Naphthalene-Based Poly (Ether-Amide)S Bearing Metylene Linkage To Enhance Processability

[1]V. N. Kadam , [2]N. N. Raut, [3]M. V. Gaikwad, [4]S. G. Gaikwad, [5]T. B. Mhamane, [6]S. P. Shirame, [7]V. P. Ubale, [8*]A. A. Ghanwat

[1] [2] [3] [4] [5] [6] BaburaoPatil College of Arts & Science, Anagar-413213, Maharashtra, India.
[8]School of Chemical Sciences, Solapur University, Kegaon, Solapur- 413 255, Maharashtra, India.
[7] D.B.F. Dayanand College of Arts & Science, Solapur-413002, Maharashtra, India.

Abstract: New diacid monomer 4,4′- (1,5-Naphthalenedioxy)dibenzoicacid(III) was prepared through reaction of 1,5-naphthol followed by 4-fluoroacetophenone to 4,4′-(1,5-Naphthalenedioxy) diacetyl(I) followed by Conrad Willgerodt reaction with Kindler variation using sulphur and morpholine formed 4,4′-(1,5-Naphthalenedioxy) thioacetomorpholide (II) further, new diacid (III) 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 (III) 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 0.35 to 0.81 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 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 396ºC to 420 ºC. The 10% weight loss was in the range491º to 516ºC. The char yield of these poly(ether-amide)s were in the range 45.26 to 59.26 % 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:*4,4′- (1,5-Naphthalenedioxy)dibenzoicacid(III), poly(ether-amide)s, Solubility, Viscosity, Thermal stability.*

1. Introduction

Polyamidesare recognized as high performance polymeric material with outstanding mechanical properties, good chemical resistance and excellent thermal stability [1-5].Due to the combination of these specific features, they are widely used in various industries. The demand for aromatic polyamides and other high performance polymericmaterials is growing steadily because of their superior performance characteristics that the increasingly expected from engineering polymers in the aero-space, electronic, automobile and other industries [6,7].However, their industrial applications were rather restricted by difficulty in fabrication due to rigid rod type structure leads to extremely high melting temperature and enhance solubility in most organic solvents. It is highly desirable that these polymers be soluble in some specific organic solvent to facilitate processing, however they should retain resistance common solvents during their use. Therefore, severalattempts have been tried to enhance their processability. One of the useful approach is the introduction of flexible groups such as aryl ether linkage into the aromatic polyamide backbone[8-14].Same of the approaches that have been successfully used were introducing flexible segments into the polymer chain [15–19], replacing symmetrical aromatic rings by the unsymmetrical ones [20–22], introducing bulky pendent groups [23–30], and bringing a non-coplanar and alicyclic monomers into the polymerization system [31–33].]. 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 stability. Hence, there has been an increased interest in the synthesis of polyamides which contain pendant methyl group and bearing ether, 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 (III) 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 1,5Naphthalol, 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 with1:1 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 of4,4'-(1,5-Naphthalenedioxy) diacetyl(I):*A 500 mL three neck round bottom flask equipped with calcium chloride guard tube, thermowell, N_2 gas inletr,* were placed 8 g 1,5-Naphthol (0.05 mol) and 13.814 g 4-Fluoroacetophenone (0.1mol) in 125 mL N,N-dimethyl acetamide (DMAc), then 13.821 g of anhydrous K_2CO_3 was added with vigorous strring . 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: 16.60 g (83.83 %)

M. P: 144 °C.

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

2.3.2. Synthesis of 4,4'-(1,5-Naphthalenedioxy)thioacetomorpholide (II):A 100 mL round bottom flask equipped with condenser and magnetic stirrer were placed 7.92 g (0.02 mol) of diacetyl 1,5 -napthol1.84g (0.06) Sulphur and 5.227g (0.06mol) of morpholine (5.2mL). The resulting reaction mixture was stirred and reflux the gently until the evolution of hydrogen sulphide subsides and more vigorously for 14 h at 140 $^{\circ}$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: 8.40 g (82.36 %)

M. P: 159 °C.

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

2.3.3. Synthesis of4,4′-(1,5-Naphthalenedioxy)dibenzoicacid(III):In 500 mL round flask equipped with reflux condenser, magnetic stirrer were placed 0.765 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 (III) was filtered, washed thoroughly with hot water and dried under vaccum.

Yield: 5.70 g (88.78 %)

M. P: 148 °C

IR: 3240 (-OH Stretch) 3055, 1696(C=O stretch), 1505, 1596, 1403, 1016, 943, 774 cm^{-1}
^{1}**H NMR** (d_6-⁻DMSO): δ = 12.3 (s, 2H), 7.92 (dd, 2H), 7.87 (dd, 4H), 7.5 (t,2H),7.34 (dd 4H), 7.12 (dd,2H) ,3.51 (s,4H)
^{13}**C NMR** (d_6- CDCl,): δ = 173, 156 , 153, 131, 130, 128, 126, 118, 117, 114, 40 ,
Mass Spectra m/e (m+1) = 428

