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Newly Synthesized Terpolymer from Pthalic Acid, P-Phenylene-Diamine and Furfuraldehyde

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Abstract: *Terpolymer was prepared by the phthalic acid (PA), Para phenylene Diamine (PD), Furfuraldehyde(F) with 2M HCl as a catalyst for polymerization with molar ratio (1:1:2). The sequence structure of terpolymer chain characterized by FTIR, 1HNMR spectroscopy. The average molecular weight of terpolymer resin was determined by non-aqueous conductometric titration method. The empirical formula and empirical formula weight of the resin were determined by elemental analysis. Thermogravimetric analysis was used for the investigation of thermal stability of the terpolymer. Additionally Freeman Carroll and Sharp Wentworth method was utilized for the calculation of activation energy. Order of reaction and free energy also from Freeman Carroll Method.*

Keywords: *Synthesis, Characterization, Molecular weight by conductometric titration method, thermal stability and activation energy of synthesized terpolymer.*

I. INTRODUCTION

Study of thermal degradation behavior and computational kinetic parameters, implementing parameters by implementing modern developing thermal degradation, kinetic techniques, the subject of interest for many investigators. The interest is fully recognized because of kinetic related with the decomposition mechanism in which thermal degradation takes place. Their mechanism allow to study the salient features of kinetic equation and kinetic study is the initiative to postulates mechanism for the thermal decomposition[1,2]. Aromatic polymers were introduced during the 1960s to match the thermal stability requirements of aerospace industry, automative industry, resistance to harsh environment, non linear optical devices. The aromatic and heterocyclic rings after conjugated rigid structure with high glass transition temperature and strong linkage, allowing good resistance in harsh environment. The automotive industry needs organic material with improved thermo-mechanical properties upto 200-220 ^{o}C. The development of new products with moderate thermos-oxidative properties could be achieved in increasing Tg of aliphatic condensation polymer with aromatic moieties. It can be achieved by polycondensation by reactive process[3,4]. Khedkar et.al[5] synthesized terpolymer resin from m-cresol, hexamine and formaldehyde in acidic medium. Senapathi et al.[6] synthesized various biologically active copolymer resins derived from resacetophenone, substituted benzoic acids, and furfural in a basic medium whereas Arora et al.[7] synthesized poly (resacetophenone]) in the presence of acidic medium. Patel et al. [8] prepared furan resins derived from o-cresol and furfural under various reaction conditions by the polycondensation technique.

Abdul Burkanudeen et. Al[9]. synthesized terpolymer from anthranilic acid salicylic acid and formaldehyde. Resin derived from salicyldehyde, ethylene diamine and formaldehyde indicates that the terpolymer has more ordered structure and involves slow decomposition reaction which was supported by the low frequency factor value Masram et.al[10]. An ecofriendly technique was adopted to synthesize a terpolymer involving antharanilic acid, thiourea and formaldehyde and the TGA data shows that the terpolymer was found to be thermally stable and the order of decomposition reaction was nearly one Azarudeen et.al[11]. Narendra Chauhan et.al[12] Synthesized, terpolymer from Vanillin, Furfural, and Halo Substituted Acetophenones. Masram et.al[13] reported the synthetic, physiochemical and kinetic study of the thermal degradation of resin derived from salicylic acid with diaminonaphthalene with formaldehyde in acid as a catalyst. Jiwatode et.al[14] synthesized the terpolymer from2,4-dihydroxyacetophenone, propylene diamine with formaldehyde

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II. MATERIALS AND METHOD

All the chemicals were used in the synthesis of terpolymer was chemically pure grade and wherever necessary the purity was tested and confirmed by thin layer chromatography. Double distilled water was used for all experiments

2.1 Apparatus

The terpolymer resin was subjected to micro analysis for C,H,N on Elementar Vivio EL III, The molecular weight of the resin measured by non-aqueous conductometric titration with the help of Elico CM 180 conductivity meter. The Infra-red spectra of the terpolymer in the region 4000-450 cm^{-1}were scanned in solid (KBr pellets) on Perkin Elmer Spectrum RX1 at Bajaj Collage of Science Civil lines, Instrumentation Wardha (M.S.) India. NMR spectra were scanned on BRUKER AVANCE II 400 NMR Spectrometer in DMSO-d_6 solvent at Sprint Testing solutions, Mumbai. The thermogravimetric analysis (TGA) terpolymer was performed on Diamond TGA thermal analyser from ambient to 900^0C Sprint Testing Solutions, Mumbai.

2.2. Synthesis of PAPDF terpolymer resin

A mixture of a Phthalic acid (0.1mol), Para phenylene diamine (0.1mol), and furfuraldehyde (0.2 mol) in a molar ratio of 1:1:2 in the 2M HCl as a catalyst has been prepared in a round bottom flask. The resultant mixture was refluxed over an oil bath at 140 ^{0}C for 6 hours with occasional shaking to ensure through mixing. As soon as the reaction period is over and cooled it for half an hour, the reaction mixture obtained was filtered. It was washed with hot water to remove unreacted monomer. The air dried terpolymer resin was extracted with ether to remove phthalic acid and furfuraldehyde copolymer, a clear solution was obtained. It was brownish colour resinous mass appeared in the solution. It was separated by filtrations and purified. The reaction is shown in (Fig.1)

