122.18, 121.77, 120.83, 119.84, 117.17, 114.46, 113.93, 113.80, 103.80.

Mass Spectra m/e (m+1) = 635.

Polymer Synthesis

Synthesis of poly (ether-azomethine)s from 2, 2'-Bis (4-phenoxy Thiazole 2- amine) -1,1-binaphthyl (III):

In a 100 mL three neck round bottom flask equipped with reflux condenser, magnetic stirrer, calcium chloride guard tube and nitrogen gas inlet were placed 0.554 g 2, 2'-Bis(4-methyl carboxyphenoxy) -1, 1-binaphthyl (IV) (1mmol), in 3 mL N, N-dimethyl acetamide (DMAc) containing 5% lithium chloride (0.150 g). After the mixture became clear, 0.134 g (0.001 mol) terephthalaldehyde (TPA) was added in flask and the resulting mixture was stirred overnight. Finally, the polymerization mixture was heated at 140°C for 4 h. The resulting viscous mass was added to a large excess of water. The fibrous polymer was isolated by filtration. The polymer (PAM-I) was washed several times with hot water to remove any inorganic impurities and was dried

under vacuum at 60°C overnight. The yield was 99% and the inherent viscosity of polymer in NMP was 0.49 dL/g.

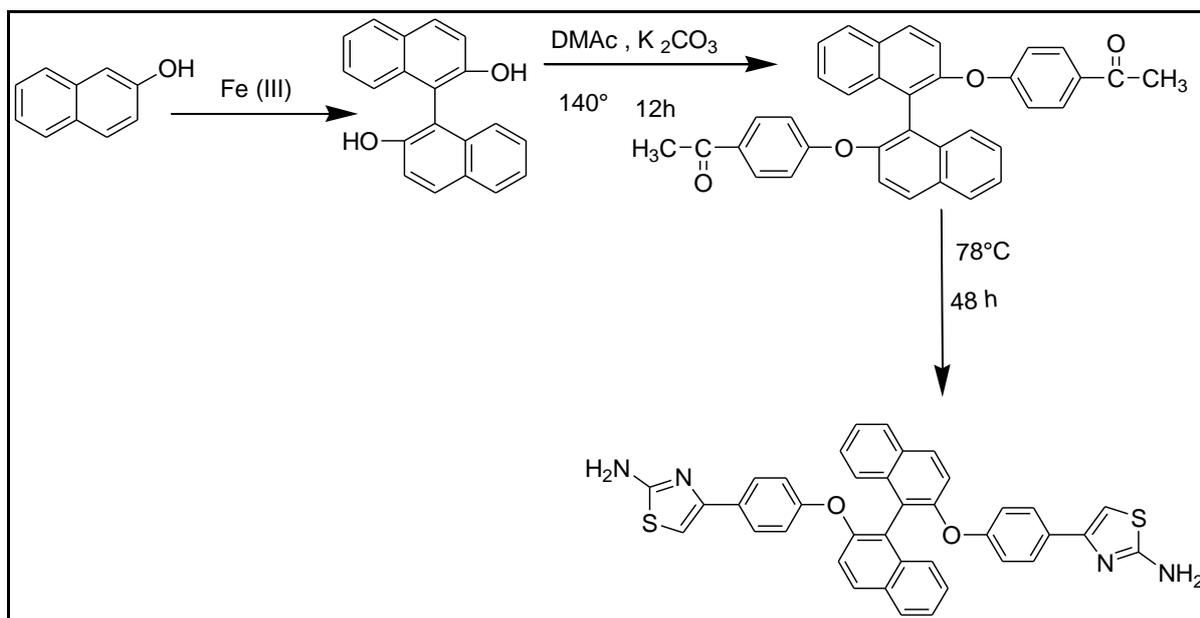
The polyazomethines and co-polyazomethines PAM-II to PAM-V were synthesized with varying mol proportion of TPA and IPA by similar procedure.

III. Results and Discussion

In this work, a new diamine monomer bearing naphthyl Moiety, viz. 2,2-Bis (4-phenoxy Thiazole 2- amine) -1,1-binaphthyl (III) have been synthesized and used as building blocks for synthesis of new series of poly (ether-azomethine)s . These poly (ether-azomethine)s were characterized by IR, inherent viscosity, solubility, thermal study and XRD.