2.4. Synthesis of poly (ether-amide)s

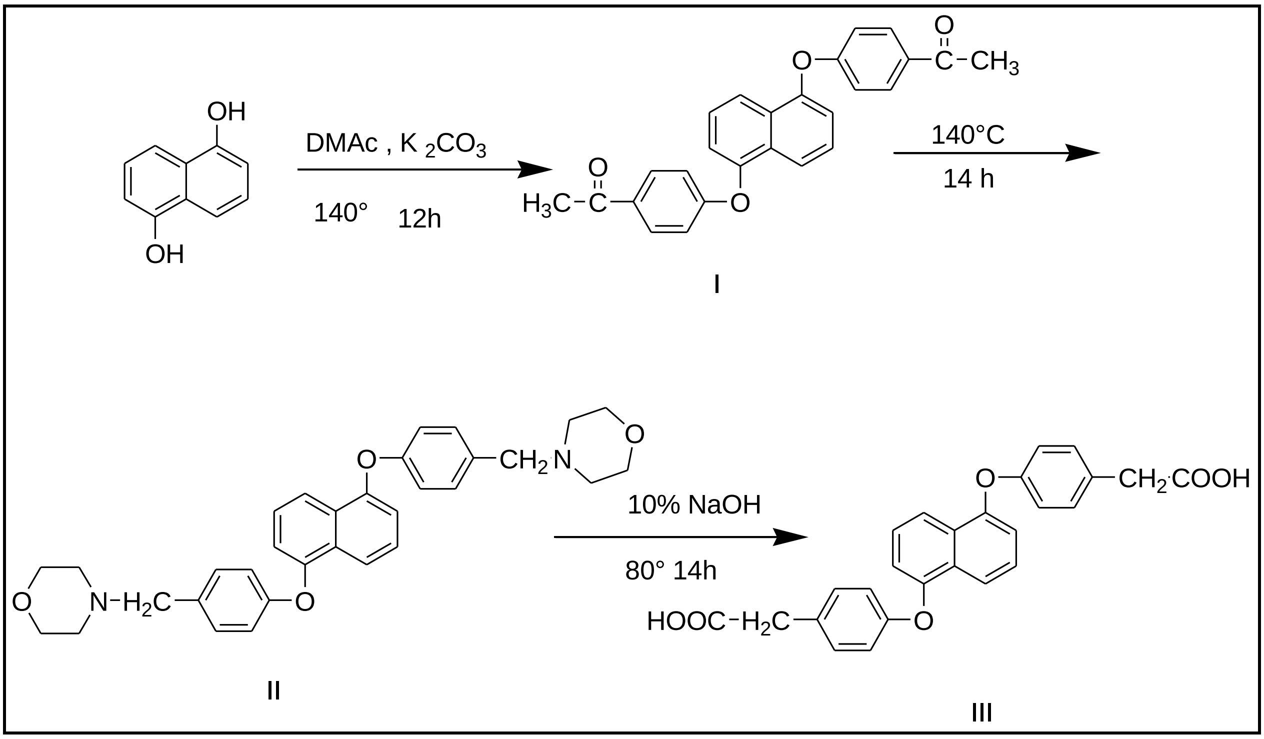
In a 100 mL three neck round bottom flask equipped with reflux condenser, magnetic stirrer, calcium chloride guard tube and nitrogen gas inlet were placed 0.428 g 4,4′- (1,5-Naphthalenedioxy)dibenzoicacid(1mmol), 0.200g (1mmol) 4,4’-diaminodiphenyl ether (ODA), 0.115g lithium chloride [5 wt % based on 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 0.41 dL/g. The poly (ether-amide)s PEA-2 to PEA-5 was synthesized with varying diamines by similar procedure.

3. Result And Discussion

3.1. Monomer Synthesis

New dicarboxylicacid4,4′- (1,5-Naphthalenedioxy)dibenzoicacid(III) were successfully synthesized from 1,5-naphthol in several steps (Scheme-1). The 4,4′-(1,5-Naphthalenedioxy) diacetyl(I) was obtained reacting 1,5-naphthol with 4-Fluoroacetophenone by using Potassium carbonate as catalyst in DMAc.

Reaction Scheme:1



Scheme 1: Synthesis of 4,4′- (1,5-Naphthalenedioxy)dibenzoicacid(III)

The infrared spectrum of(I) (**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 naphthyl and 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.

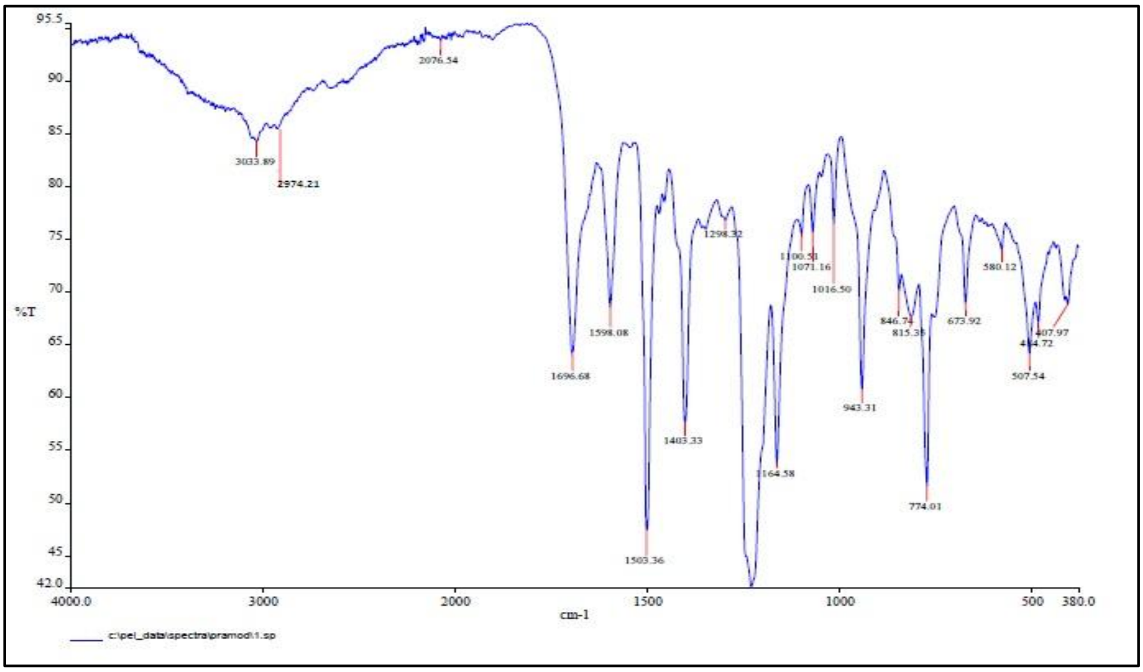


Fig 1:FT-IR Spectrum of 4,4’-(1,5-Naphthalenedioxy) diacetyl(I)

The formation of intermediate thiomorpholide derivative (II) was confirmed by infrared spectroscopy. The infrared spectrum of II (**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 Nephthyl 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.

