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Synthesis, Characterization and Properties of New Processable Poly (ether-amide) s from 2, 2-Bis (p-methyl carboxyphenoxy) -1, 1-binaphthyl and Aromatic Diamines

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Abstract: New diacid monomer 2,2-Bis (p-methyl carboxyphenoxy) -1,1-binaphthyl (IV) was prepared through reaction of 2-naphthol with iron (III) chloride to 1,1-bis-2-naphthol (I) followed by 4-Fluoroacetophenone to diacetyl 1,1-bis -2-napthol (II) followed byconardwillgerodt reaction with Kindler variation using sulphur and morpholine gave 1,1-bis-2-napthol thioacetomorpholide (III) and further, new diacid (IV) monomer was obtained by base catalyzed hydrolysis of intermediate thiomorpholide in ethanol. The structure of diacid was confirmed by ^{1}H NMR, ^{13}C NMR, FT-IR and Mass Spectra. A new series of poly(ether-amide)s were synthesized by using direct Yamazaki's phosphorylativepolycondensation method from new diacid (IV) monomer with different commercial diamines such as 4,4'-diamino diphenyl ether (ODA), diaminodiphenyl methane (MDA), 4 amino phenyl sulfone , P-phenylenediamine M-phenylenediamine. Inherent viscosities of these poly(ether-amide)s were in the range 0.40 to 0.91 dL/g indicating moderate molecular weight built-up. These poly(ether-amide)s exhibited excellent solubility in various polar aprotic solvent such as N-methyl pyrrolidone (NMP), N, N-dimethyl sulphoxide (DMSO), N, N-dimethyl acetamide (DMAc) N,N-dimethyl form amide (DMF) etc. All these poly(ether-amide)s were showed good solubility in pyridine-cresol and they partial solubility in common solvent such as THF, DCM. The initial decomposition temp (Ti) of synthesized poly(ether-amide)s were in the range 340 to 404 ºC. The 10% weight loss was in the range437º to 498ºC. The char yield of these poly(ether-amide)s were in the range 62.34 to 65.54 %.at 800 ºC. X-ray diffraction pattern showed that in corporation of methyl substituent on aromatic backbone and naphthyl moiety containing ether linkage and methylene spacer would disturb the chain regularity and packing, leading to amorphous nature. The structure-property relationship of naphthyl containingpoly(ether-amide)s was studied in the view of potential applications of these polymer as thermally stable and processable high performance polymers.

Keywords*2,2-Bis (p-methyl carboxyphenoxy) -1,1-binaphthyl (IV), poly(ether-amide)s, Solubility, Viscosity, Thermal stability.*

1. Introduction

Polyamides occupy an importantplace in the world of polymers because they offer excellent physio- chemical properties, thermal and oxidative stability, flame resistance, and superior mechanical and dielectric properties [1–3].However, due to their partial solubility in most organic solventsand high melting or softening temperatures, considering this challenges many efforts havebeen invested to modify the structure of polyamides by chemicalmeans to improve their solubility (process ability) without severelysacrificing their outstanding thermal properties [4–6]. Thesestudies include: Introducing flexible segments into the polymerchain [7–11], replacing symmetrical aromatic rings by the unsymmetricalones [12–14], introducing bulky pendent groups [15–22],and bringing a non-coplanar and alicyclic monomers into the polymerizationsystem [23–25].All these approach demonstrated that combining aryl ether, methylene linkages, and unsymmetrical, non-coplanar units always polymer backbone could increase the solubility of polyamides without sacrificing their high thermal

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stability. Hence, there has been an increased interest in the synthesis of polyamides which contain bearing ether and methylene linkages in the polymer backbone in order to improve their processability. In the present study series of new poly (ether-amide)s were synthesized by using Yamazaki 's direct phosphorylativepolycondensation of newdiacids (IV) monomer with commercial diamines such as 4,4'-diamino diphenyl ether (ODA), diaminodiphenyl methane (MDA), 4 amino phenyl sulfone , P-phenylenediamine, M-phenylenediamine. A new aromatic diacide monomer having naphthyl moiety with ether and methylene linkage was designed and synthesized. Further synthesized these poly (ether-amide) s were characterized by FT-IR spectroscopy, solubility, inherent viscosity, thermal analysis (TGA DSC) and X-ray diffraction Studies. Thus, our synthetic research effort has been directed towards structural modifications designed to disturb regularity and chain packing thus providing better solubility.

2. Experimental

2.1. Measurements:

FTIR spectra were recorded using monomer on a Perkin-Elmer Spectrum GX spectrophotometer. NMR spectra were recorded on a Bruker 400 MHz spectrometer for ^{1}H spectrum and 100 MHz for ^{13}C spectrum measurements using (d_6-⁻DMSO). Inherent viscosity of polymers was measured with 0.5 % (w/v) solution of polymer in N, N-dimethylformamide at 30±0.1 ^{o}C using an Ubbelohde suspended level viscometer. Solubility of polyamides were determined concentration of 0.5 g/mL in different solvents at room temperature or on heating. X-Ray diffraction patterns of polymers were obtained on a RigakuDmax 2500 X-ray diffractometer. Dried polymer powder was used for X- ray measurements. Thermogravimetric analysis (TGA) was performed on Perkin Elmer TGA-7 thermal analyzer system at a heating rate of 10 °C / minute under nitrogen atmosphere

2.2 Materials

All the reagents such as 2- Naphthalol , 4-fluo acetophenone 4,4'-diamino diphenyl ether (ODA), Diaminodiphenyl methane (MDA), 4-Amino phenyl sulfone , M-phenylenediamine, N, N-dimethyl sulphoxide (DMSO) and triphenyl phosphate were purchased from Sigma Aldrich and used as received. The p-Phenylenediamine was purified by recrystallization in ethanol. m-Cresol were purchased from Spectrochem and used as received. N, N-dimethylformamide was vaccum distilled from P_2O_5, N, N-dimethylacetamide was purified by vaccum distillation from barium oxide. Commercially available Sulfur was also purified by refluxing with calcium oxide for 30 min and reprcipitated with 1:1 HCl. Pyridine was refluxed with solid potassium hydroxide pellets, fractionally distilled and stored over Linde type 4 Å Molecular sieves. N-Methyl-2 pyrrolidone (NMP) was dried by azeotropic removal of water with benzene for 6 h, distilled under reduced pressure and stored over Linde type 4 Å Molecular sieves. Morpholine was dried with potassium hydroxide and fractionally distilled. Potassium carbonate (K_2CO_3) was dried under vacuum at 150 °C for 6h. Lithium chloride was dried under vacuum at 150°C for 6h.