3.1. Synthesis of 2, 2-Bis (4-phenoxy Thiazole 2- amine) -1, 1-binaphthyl (III)

New Thiazole amine 2, 2-Bis (4-phenoxy Thiazole 2- amine) -1, 1-binaphthyl (III) were successfully synthesized in several steps starting from naphthol (Scheme-1).



Scheme 1. Synthesis of 2, 2-Bis (4-phenoxy Thiazole 2- amine) -1, 1-binaphthyl (III)

The 2, 2'-dihydroxy-1, 1-binaphthyl (I) was synthesized by reacting 2-naphthol in presence of crystallized iron (III) chloride as catalyst. The 2, 2'-Bis(4-acetophenoxy)-1, 1-binaphthyl (II) was obtained by reacting 2, 2'-dihydroxy-1, 1-binaphthyl with 4-Fluoroacetophenone and Potassium carbonate as catalyst in DMAC. The structure of (II) was characterized by infrared spectroscopy.

The infrared spectrum of (II) (Fig 1.) showed strong absorption bands at 3033 and 2974 cm^{-1} corresponding to aromatic and aliphatic C-H stretching vibrations. Spectrum also showed absorption near 1403 cm^{-1} due to C-H bending vibration. Absorption at 1696 cm^{-1} of carbonyl (C=O) stretching absorption was corresponding to acetyl carbonyl moiety. The absorption bands in the region 1222 cm^{-1} and 1071 cm^{-1} showed aromatic and aliphatic C-O-C stretch.

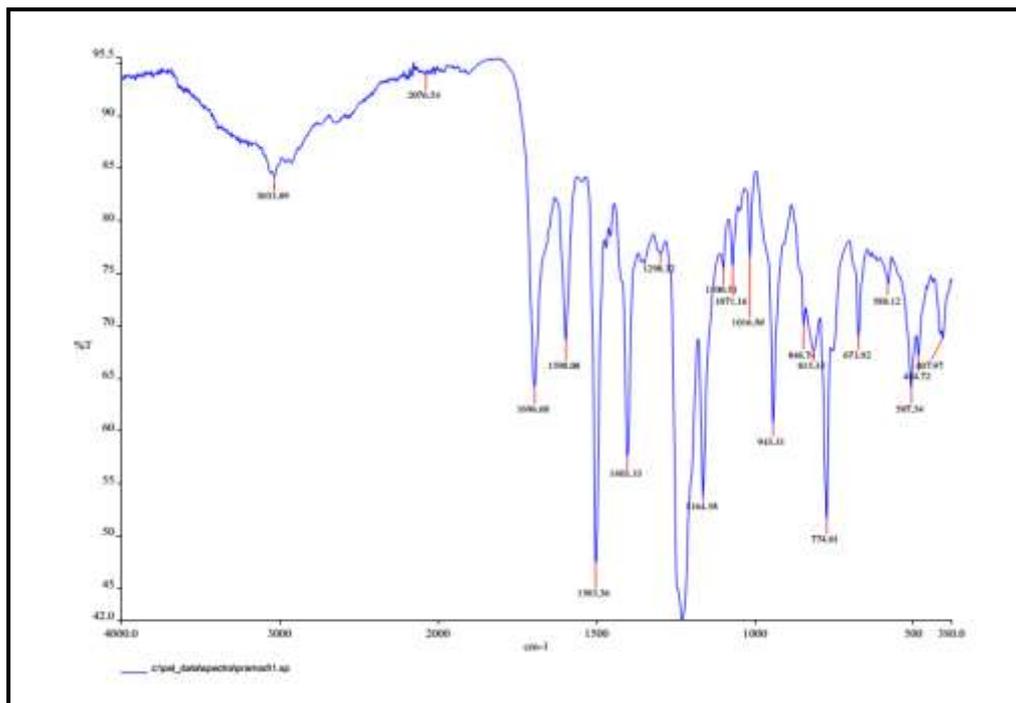


Fig1. FT-IR spectrum of 2, 2'-Bis (4-acetophenone) - 1, 1binaphthyl (II)

The new thiazole amine monomer 2, 2-Bis (4-phenoxy Thiazole 2- amine) -1, 1-binaphthyl (III) was confirmed by IR, NMR (¹H and ¹³C) and mass spectroscopy. FT-IR spectrum of 2,2-Bis (4-phenoxy Thiazole 2- amine) -1,1-binaphthyl (III) (Fig 2) exhibited N-H stretching absorption bands at 3275 cm⁻¹(asymmetric N-H stretching) and 3091 cm⁻¹(symmetric N-H stretching) and C-O-C stretching at 1219 cm⁻¹ and 1157 cm⁻¹. Band at 3010 cm⁻¹ is due to aromatic C-H stretching OF naphthyl moiety and very broad absorption bands near 3300 cm⁻¹ was corresponding to N-H stretch of -NH₂ group.