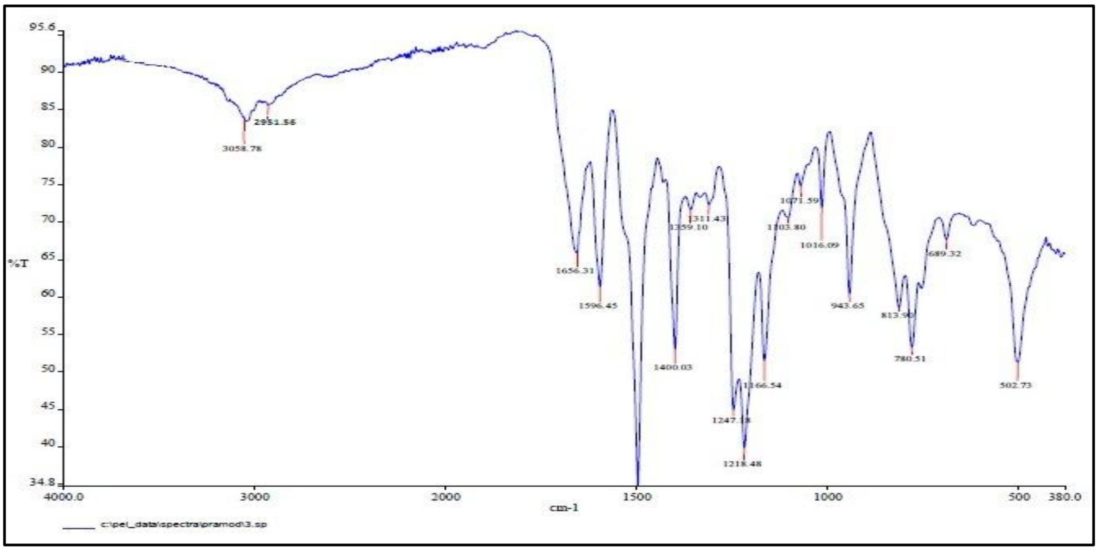


Fig 2:FT-IR spectrum of 4,4′-(1,5-Naphthalenedioxy) thioacetomorpholide (II)

4,4′- (1,5-Naphthalenedioxy)dibenzoicacid(III) was obtained by the Conrad Willgerodt reaction with Kindler variation.Thiomorpholide derivative on alkaline hydrolysis yielded the 4,4′- (1,5-Naphthalenedioxy)dibenzoicacid(III) . The structure of (III) was confirmed by IR, NMR (^{1}H and ^{13}C) and mass spectroscopy. The infrared spectrum of 4,4′- (1,5-Naphthalenedioxy)dibenzoicacid(III) (Fig. 3) showed absorption bands at 3104 cm^{-1} (-COOH stretching) and 1712 cm^{-1} (C=O) indicating the presence of carboxyl group. The IR spectrum also showed the absorption bands at 3012 and 2856 cm^{-1} corresponding to the in plane and out of plane stretching vibrations of the ($-CH_2-$) of the methyl group and naphthelene unit. Spectrum also shows absorption near 1492 cm^{-1} due to C-H bending vibration.

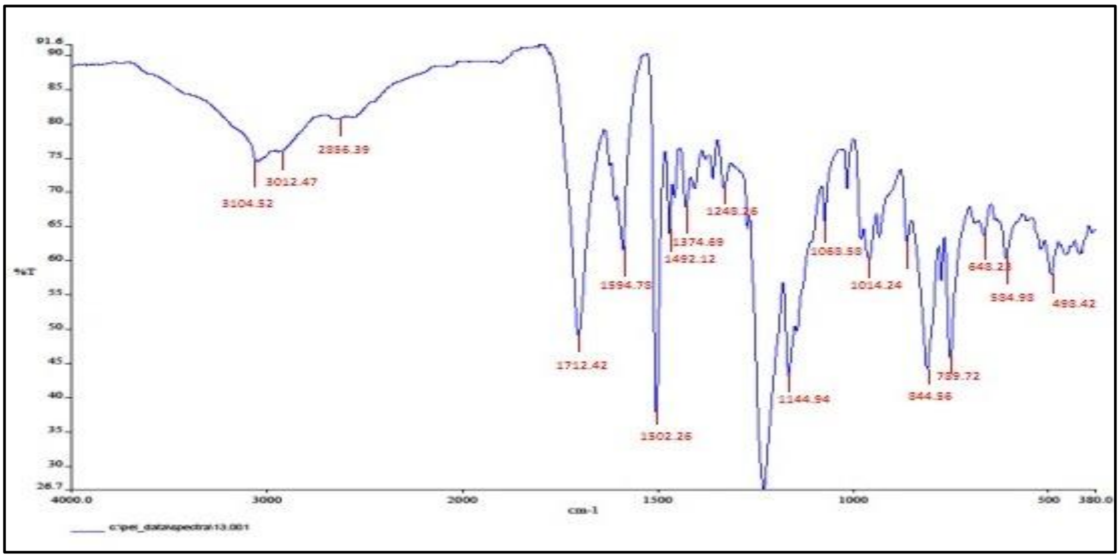


Fig 3:FT-IR spectrum of 4,4′- (1,5-Naphthalenedioxy)dibenzoicacid(III)

The proton NMR spectrum (**Fig. 4**) of (III) showed the NMR singlet at 12.3 δ corresponding to group of acid proton –COOH. The signals in the range of 7.34 and 7.12 δ of (4H) are attributed to the aromatic protons of phenylene rings whereas signal at 7.92, 7.87 and 7.5 δ(6H) are assigned to naphthalene proton. The NMR signal appears at 3.51 δ singlet attributed to methylene (2H) of $-CH_2COOH$ group attached to aromatic ring.