2.3. Monomer Synthesis:

2.3.1. Synthesis of 2,2'-Dihydroxy-1,1binaphthyl (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 and heated to boiling point. 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 add a solution of 0.1 mole of 2-naphthol in 600 mL water is heated upto boiling; following by dropwise addition slowly through the dropping funnel and with vigorous stirring. The oily drops of 2-naphthol was disappeared and the*2,2'-Dihydroxy-1,1binaphthyl* (I) separates out in flasks boiled for 10 minutes. The hot suspension was filtered at the pump through the previously warmed buckner funnel; the product was washed with boiling water and dried well. The*2,2'-Dihydroxy-1,1binaphthyl* was recrystallized from toluene (about 150 mL) to get colorless crystals.

Yield: 13.12 g (92.25 %)

Melting point: 217 °C.

2.3.2. Synthesis of 2,2'-Bis(p-acetophenone)-1,1binaphthyl (II): In 500 mL three neck round bottom flask equipped with calcium chloride guard tube, thermowell, N_2 gas inlet were placed 14.3 g *2,2'-Dihydroxy-*

1,1binaphthyl (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 K_2CO_3 was added . The resulting reaction mixture was refluxed for 5 h .The progress of reaction was studied by TLC method. After completion of reaction mixture was cooled at room temp. Then water was added to reaction mixture 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 %)

Melting point: 130 °C

IR: 3033, 2974, 1696, 1594, 1403, 1222, 1071, 1062, 815,774 cm^{-1}.

2.3.3. Synthesis of 2,2'-Bis (p- thioacetomorpholide)-1,1binaphthyl (III) :In 100 mL round bottom flask equipped with reflux condenser and magnetic stirrer were placed 10.44 g (0.02 mol) of diacetyl 1,1-bis -2-napthol,1.84g (0.06) Sulphur and 5.227g (0.06mol) of morpholine (5.2mL). The resulting reaction mixture was stirred and reflux gently until the evolution of hydrogen sulphide subsides and more vigorously for 14 h at 140 °C. The reaction mixture was allowed to cool at R.T. 90 mL ethanol was added to obtain buff colored product which was filtered, washed with excess ethanol and dried. . The product was confirmed by FTIR Spectra.
Yield: 11.80 g (81.49 %)

Melting point: 145 °C.

IR: 3021, 2951, 1661, 1595, 1490, 1230, 1181, 1014, 827,781 cm^{-1}.

2.3.4. 2,2-Bis (p-methyl carboxyphenoxy) -1,1-binaphthyl (IV) :In a 500 mL round flask equipped with reflux condenser, magnetic stirrer were placed 10.84 g (0.015 mole) of (III) and 200 mL ethanolic 10% NaOH solution, reaction mixture was refluxed with stirring for 12 h. The most of ethanol was distilled out under reduced pressure. To the residual product 200 mL hot water was added and filtered. The filtrate was acidified by 1:1 HCL. The precipitated product diacid (IV) was filtered, washed thoroughly with hot water and dried under vacuum.
Yield: 7.23. g (84.75 %)

Melting point: 140 °C.

IR:3055, 1696, 1505, 1596, 1403, 1016, 943, 774 cm^{-1}
^{1}H NMR (d_6-⁻DMSO): δ = 12.8 (s, 2H), 8.14 (d, 2H), 7.92 (d, 2H), 7.49 (t, 2H), 7.32 (t, 2H), 7.29 (d, 2H), 7.14 (d, 2H), 6.81(d, 4H), 6.71(d, 4H), 3.53 (s, 4H).
^{13}C NMR (d_6- CDCl,): δ = 168.95, 151.48, 147.68, 129.32, 125.51, 124.81, 124.21, 123.26, 121.77, 120.83, 119.84, 117.17, 114.46, 113.93, 113.80, 35.66.
Mass Spectra m/e (m+1) = 554

2.4. Synthesis of poly (ether-amide)s

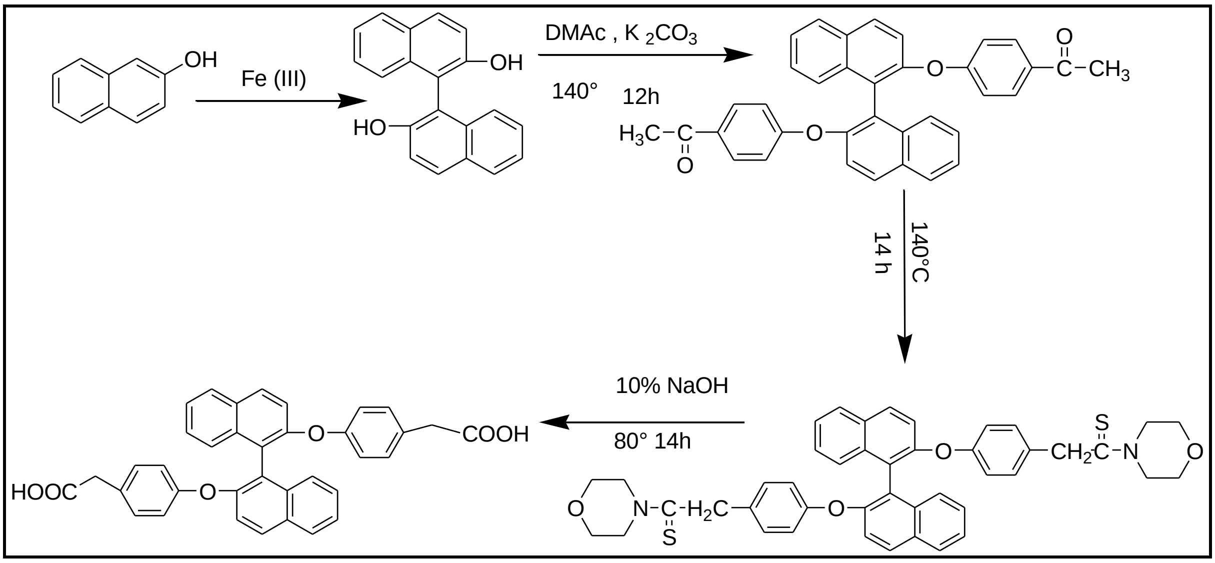
In a 100 mL three neck round bottom flask equipped with reflux condenser, magnetic stirrer, calcium chloride guard tube and nitrogen gas inlet were placed 0.428 g 1, 5-naphthol diacid (1mmol), 0.200g (1mmol) 4,4'-diaminodiphenyl ether (ODA), 0.115g lithium chloride [5 wt % based on solvent N-methyl pyrrolidone (NMP) and pyridine mixture] and 0.744g (0.63 mL, 2.4 mmol) triphenylphosphite (TPP), 0.5 mL pyridine and 2 mL NMP. The mixture was stirred well and temperature was slowly raised to 100°C over a period of 30 min. The mixture was heated at 130°C for 3h under nitrogen. After cooling, the resulting viscous solution was poured into 200 mL of methanol under vigorous stirring. The precipitated polymer (PEA-1) was filtered, washed with methanol and dried. The polymer was purified by dissolving in N, N-dimethylacetamide (DMAc) and reprecipitating in methanol. It was filtered, washed with methanol and dried under vacuum at 100°C for 6 h. The yield was 97% and the viscosity of polymer in DMAc was 0.46dL/g. The poly (ether-amide) s PEA-2 to PEA-5 was synthesized with varying diamines by similar procedure.