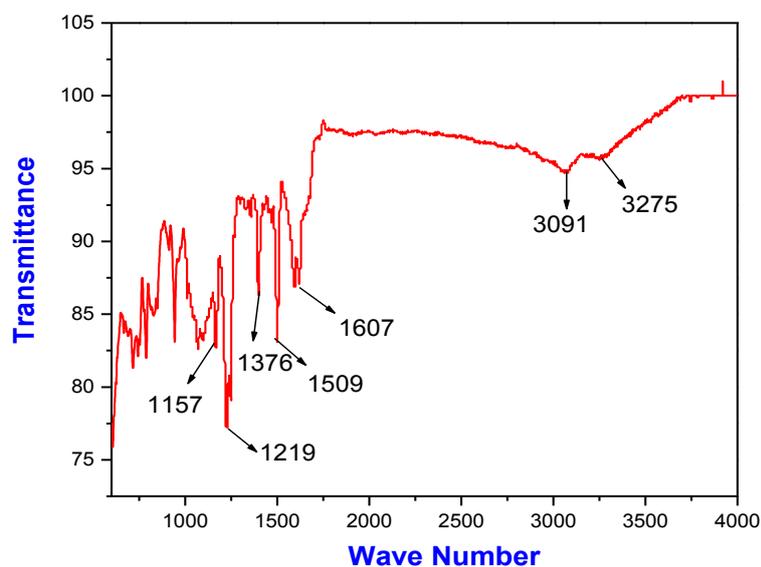


Fig 2. FT-IR spectrum of 2,2-Bis (4-phenoxy Thiazole 2- amine) -1,1-binaphthyl (III)

The proton NMR spectrum (Fig 4.) of (III) showed the NMR singlet at 3.93 δ corresponding to amine (-NH₂) proton. The signals in the range of 7.29 and 6.81 δ of (8H) are attributed to the aromatic protons of phenylene rings whereas signal at 8.14, 7.92, 7.49 and 7.32 δ(12H) are assigned to naphthalene proton. The NMR signal appears at 6.09 δ singlet attributed to methylene of -CH attached to ring.

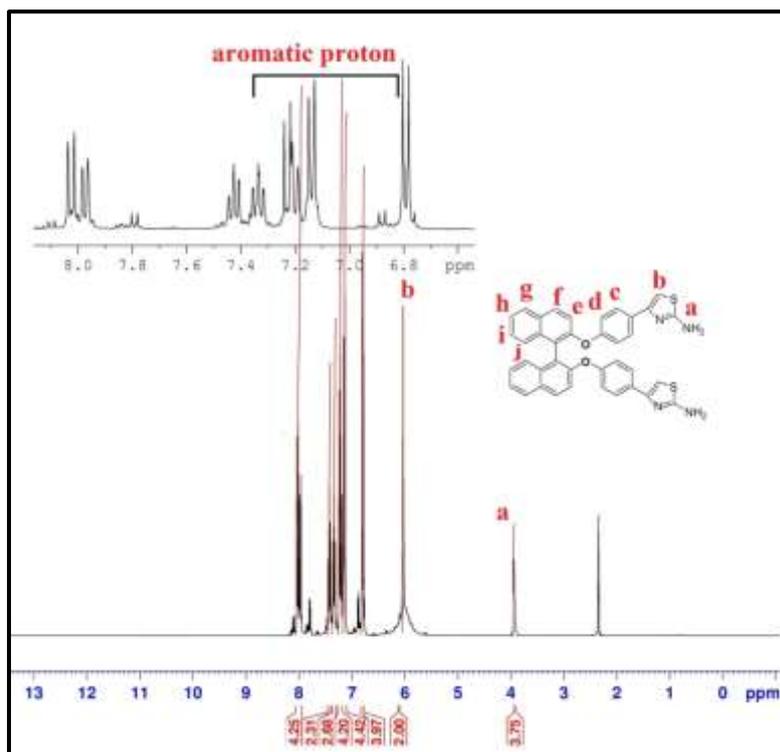


Fig 3. $^1\text{H-NMR}$ of 2,2-Bis (4-phenoxy Thiazole 2- amine) -1,1-binaphthyl (III)

The ^{13}C NMR spectrum (Fig 5.) of 2,2-Bis (4-phenoxy Thiazole 2- amine) -1,1-binaphthyl (III) Seventeen NMR signals corresponding to 17 different types of carbons. The amine attached carbon appeared at 168.95δ for (C-NH₂); whereas quaternary carbons showed signals at $151.48, 147.68, 129.32, 125.51, 123.26, 119.84 \delta$. The CH carbons appeared at $124.82, 124.21, 123.27, 121.78, 120.83, 119.85, 114.46,$ and 113.93δ whereas CH carbon bind with Sulphur showed NMR signals at 103.80δ .