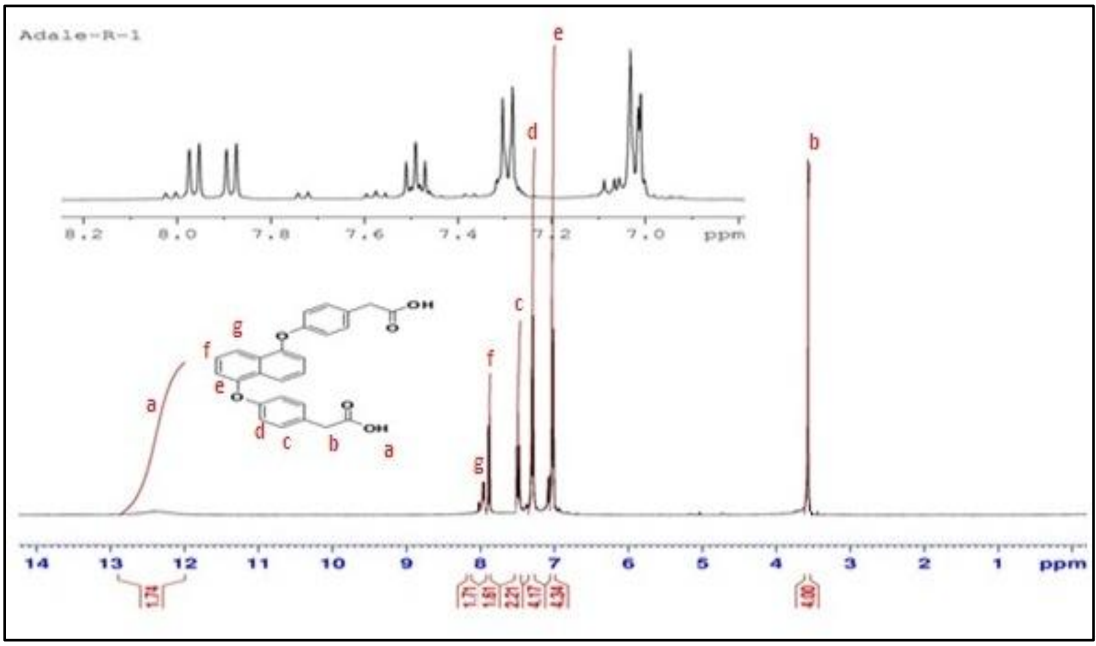


Fig 4:^{1}H-NMR of4,4′- (1,5-Naphthalenedioxy)dibenzoicacid(III)

^{13}C NMR spectrum (Fig. 5) of 4,4′- (1,5-Naphthalenedioxy)dibenzoicacid(III)showed 11 NMR signals corresponding eleven types of different carbons of which carbonyl carbon appeared at 173 δ for (C=O); whereas quaternary carbons showed signals at 156, 153, 130 and 128 δ. The CH carbons appeared at 131, 126; 118 117; 114 δ, whereas CH_2 carbon gave NMR signals at 40 δ confirms the formation of methyl substituted4,4′- (1,5-Naphthalenedioxy)dibenzoicacid(III) monomer.

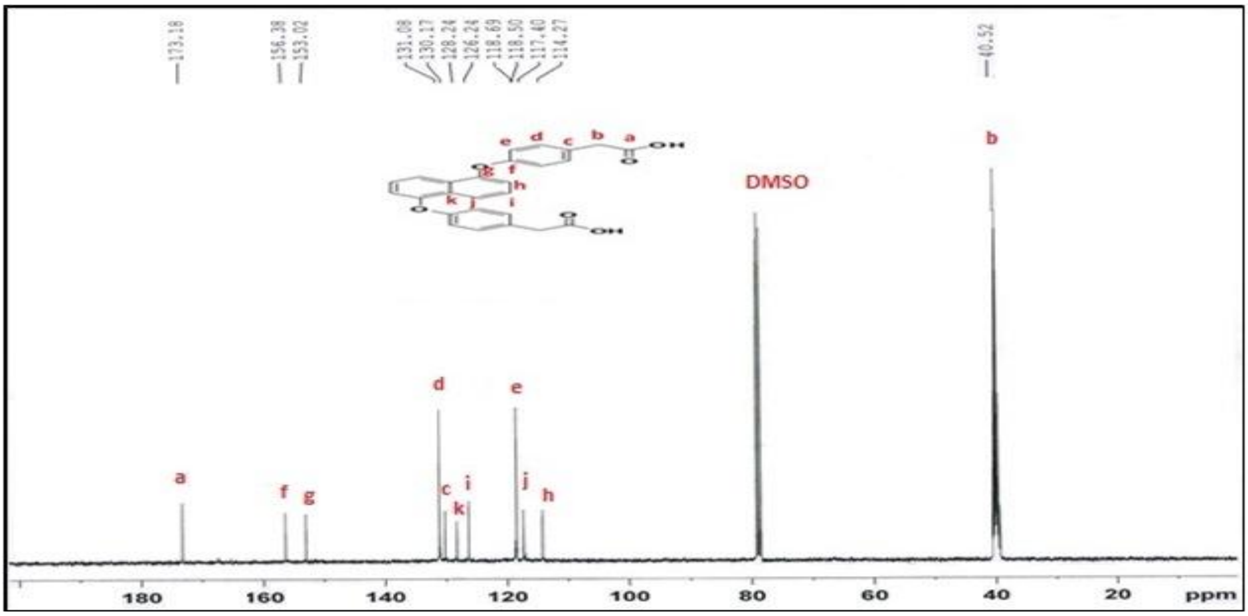


Fig 5:^{13}C-NMR of4,4′- (1,5-Naphthalenedioxy)dibenzoicacid(III)