3. Results And Discussion

3.1. Monomer Synthesis

New dicarboxylicacid 2,2-Bis (p-methyl carboxyphenoxy) -1,1-binaphthyl were successfully synthesized in several steps (Scheme-1). The 2, 2'-*Bis (p-acetophenone)-1,1binaphthyl* (II) was obtained reacting with 4-Fluoroacetophenone by using Potassium carbonate as catalyst in DMAc.

Reaction Scheme: 1



Scheme 1: Synthesis of 2,2-Bis (p-methyl carboxyphenoxy) -1,1-binaphthyl (IV)

The infrared spectrum of (II) (**Fig. 1**) showed medium strong absorption bands at 3033 and 2974 cm^{-1} showing the presence of aliphatic C-H stretching corresponding to the in phase and out phase vibration of bisnaphthyland methylene groups. Spectrum also shows absorption near 1403 due to C-H bending vibration. Absorption at 1696 cm^{-1} of carbonyl (C=O) stretching adsorption indicated acetyl carbonyl moiety. The peak at 1222 and 1071 shows C-O-C stretch.

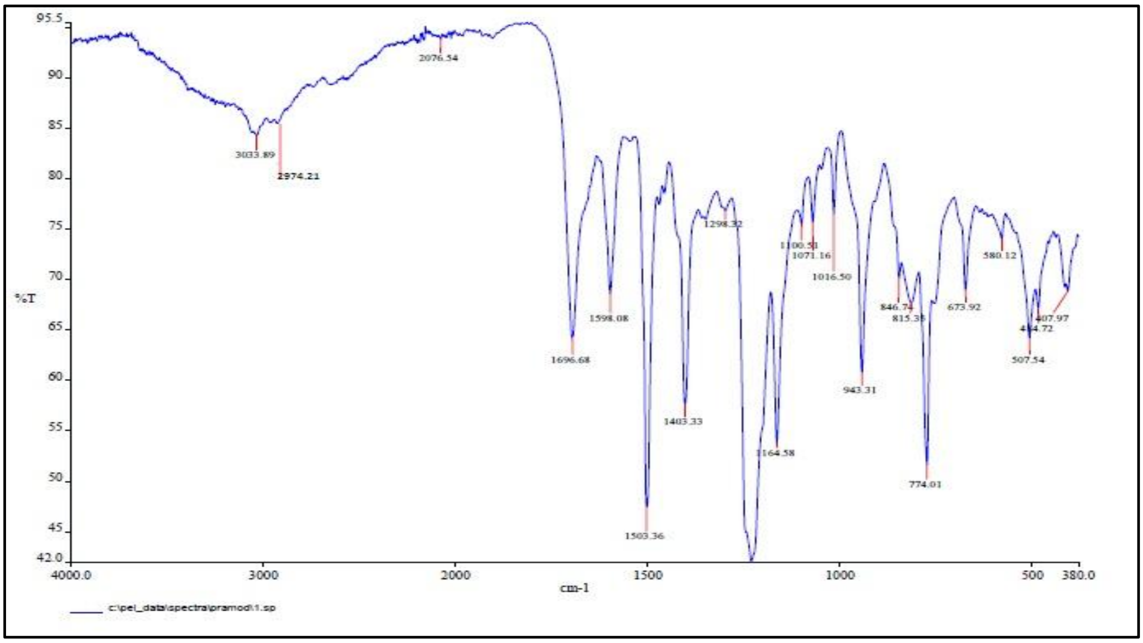


Fig 1:FT-IR spectrum of 2, 2'-Bis (p-acetophenone)- 1,1 binaphthyl (II)

Bis (p-acetophenone)- 1,1 binaphthyl (II)The formation of intermediate thiomorpholide derivative (III) was confirmed by infrared spectroscopy. The infrared spectrum of III (**Fig. 2**) showed medium strong absorption bands at 3021 and 2951 cm^{-1}, due to the presence of aliphatic C-H stretching corresponding to the in phase and out phase vibration of bisnaphthyl unit and methylene group. Spectrum also shows absorption near 1490 cm^{-1} due to C-H bending vibration. The appearance of absorption at 1230 cm^{-1} (C=S) confirm formation of thiomorpholide.

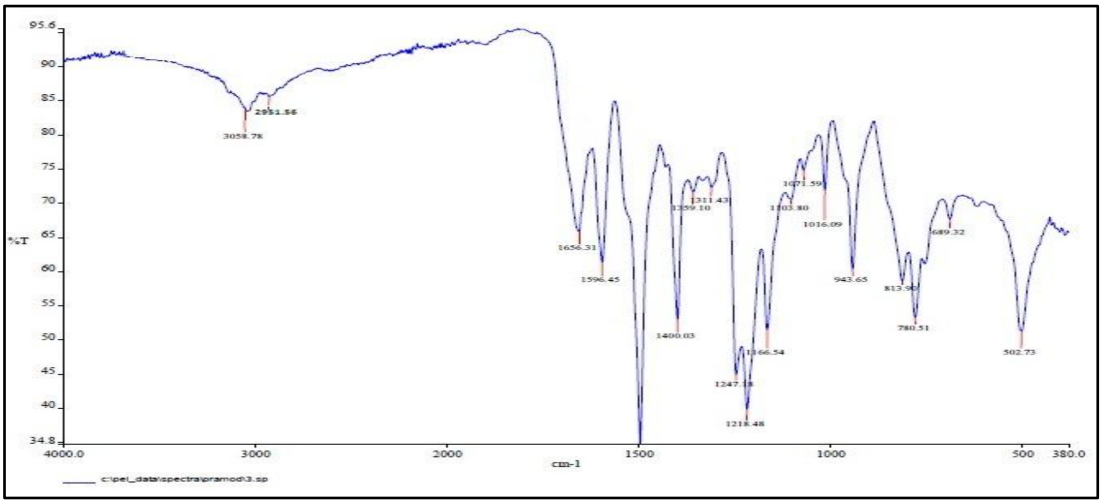


Fig 2: FT-IR spectrum of *2,2'-Bis (p*-thioacetomorpholid*e)-1,1binaphthyl* (III)