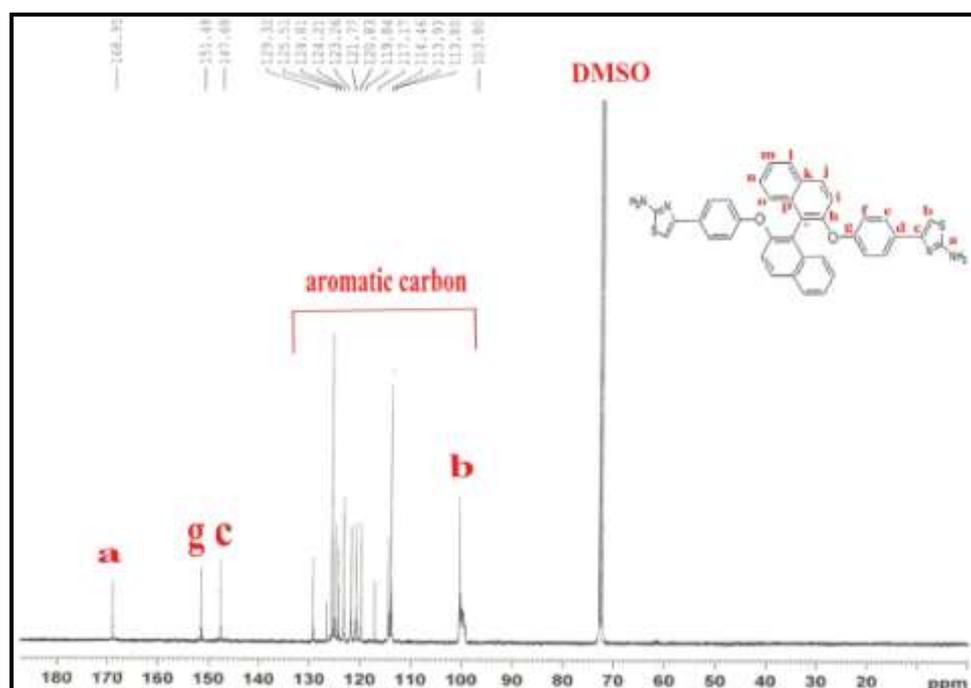


Fig 4. $^{13}\text{C-NMR}$ of 2,2-Bis (4-phenoxy Thiazole 2- amine) -1,1-binaphthyl (III)

The mass spectrum of 2,2-Bis (4-phenoxy Thiazole 2- amine) -1,1-binaphthyl (III) (Fig 5) showed molecular ion peak at m/e ($m+1$) at 635 corresponding to molecular weight of 2,2-Bis (4-phenoxy Thiazole 2- amine) -1,1-binaphthyl (III).