The DEPT spectrum (Fig. 6) of 4, 4’- (1,5-Naphthalenedioxy)dibenzoicacid(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 at 131, 126; 118 117; 114 δ, and CH_2 appeared at down side at 40 δ.

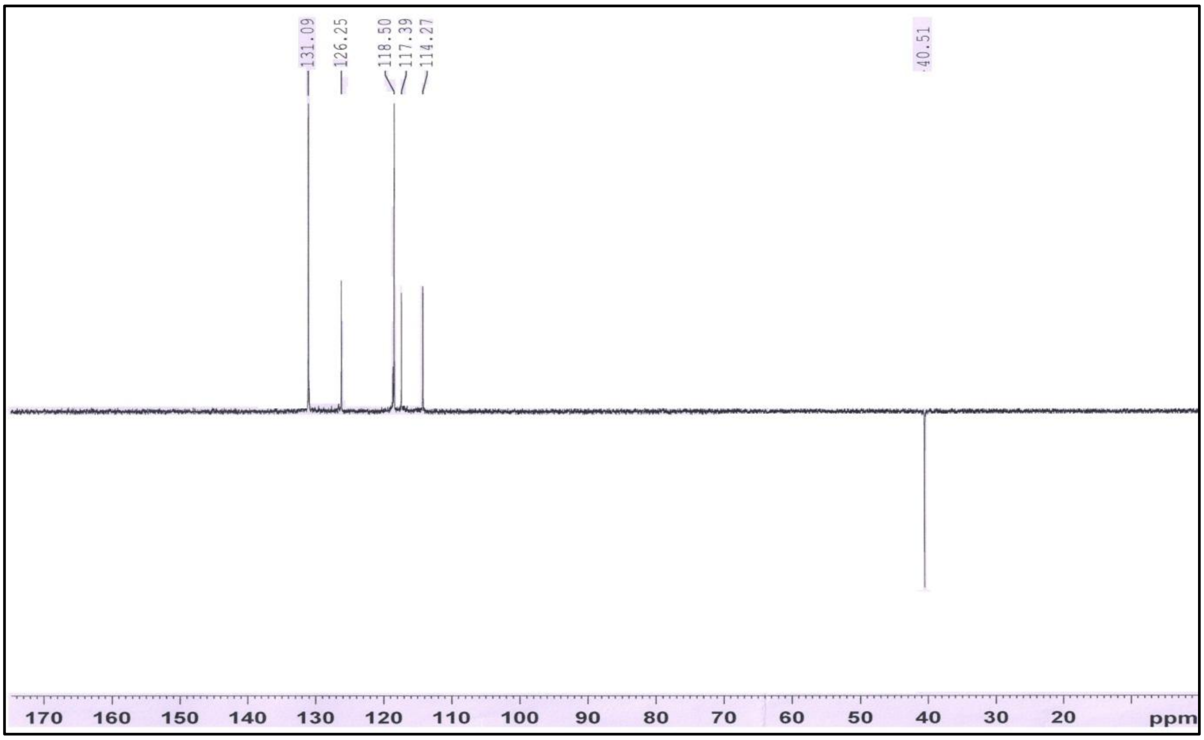


Fig 6:DEPT spectrum of 4,4′- (1,5-Naphthalenedioxy)dibenzoicacid(III)

The mass spectrum of (Fig. 7) 4,4′- (1,5-Naphthalenedioxy)dibenzoicacid(III)showed molecular ion peak at m/e (m+1) 428 corresponding to molecular weight of 1, 5--naphtholdiacetic acids (III)