The 2, 2-Bis (p-methyl carboxyphenoxy) -1,1-binaphthyl (IV) was obtained by the Conrad Willgerodt reaction with Kindler variation. Thiomorpholide derivative on alkaline hydrolysis yielded the 2, 2-Bis (p-methyl carboxyphenoxy) -1,1-binaphthyl (IV) . The structure of (IV) was confirmed by IR, NMR (^{1}H and ^{13}C) and mass spectroscopy. The infrared spectrum of 2,2-Bis (p-methyl carboxyphenoxy) -1,1-binaphthyl (IV) (Fig. 3) showed absorption bands at 3156 cm^{-1} (-COOH stretching) and 1696 cm^{-1} (C=O) indicating the presence of carboxyl group. The IR spectrum also showed the absorption bands at 3033 and 2910 cm^{-1} corresponding to the in plane and out of plane stretching vibrations of the (-CH$_2$-) of the methylene group, and naphthelene unit. Spectrum also shows absorption near 1503 cm^{-1} due to C-H bending vibration.

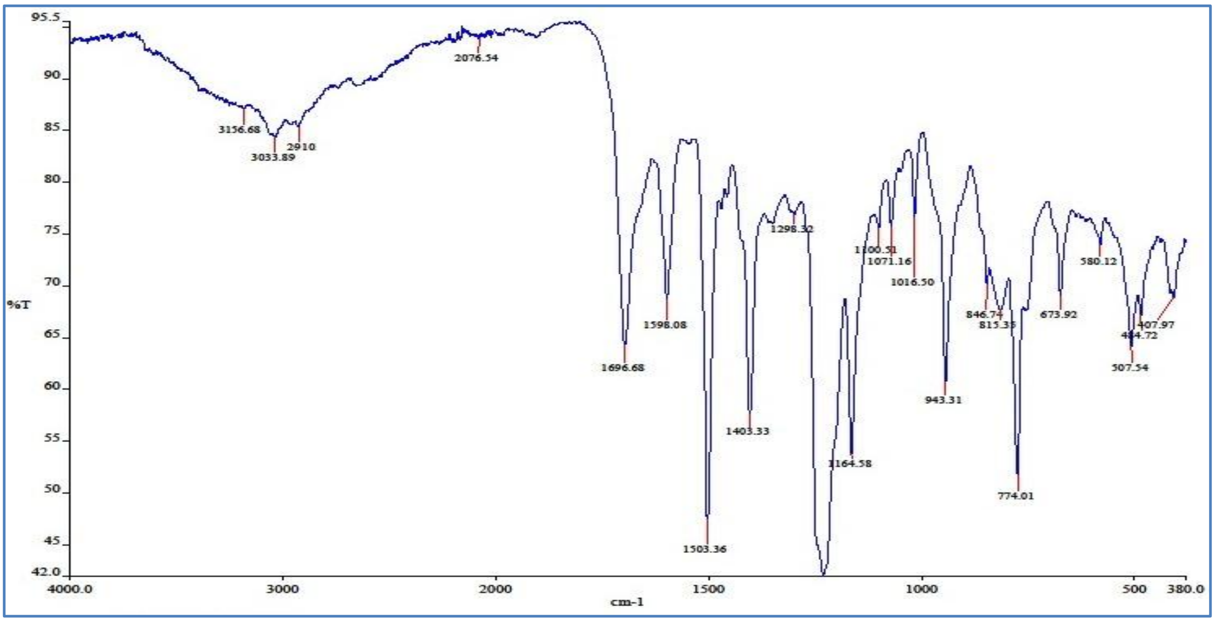


Fig 3:FT-IR spectrum of 2,2-Bis (p-methyl carboxyphenoxy) -1,1-binaphthyl (IV)

The proton NMR spectrum (**Fig. 4**) of (III) showed the NMR singlet at 12.8 δ corresponding to group of acid proton –COOH. The signals in the range of 6.81 and 6.71 δ of (8H) are attributed to the aromatic protons of phenylene rings whereas signal at 8.14, 7.92, 7.49,7.32, 7.29, and 7.14 δ(12H) are assigned to naphthalene proton. The NMR signal appears at 3.53 δ singlet attributed to methylene (2H) of –CH_2COOH group attached to aromatic ring.

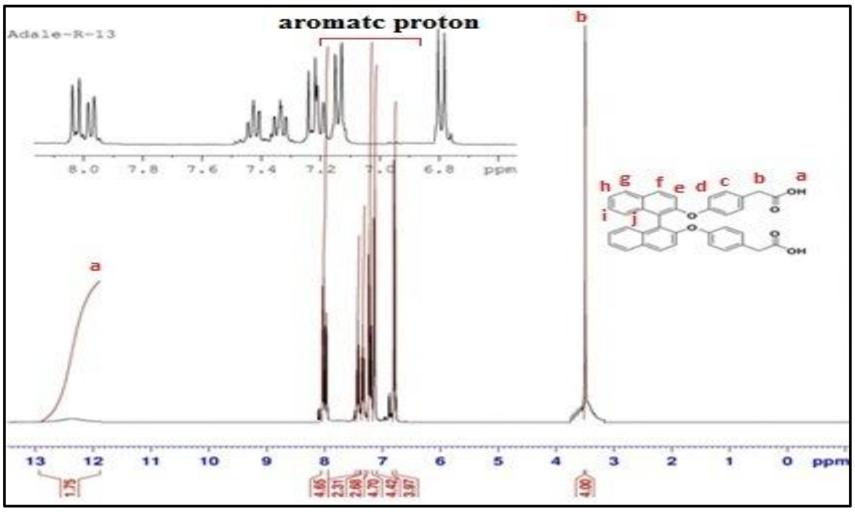


Fig 4:^{1}H-NMR of 2,2-bis (p-methyl carboxyphenoxy) -1,1-binaphthyl (IV)