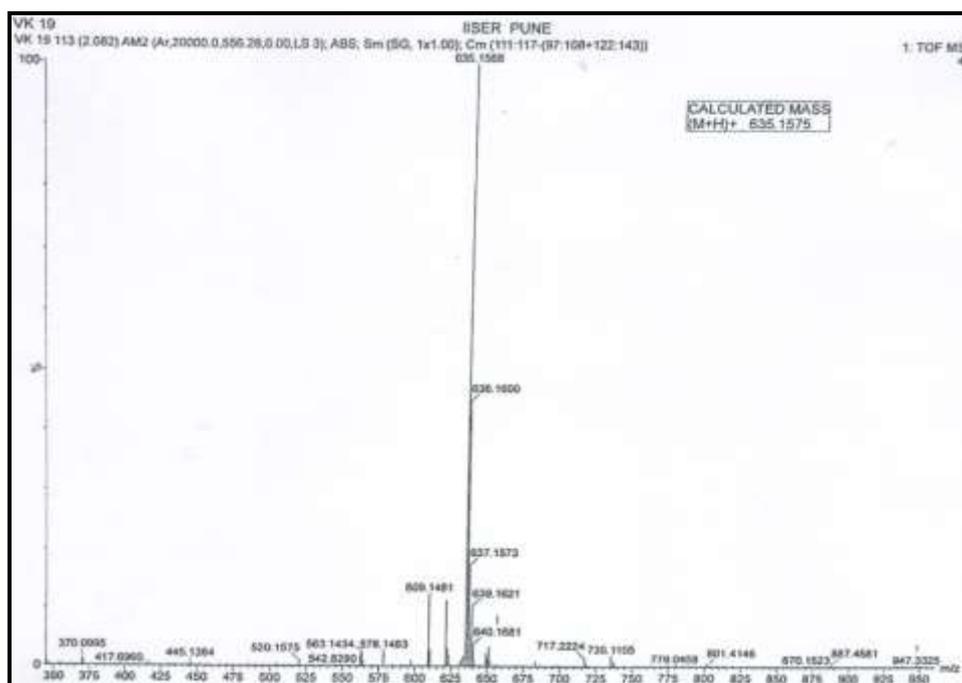
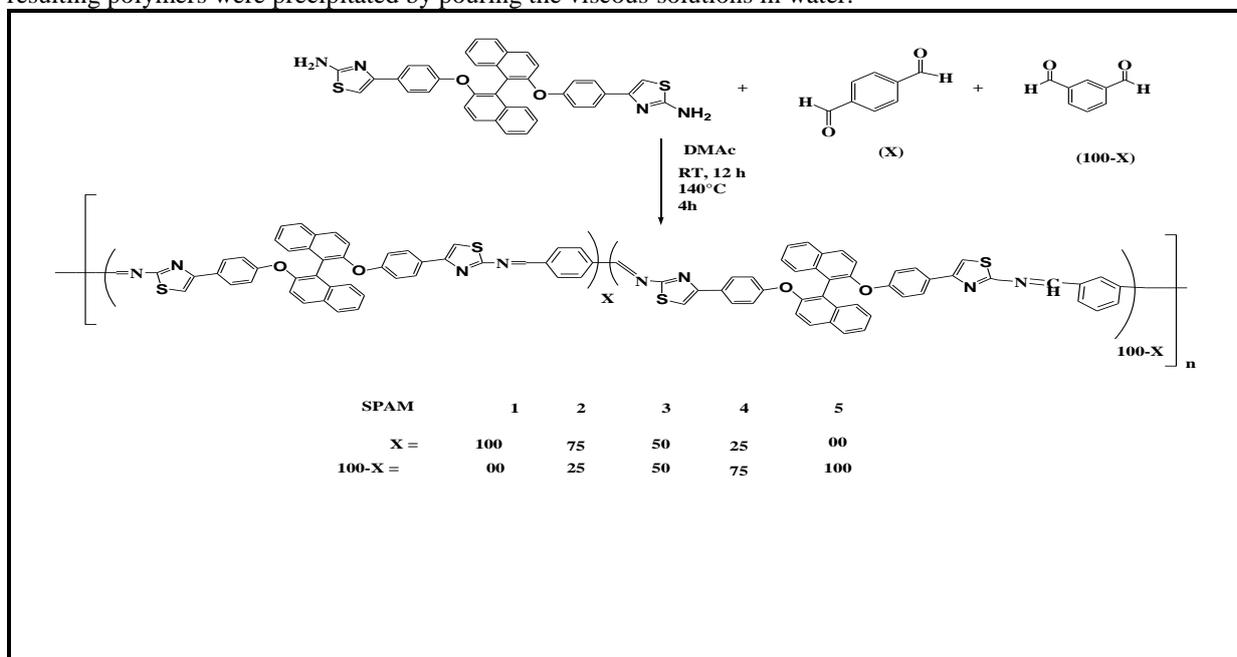


Fig 5. Mass spectrum of 2,2-Bis (4-phenoxy Thiazole 2- amine) -1,1-binaphthyl (III)

From all above spectral characterization data, it confirmed that, 2,2-Bis (4-phenoxy Thiazole 2- amine) -1, 1-binaphthyl (III) diamine monomer was formed.

A series of methyl substituted homo and co-poly(ether-azomethine)s were synthesized as outlined in Scheme 2 by elevated temperature solution polymerization of 2,2-Bis (4-phenoxy Thiazole 2- amine) -1,1-binaphthyl (III) with dialdehydes TPA and/or IPA in DMAc containing LiCl. Lithium chloride was used to absorb water formed during the polycondensation. The polymerization proceeded smoothly giving highly viscous solution. The resulting polymers were precipitated by pouring the viscous solutions in water.