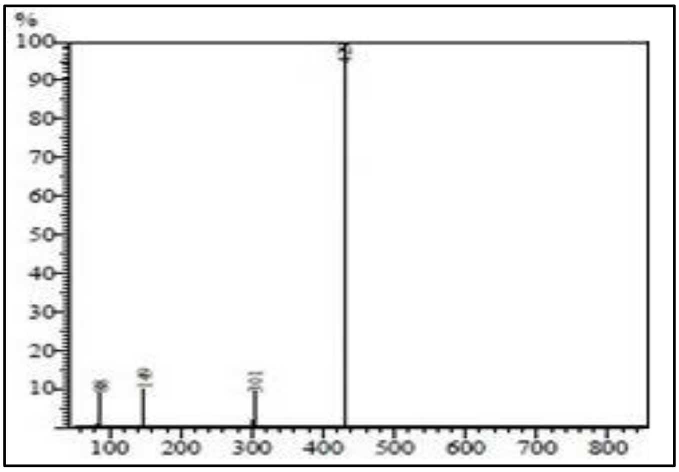
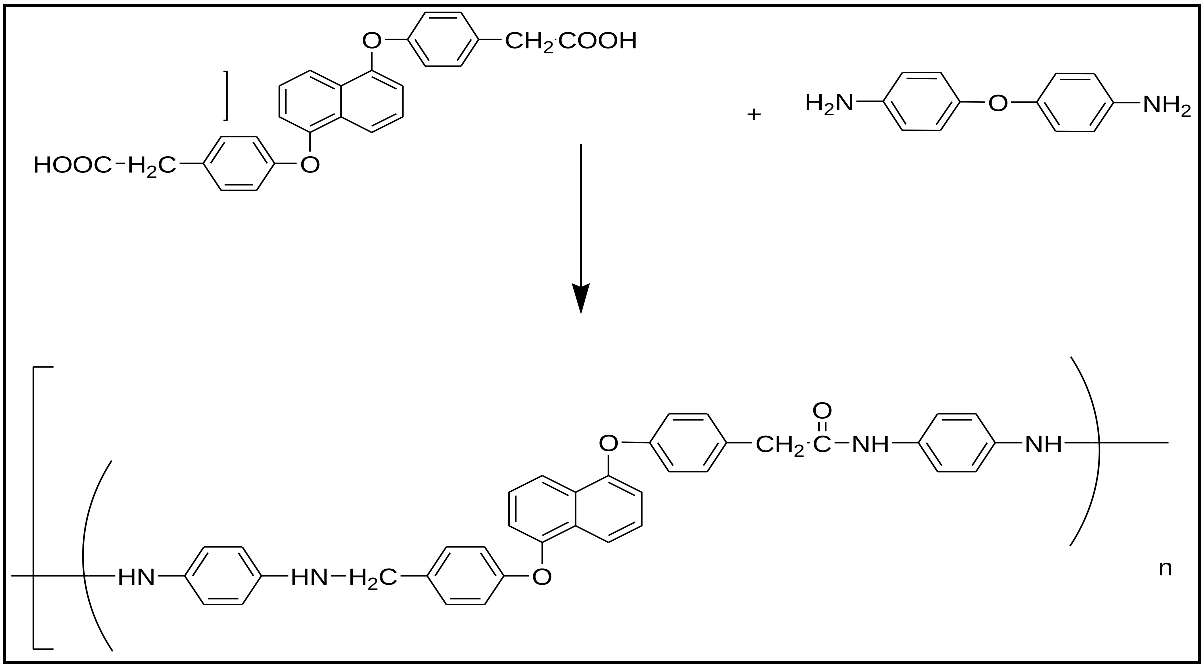


Fig 7: Mass spectrum of4,4′- (1,5-Naphthalenedioxy)dibenzoicacid(III)

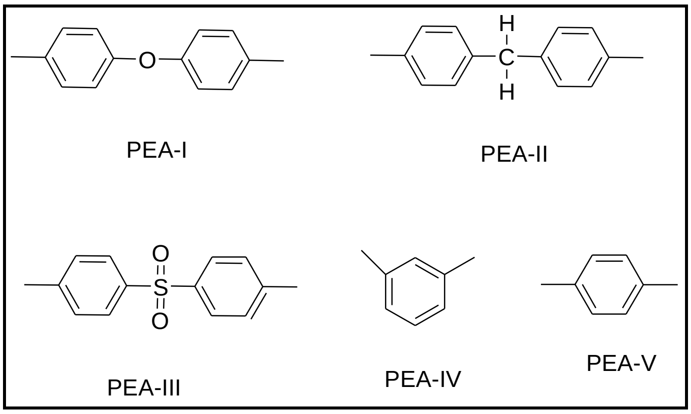
3.2 Polymerization

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



PEA



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

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 0.35 to 0.81 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**	**0.41**
PEA II	**MDA**	**0.52**
PEA III	**SDA**	**0.35**
PEA IV	**M-BDA**	**0.68**
PEA IV	**P-BDA**	**0.81**

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.