^{13}C NMR spectrum (**Fig. 5**) of 2,2-bis (p-methyl carboxyphenoxy) -1,1-binaphthyl (IV) showed 16NMR signals corresponding sixteen types of different carbons of which carbonyl carbon appeared at 168.95 δ for (C=O); whereas quaternary carbons showed signals at151.48, 147.68,129.32, 125.51,123.26 119.84 δ. The CH carbons appeared at124.82, 124.21, 123.27, 121.78, 120.83, 119.85, 114.46, and 113.93 δ whereas CH_2 carbon gave NMR signals at 35.66
δ confirms the formation of methyl substituted 2,2-bis (p-methyl carboxyphenoxy) -1,1-binaphthyl (IV)diacid monomer.

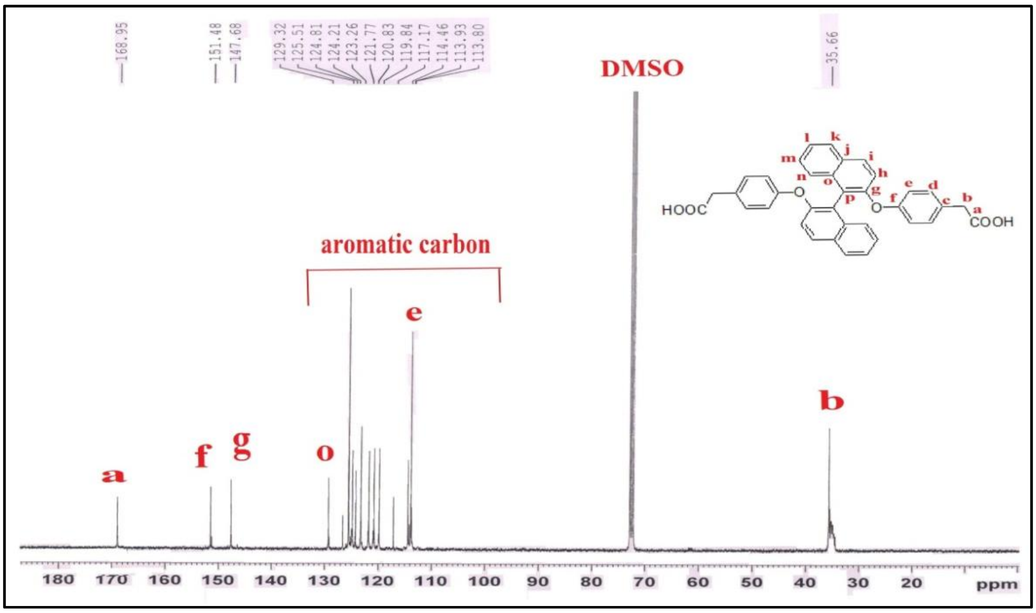


Fig 5:^{13}C-NMR OF 2, 2-bis (p-methyl carboxyphenoxy) -1,1-binaphthyl (IV)

The DEPT spectrum (Fig. 6) of 2, 2-bis (p-methyl carboxyphenoxy) -1,1-binaphthyl (IV) 1, 5-naphthol diacetic acids (III) also confirms the diacid monomer where all the quaternary carbons are absent in the spectrum and the peaks of CH carbons are upper sides at124.82,124.21, 123.27, 121.78, 120.83, 119.85, 114.46, and 113.93 CH_2 appeared at down side at 35.66 δ.

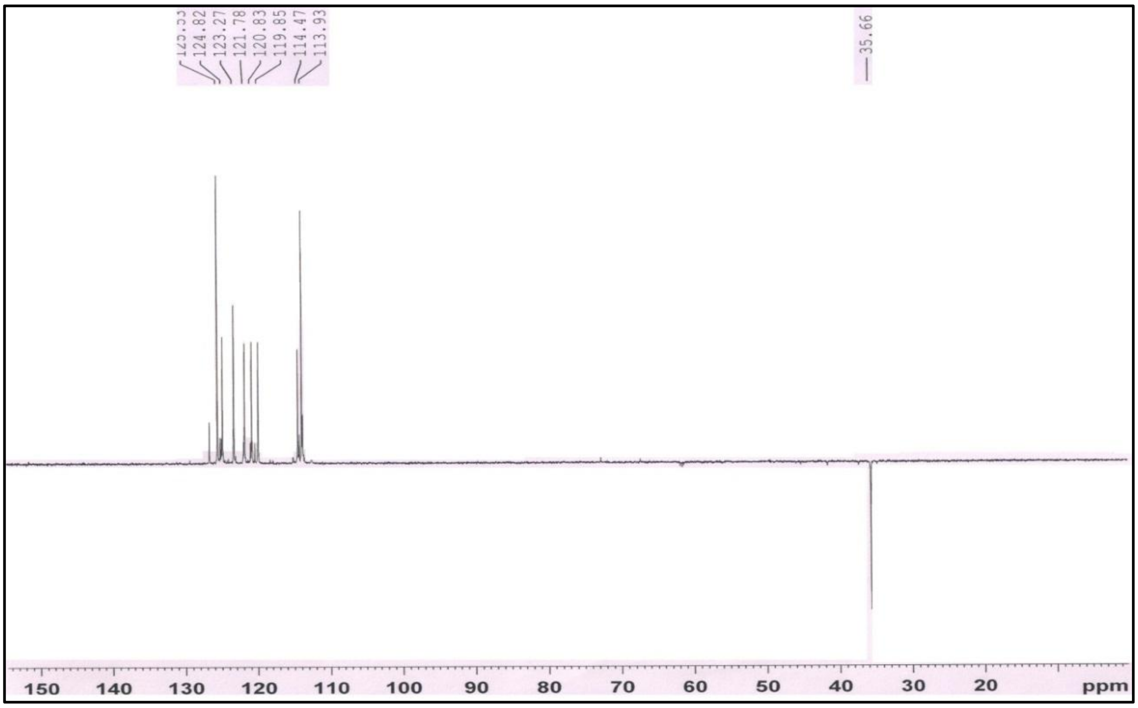


Fig 6: DEPT spectrum of2,2-bis (p-methyl carboxyphenoxy) -1,1-binaphthyl (IV)