Scheme 2. Synthesis of poly (ether-azomethine)s from 2,2-Bis (4-phenoxy Thiazole 2- amine) -1,1-binaphthyl (III)

The yield and viscosity obtained are presented in Table 1. The inherent viscosities of all these polymers were determined in NMP and ranged from 0.30 to 0.49 dL/g. The data of these poly (ether-azomethine)s are presented in Table 1

Table.1 Yield and Viscosity of Poly(ether-azomethine)s

Polymer Code	Monomers			Yield %	Inherent Viscosity dL/g a
	Diamine Mol %	TPA Mol%	IPA Mol%		
PAM I	100	100	0	99	0.49
PAM II	100	75	25	98	0.38
PAM III	100	50	50	97	0.43
PAM IV	100	25	75	98	0.30
PAM V	100	0	100	99	0.34

^ainherent viscosity was measured at a concentration of 50mg/10 ml in NMP at 30°C.

The polymers were characterized by the infrared spectroscopy. The IR spectrum of poly (ether-azomethine) PAM-I, Fig 6 showed the characteristic absorption at 1624 cm⁻¹ (CH = N stretching). The sharp bands occurring at 1229 and 1121 cm⁻¹ in the spectra of the polymers are due to the asymmetrical and symmetrical vibrations of the (C-O-C) ether linkage. The presence of bands at 2970 and 2865 cm⁻¹ can be assigned to asymmetric and symmetric aliphatic (C-H stretching) vibrations. The vibration at 827 cm⁻¹ indicates para catenation of aromatic rings.

The IR spectrum of poly (ether-azomethine) PAM-II, Fig 6 showed the characteristic absorption at 1611 cm⁻¹ (CH = N stretching). The sharp bands occurring at 1233 and 1151 cm⁻¹ in the spectra of the polymers are due to the asymmetrical and symmetrical vibrations of the (C-O-C) ether linkage.

The IR spectrum of poly (ether-azomethine) PAM-III, Fig 6 showed the characteristic absorption at 1613 cm⁻¹ (CH = N stretching). The sharp bands occurring at 1260 and 1151 cm⁻¹ in the spectra of the polymers are due to the asymmetrical and symmetrical vibrations of the (C-O-C) ether linkage.

The IR spectrum of poly (ether-azomethine) PAM-IV, Fig 6 showed the characteristic absorption at 1621 cm⁻¹ (CH = N stretching). The sharp bands occurring at 1246 and 1151 cm⁻¹ in the spectra of the polymers are due to the asymmetrical and symmetrical vibrations of the (C-O-C) ether linkage.

The IR spectrum of poly (ether-azomethine) PAM-V, Fig 6 showed the characteristic absorption at 1618 cm⁻¹ (CH = N stretching). The sharp bands occurring at 1212 and 1164 cm⁻¹ in the spectra of the polymers are due to the asymmetrical and symmetrical vibrations of the (C-O-C) ether linkage.

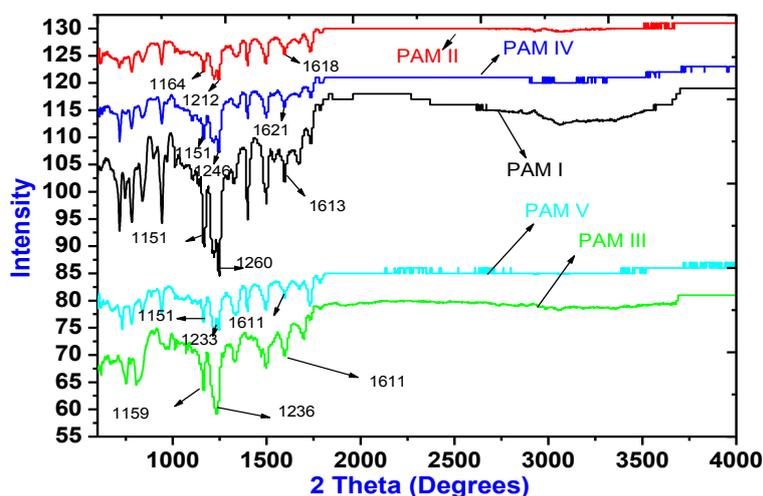


Fig 6. FT-IR spectrum OF PEA-I to V

Solubility properties of poly (ether-azomethine).