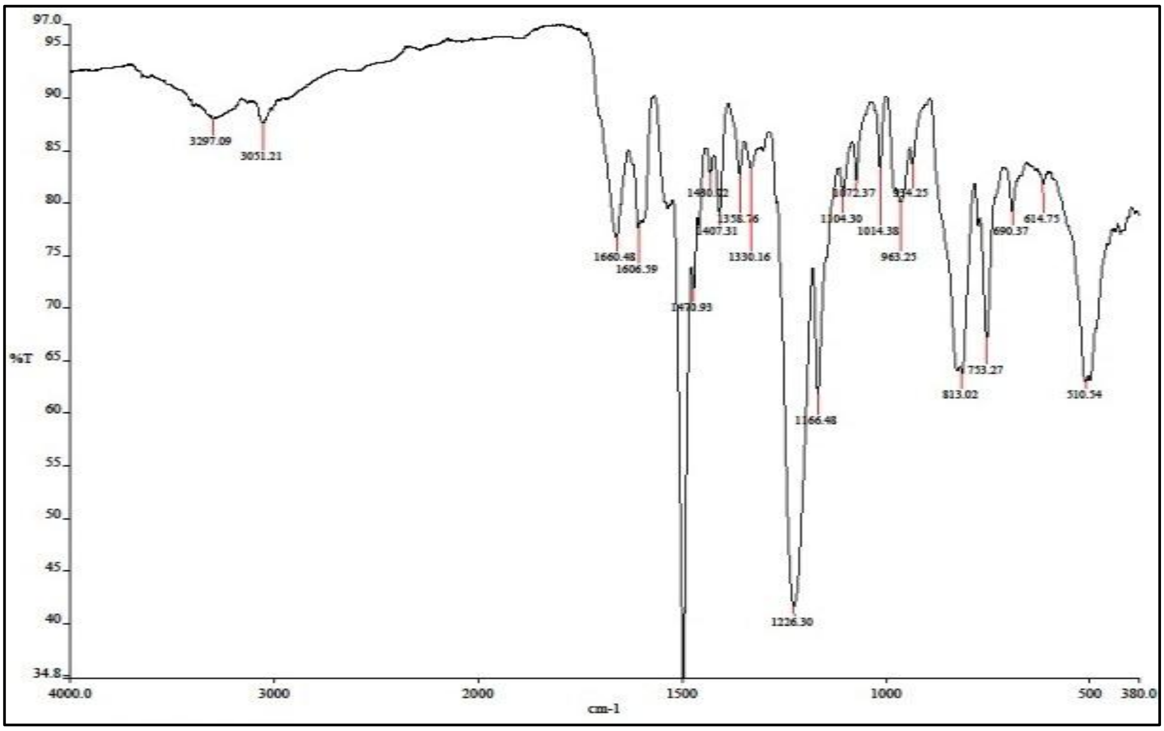


Fig 8: FT-IR spectrumof PEA-I

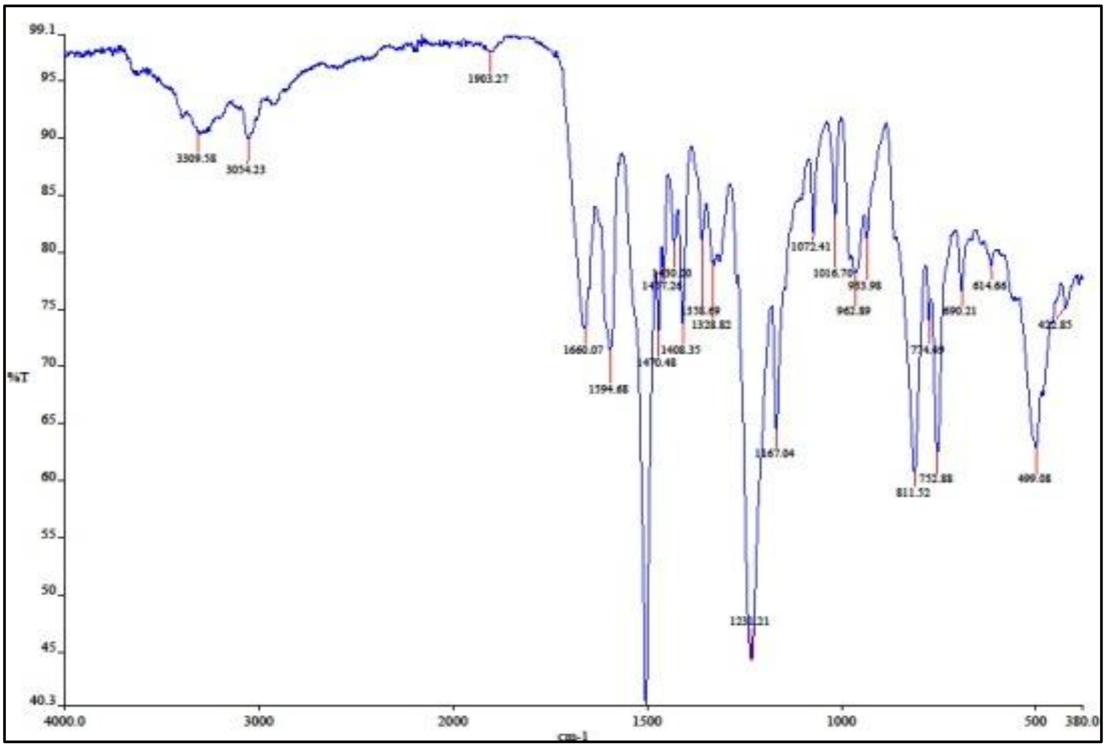


Fig 9: FT-IR spectrum of PEA-II

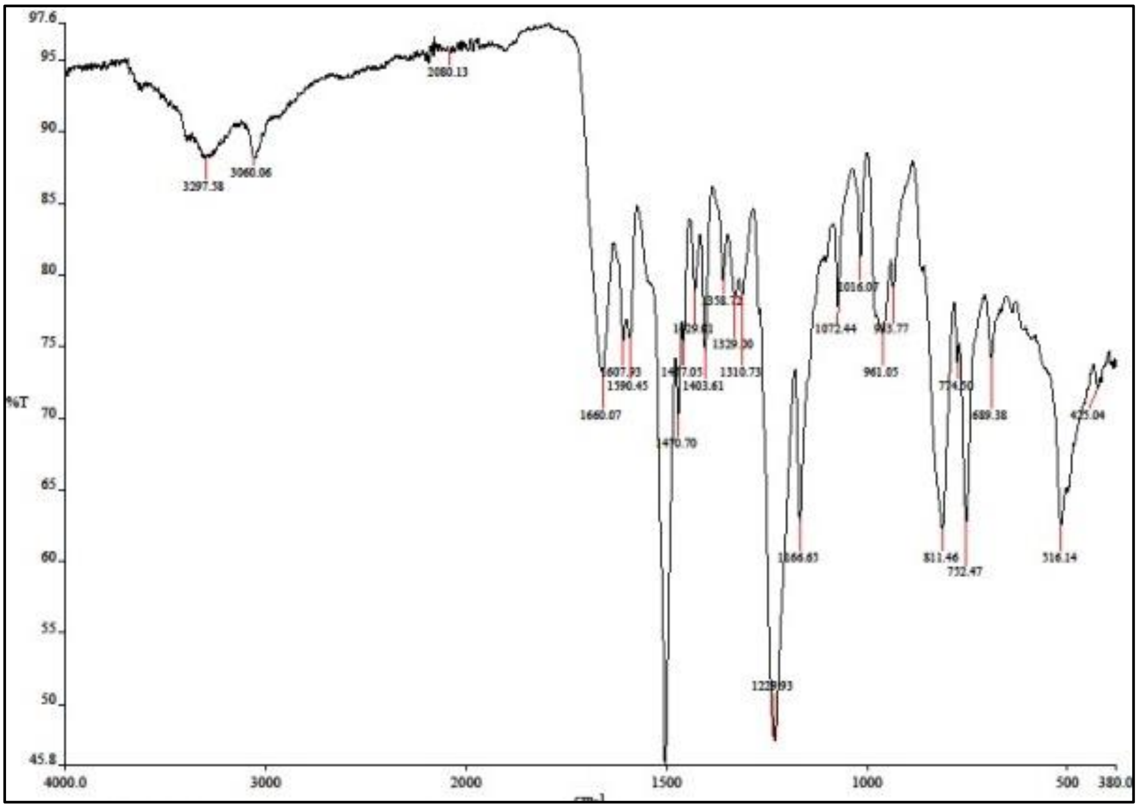


Fig 10: FT-IR spectrum of PEA-V

Table II shows the solubility behavior of polyetheramides .all polyetheramide are mostly soluble in polar aprotic organic solvent such as DMAc, DMSO, DMF. All polymer are soluble in THF after heating and polymer I,II Soluble at room temperature , polymer III, IV, V insoluble in DCM. Polymer II ,IV, V is partially soluble in m-Cresol. Polymer I,II,III Soluble at room temperature and IV, V soluble after heating in Pyridine. partially soluble. Solubility of polyethramide was greatly improved by ether linkage and addition of naphthy group.

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 a polymer concentration of 0.05 g/mL.

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

Table 3: Physical properties of poly (ether-amide) s

Polymer Code	T_i (ºC) In **N_2**	T_d (ºC) In N_2	Rd Wt% 800ºC
PEA I	396	491	50.09
PEA II	400	498	45.58
PEA III	406	502	45.26
PEA IV	413	509	47.88
PEA V	420	516	59.26

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

Thermal behavior of polymer was evaluated by means of dynamic thermogravimetriy and differential scanning calorimetry incorporate the thermal data such as initial decommpostion 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 and char yield at 800 ºC were determined from the original thermograms. (T_i) values range in bet weens 396 to 420 ºC. T_{10} values between 491 to 516ºC, respectively. Residual wt% loss at 800 ºC was in the range of 45.26 to 59.26%.