The mass spectrum of 2,2-bis (p-methyl carboxyphenoxy) -1,1-binaphthyl (IV) (**Fig. 7**) showed molecular ion peak at m/e (m+1) 554 corresponding to molecular weight of 2,2-bis (p-methyl carboxyphenoxy) -1,1-binaphthyl (IV)

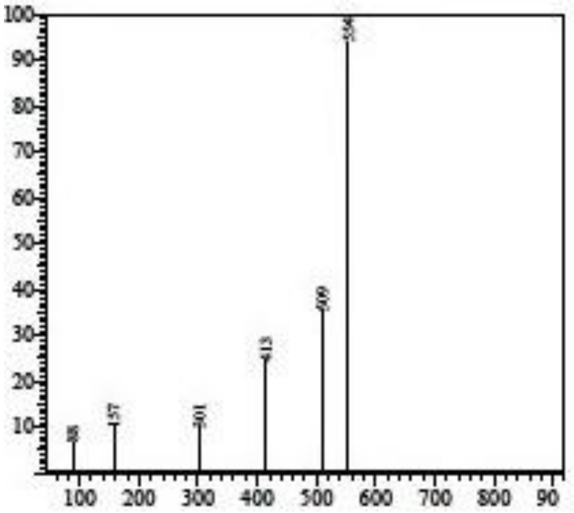
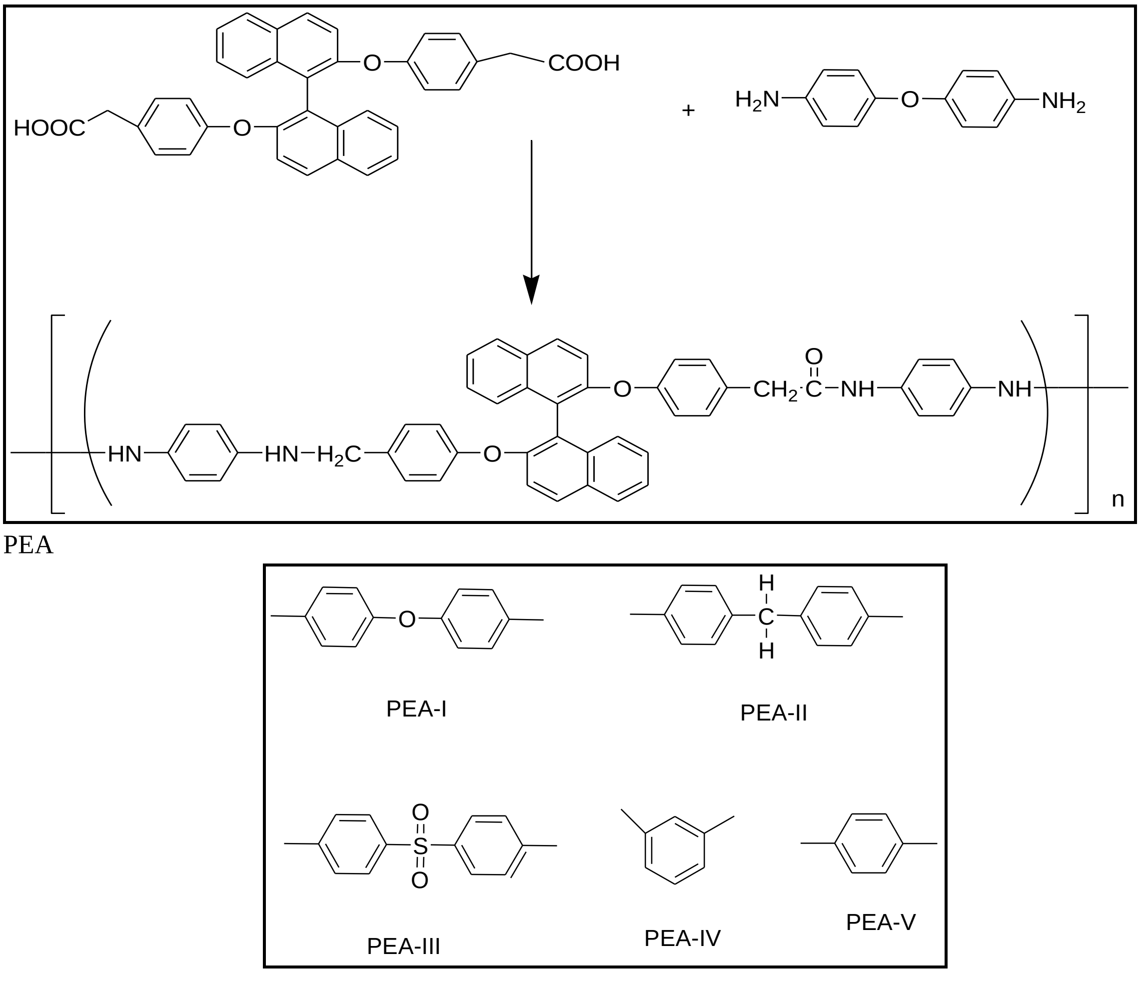


Fig 7: Mass spectrum of 2,2-bis (p-methyl carboxyphenoxy) -1,1-binaphthyl (IV)

3.2 Polymerization

The poly(ether-amide)s containing naphthyl group were synthesizedYamazaki's phosphorylation method .Series of poly (ether-amide)s were synthesized from the stoichiometric quantities of naphthyldiacid and various aromatic diamines by direct polycondensation method (Scheme 2), triphenylphosphite was used as the condensing agent, where in the mixture of NMP and pyridine (4:1 by volume) containing 8 wt. % anhydrous lithium chloride was employed as solvent. The polymerization was carried out at 100°C for 3 h. The different aromatic diamines used are shown below. The polymerization proceeded smoothly giving highly viscous solution. The resulting polymers were precipitated by pouring the viscous solutions in methanol.

Reaction Scheme 2:



Scheme 2: Synthesis of poly (ether-amide)s (PEA-I to PEA-V)

Polymer Characterization

The result obtained are present in table –I. All polyamides obtained good yield as white fibrous material. The inherent viscosities of their **synthesized** polymer were in the range 0.40 to 0.91 dL/g respectively. This indicate from of moderate to high M.W. of polyamides. The polymerization was carried out with 1mmol of naphtholdiacid and 1mmol of.