Solubility characteristics of poly(ether-azomethine)s are summarized in Table 2. It is observed that the entire poly (ether-azomethine) PAM-1 to PAM-5 exhibited solubility in organic solvent N-methylpyrrolidone (NMP) and also shows partial solubility in solvents such as THF and DCM. All these Polyazomethines (PAM-1 to PAM-5) are insoluble in solvents such as DMF, DMAc and DMSO. Polymer PAM-1 synthesized from terphthaldehyde (TPA) exhibit less solubility due to its stiff structure attributed more close packing of polymer

chains. But polyazomethinePAM-4 shows better solubility in solvents DMF, DMAc, NMP and DMSO, thus good improvement in solubility of these polymer, as expected; can be attributed to the copolymerization of novel diamine with TPA and IPA ,introduction of Cardonaphthyl moiety and ether linkages in the polymer backbone.

Table 2.Solubility behavior of Poly(ether-azomethine)s

Polymer Code	Solvents							
	DMF	DMAc	DMSO	NMP	THF	CHCl3	DCM	C.H2SO4
PAM I	--	--	--	++	+-	--	++	++
PAM II	+-	+-	--	++	+-	--	++	++
PAM III	+-	+-	--	++	+-	--	++	++
PAM IV	++	++	++	++	+-	--	+-	++
PAM V	+-	+-	+-	++	+-	--	+-	++

++: Soluble; -- : Insoluble on heating; +- : Sparingly soluble

^a Solubility measured at polymer concentration of 3% (w/v)

Thermal Properties

Thermal behaviour of polymers was evaluated by means of thermogravimetry and differential scanning calorimetry. Table 3 incorporate the thermal data such as glass transition temperature (T_g), initial decomposition temperature (T_i), 10 % decomposition temperature (T_d) and residual weight at 900°C.

Table 3 Physical properties of Poly(ether-azomethine)s

Polymer Code	Thermal behaviour b			
	T _i °C	T _d °C	T _g °C	Residual Wt % at 800°C
PAM-1	311	421	248	59
PAM-2	301	398	243	58
PAM-3	285	392	225	57
PAM-4	276	382	224	58
PAM-5	271	378	227	60

Temperature at which onset of decomposition was recorded by TG at a heating rate of 10°C/min.

T_g- Glass transition temperature determined at second heating by DSC at a heating rate of 10°C/min

T_d – Temperature of 10% decomposition

T_i – Initial decomposition temperature.

The thermal stability of poly(ether-azomethine)s outlined in Fig 4 was studied at a heating rate of 10°C/min in nitrogen atmosphere by thermogravimetric analysis. T_d values were in the range of 378°C to 421°C. In general, these polymers are good thermal stability in nitrogen; 10% weight loss only takes place when they are heated beyond 378 °C in nitrogen. The initial decomposition temperature (T_i) was in the range of 271°C to 311°C. The residual weight at 900°C was in the range of 57%-60%.

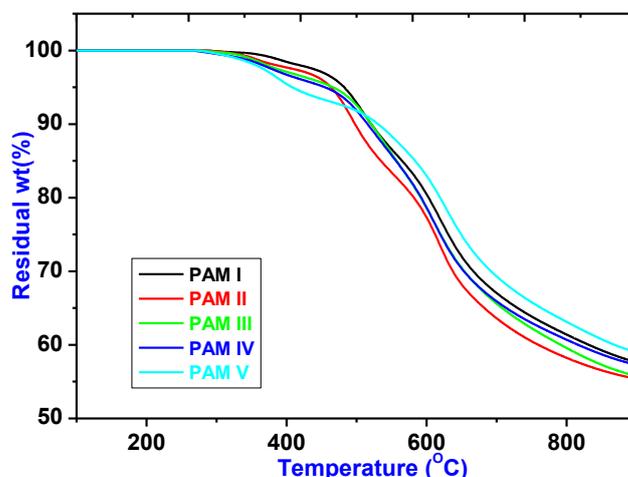


Fig 7.TGA curve ofPoly(ether-azomethine)s

The DSC curves (Fig.5) represent the T_g values of poly (ether-azomethine)s.

All these polyazomethines exhibits a T_g indicative of an amorphous. All the polymers show T_g in between 224-248°C. The higher T_g of PAM-I compared to the T_g of other polymers is due to the usage of terphthaldehyde (TPA) which exhibits rigid structure attributed more close packing of polymer chains.