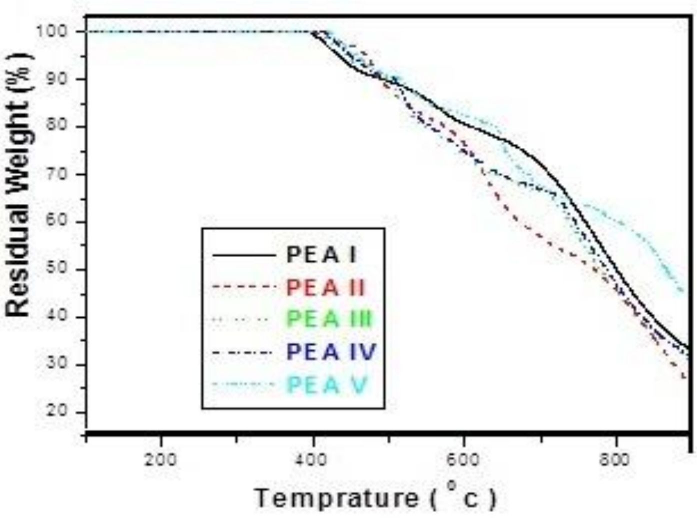


Fig 11: TGA curve of poly (ether-amide)

The wide angle X- ray diffraction pattern of all polyamides is shown in **Fig. 12.** It is observed that all the polymers exhibit good amorphous nature. Introduction of non-copolymer moiety and aliphatic methylene linkage may have disturb chain regularity and packing leading to amorphous polyamides.Thus amorphous nature of the polymers was depending on the structures of novel diacid and diamines chosen for synthesis of polymers**.**

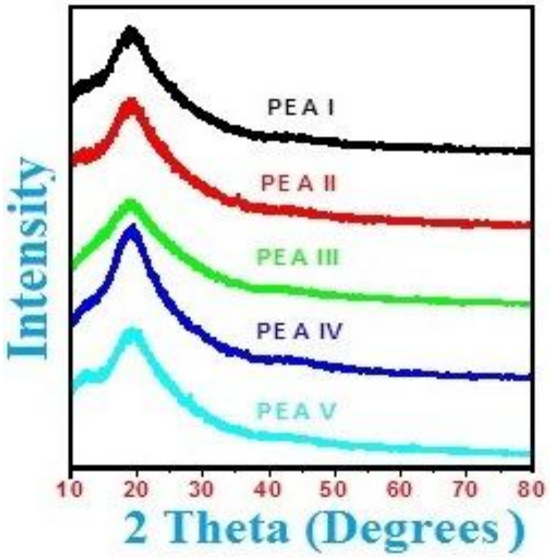


Fig12:XRD curve of poly (ether-amide)

4. Conclusion

New successfully synthesized poly (ether-amide)s containing kinked, and cranked polyamidenaphthylgroup has improved solubility &Processability of the polymers in various organic solvents. The inherent viscosities of theses polyamide were in the range from 0.35 to 0.81dL/g. These polyamide are amorphous in nature and soluble in a various organic solvent, such as DMAc,DMSO, DMF etc. All polymers are soluble in THF on heating. Solubility of polyethramide was greatly improved by insertion of methylene linkageand naphthyl group.The 10%loss of weight in nitrogen range from 441 to 461ºC. So these polyamide with improved can be used for various engineering application.

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