Table 1: Viscosity of poly (ether-amide) s

Polymer	**Diamine**	Inherent viscosity dL/g
PEA I	**ODA**	**0.46**
PEA II	**MDA**	**0.58**
PEA III	**SDA**	**0.40**
PEA IV	**M-BDA**	**0.72**
PEA IV	**P-BDA**	**0.91**

The structures of polymers were characterized by infrared spectroscopy. Polyamide PA-1 (**Fig. 8**) showed an absorption band at 3300 cm^{-1} (-NH stretching), a sharp band at 1500 cm^{-1} (characteristic for –NH bending) and absorption band at 1660 cm^{-1} (due to C=O in amide group). The absorption band at 3031 cm,$^{-1}$ characteristic for aromatic –CH stretching.

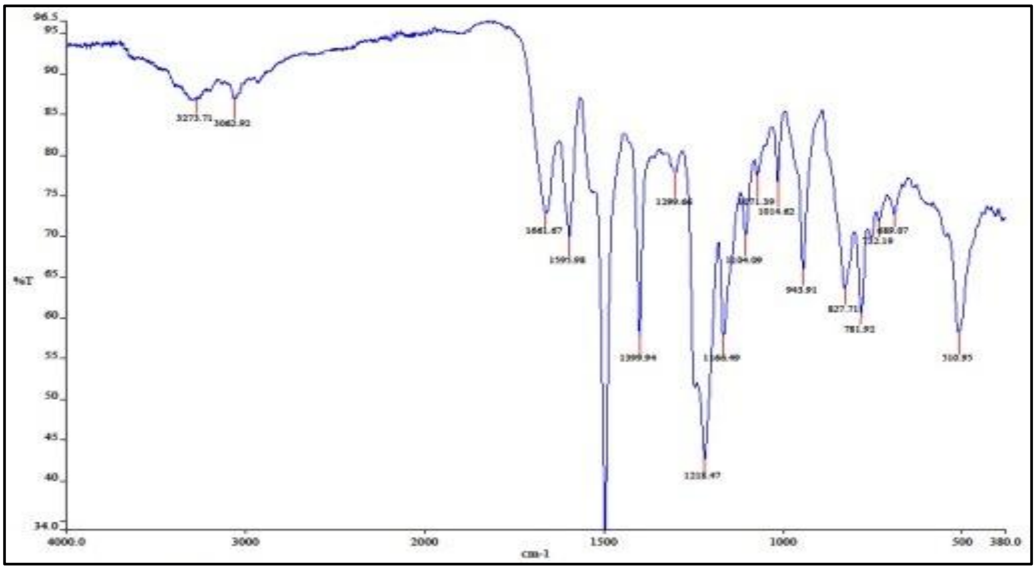


Fig 8: FT-IR spectrum OF PEA-I

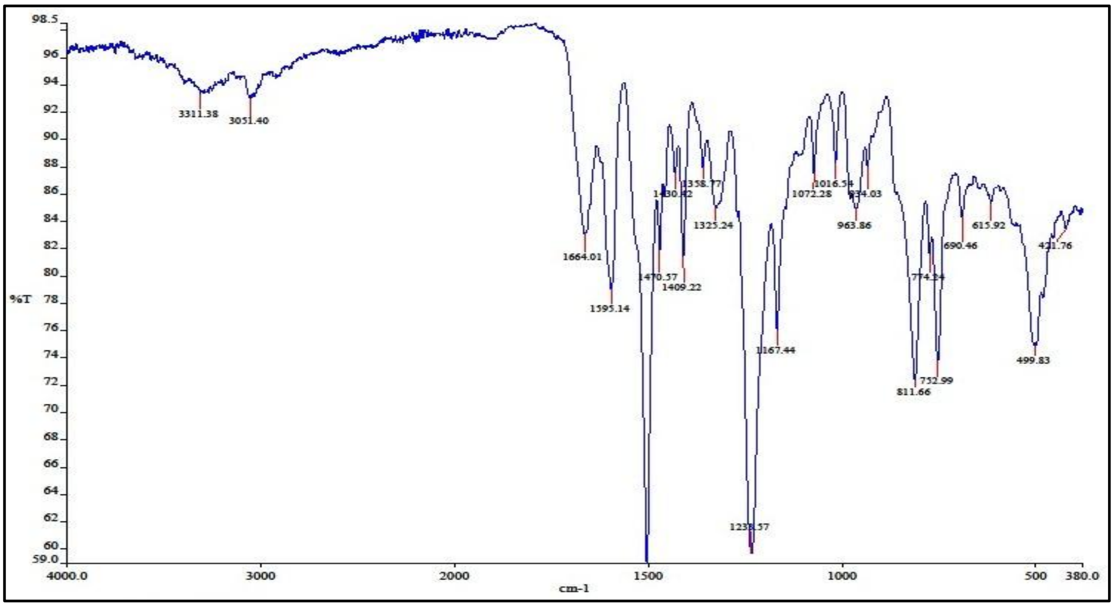


Fig 9: FT-IR spectrum OF PEA-II

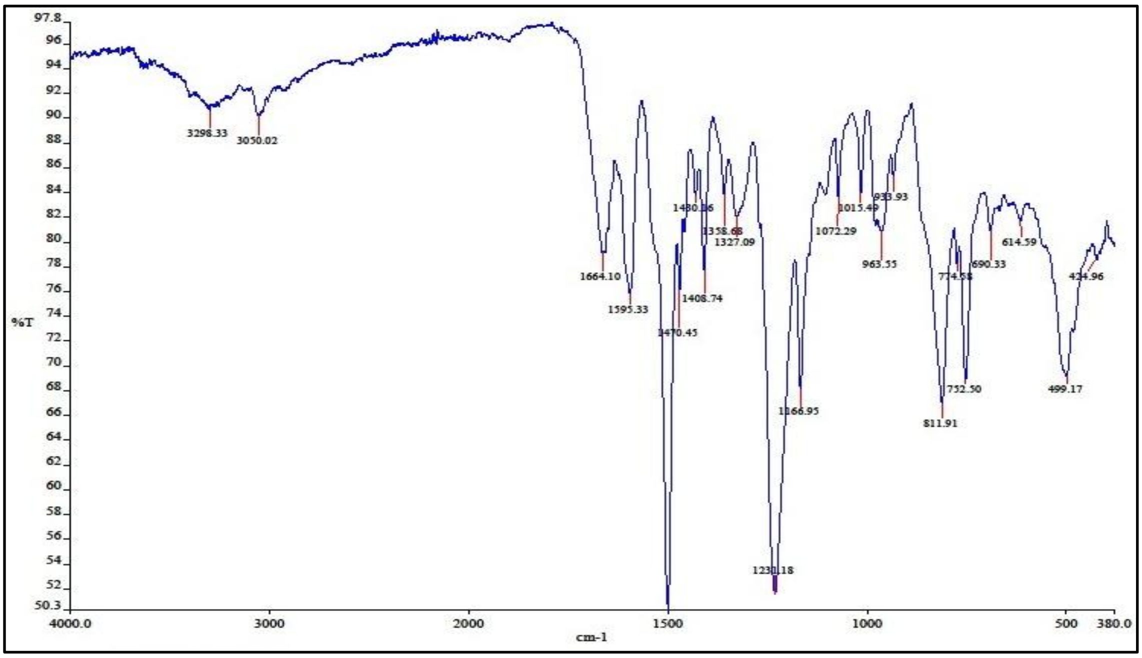


Fig 10: FT-IR spectrum OF PEA-V

Table 2: Solubility of poly (ether-amide) s

Solvent	PEA I	PEA II	PEA III	PEA IV	PEA V
H_2SO_4	++	++	++	++	++
DMAc	++	++	++	++	++
DMSO	++	++	++	++	++
DMF	++	++	++	++	++
THF	+-	+-	+-	+-	+-
DCM	+-	--	+-	+-	+-
m-Cresol	++	++	++	++	++
Pyridine	++	++	++	++	++

(++) Soluble at room temperature, (+) partially soluble, (+-) soluble after heating, (--) insoluble,

[a] Solubility: measured at apolymer concentration of 0.05 g/mL.

[b] NMP: N-methyl-2-pyrrolidone; DMSO: dimethyl sulfoxide; DMAc: N,N-dimethylformamide; THF: tetrahydrofuran.

Table II shows the solubility behavior of polyetheramides .all polyetheramide are mostly soluble in polar aprotic organic solvent such as DMAc,DMSO, DMF. Only THF and DCM these tow solvent polyetheamide are partially soluble.solubility of polyethramide was greatly improved by ether linkage and addition of naphthy group.

Thermogravimetric analysis (TGA)

Table 3: Thermal Properties of poly (ether-amide)

Polymer Code	T_i (ºC) in N_2	T_d (ºC) in N_2	ResidualWt% at 800ºC
PEA I	340	437	62.34
PEA II	365	461	63.51
PEA III	360	456	63.28
PEA IV	376	473	63.95
PEA V	405	498	65.54

[a]Temprature at which onset of decomposition was recorded by TG at heating rate of 10ºC/min.