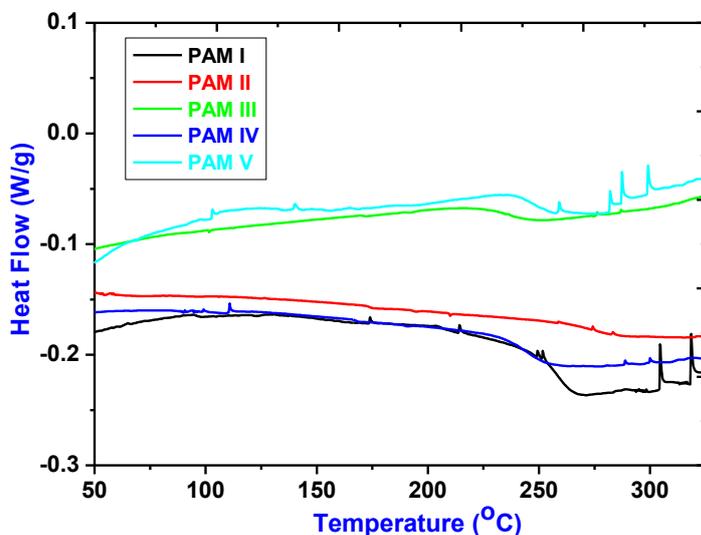


Fig 8.DSC of curve of Poly(ether-azomethine)s

X-Ray diffraction

X-Ray diffractograms of polymers exhibited a broad halo in the wide angle region (at about $2\theta \approx 20^\circ$) indicating that, the polymers were amorphous in nature. Poly (ether-azomethine)s were also characterized by the wide angle X-ray diffractometer. The X-ray diffraction pattern of all poly (ether-azomethine)s is shown in Fig 6. It is observed that, the all polymer is highly amorphous in nature. This may be attributed to the copolymerization of novel diamine with IPA and TPA, introduction naphthyl moiety of novel diamine monomer and ether linkages which may have disrupted the chain regularity and packing leading amorphous nature.

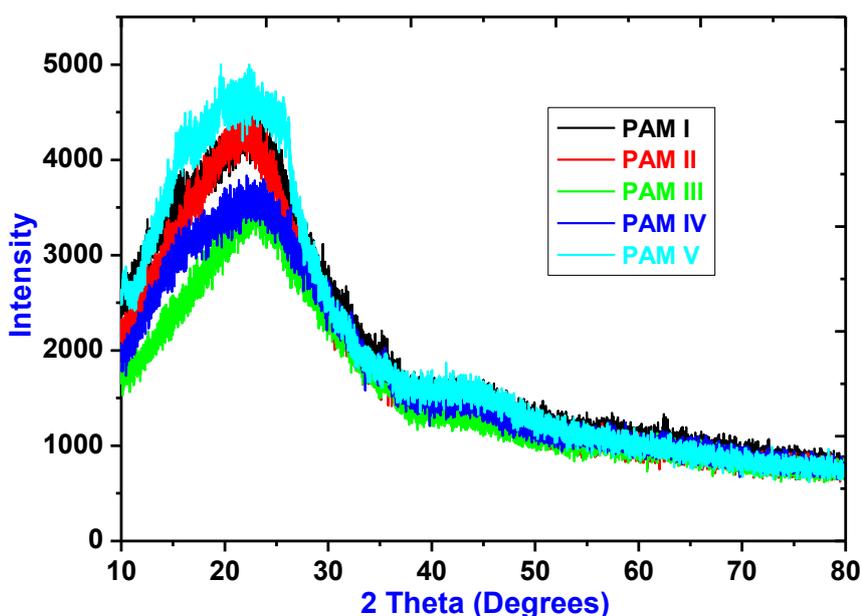


Fig 9.XRD curve of Poly(ether-azomethine) s

IV. Conclusion

We successfully synthesized a Series of new aromatic poly(ether-azomethine)s containing naphthyl units in the polymer backbone were synthesized with the aim of improving the solubility and processability of polymers. The inherent viscosities of these polymers range from 0.30 to 0.49 dL/g. The most of these polymers are amorphous and soluble in a number of organic solvents, such as DMF, NMP, DMAc, THF and CHCl₃. The Tg's in nitrogen and Td's for 10% weight loss in nitrogen range from 224-248°C and 378°C to 421°C, respectively, depending on the exact polymer structure.

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