T_i-Initial decomposition temperature.
T_d–Temperature of 10% decomposition.

Thermal behavior of polymer was evaluated by means of dynamic thermogravimetric and differential scanning calorimetry incorporate the thermal data such as initial decomposition temperature (T_i), 10% Wt. loss temperature (T_{10}) and residual weight at 800ºC.

The thermal stability of the polyamides was studied by thermogravimetric analysis at a heating rate of 10 ºC/min in nitrogen atmosphere. The initial weight loss (T_i) temperature at which 10% Wt. Loss andchar yield at 800 ºC were determined from the original thermograms. (T_i) values range in bet ween 340 to 404 ºC. T_{10} values between 437 to 498 ºC, respectively. Residual wt% loss at 800 ºC was in the range of 62.34 to 665.54 %.

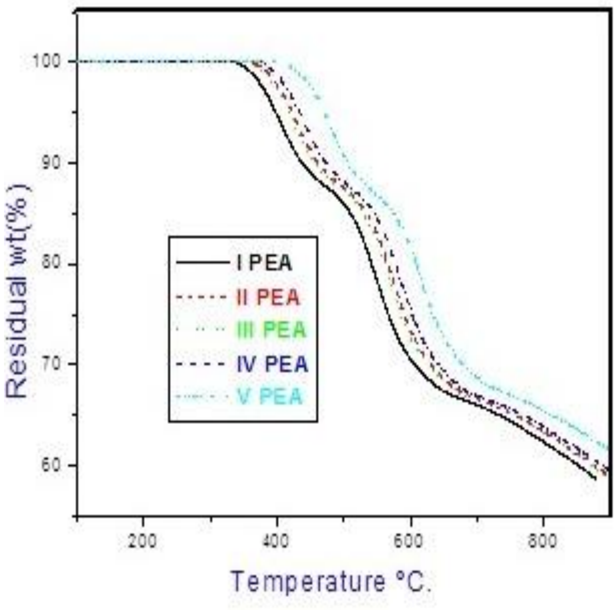


Fig 11: TGA curve ofpoly (ether-amide)

X-Ray diffraction Studies-

All the poly (ether-amide)s were structurally characterized by wide - angle X-Ray diffraction (WAXD) studies . It is observed that PEA-I to PEA-IV all of polymers completely amorphous. But PEA-V are observed semi crystalline nature. Thus, the amorphous nature of these polymer was reflected in there excellent solubility.

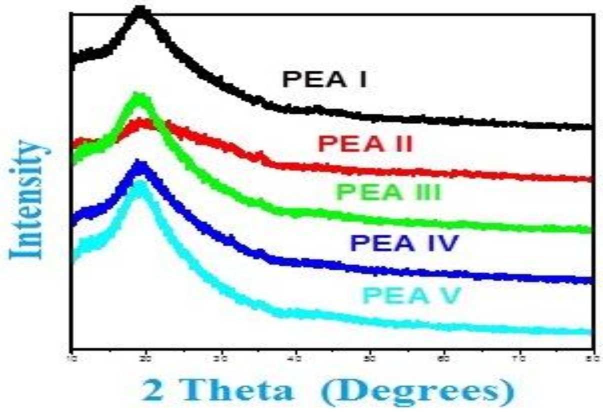


Fig 12: XRD curve of poly (ether-amide**)**

4. Conclusion

New poly (ether-amide)s containing binaphthylbulky, kinked, and cranked polyamide backbone improved solubility &Processability of the polymers in various organic solvents. The inherent viscosities of theses polymer range from 0.40 to 0.90 dL/g. these polymers are amorphous and partly or completely soluble in a number of organic solvent, such as DMAc,

DMSO, DMF, THF, DCM, m-Cresol, Pyridine etc. The Td's for 10% weight loss in nitrogen range from387 to 444 °C. These polymers are used in high-tech artificial fibers in the manufacturing of protective apparel for firemen, soldiers, race car drives, etc. application

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