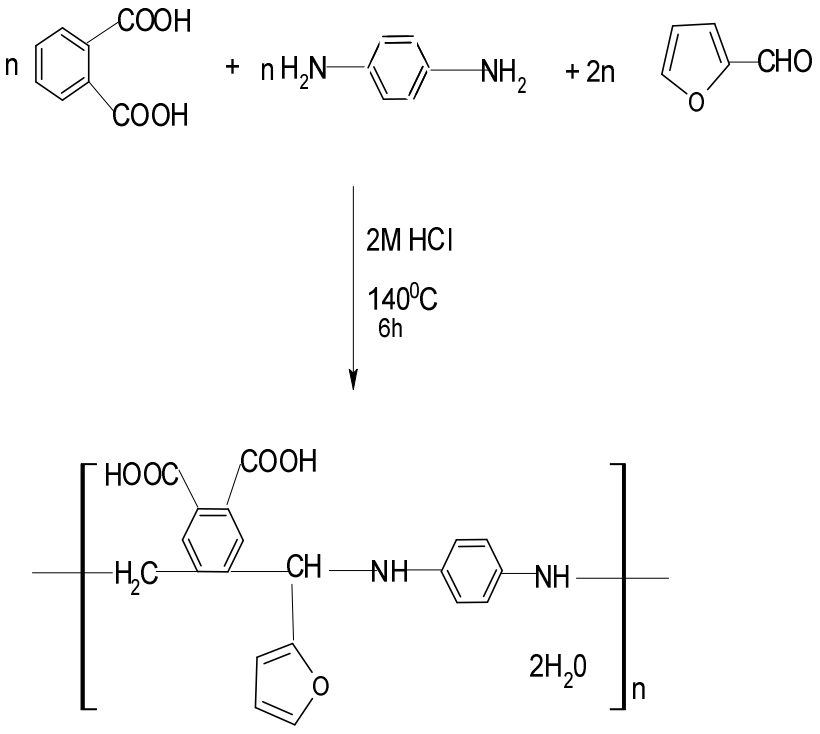

Fig.1. Reaction scheme of PAPDF

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III. RESULTS AND DISCUSSION

Resin sample was brown in color, insoluble in commonly used organic solvents, but it was soluble in DMSO and DMF.

3.1. Characterization of synthesized resin

The number average molecular weight of newly synthesized PAPDF terpolymer resin was determined by conductometric titration in non-aqueous medium using potassium hydroxide as a titrant. The specific conductance was plotted on y axis against milliequivalents of ethanolic KOH on x axis required for neutralization of 100 gm of PAPDF terpolymer. The plot of conductometric titration method in non aqueous medium have been presented in (Fig.2). Mn of these polymer was found to be 2016 which is obtained by multiplying the $(\overline{DP})$ by the formula weight of the repeating unit.

$$\overline{DP} = \frac{\text{Total milliequivalents of base required for complete neutralization}}{\text{Milliequivalents of base required for smallest interval}}$$

$\overline{Mn} = \overline{DP}$ x Repeat unit weight

Repeat unit weight was determined by elemental analysis.

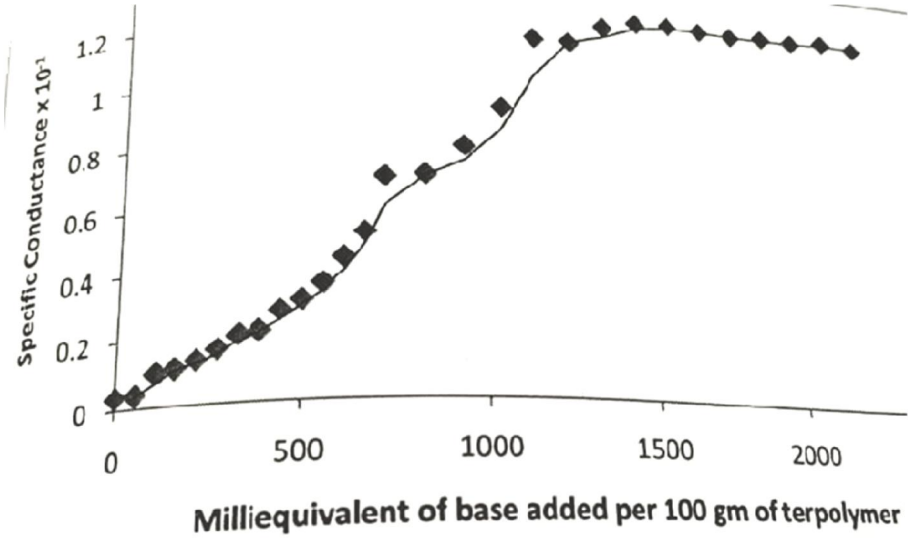

Fig.2. Conductometric titration curve of PAPDF

3.2. Spectral studies

The recorded FT-IR spectrum of the terpolymer resin PAPDF is shown in Fig.3. The strong absorption band at 3342.72 cm^{-1} indicates the presence of the COOH groups in pthalic acid (monomer) moieties. The band at 1605.22 cm^{-1} an intense band may be assigned to υ (C=O) of COOH groups. The band at 1241.18 cm^{-1} is due to bridge -CH_2 which clearly indicates formation of terpolymer resin. The tetrasubstituted in benzene ring was established by presence of the medium bands in the range of 783.43 to 1241.18 cm^{-1}. The band obtained at 1506.84 cm^{-1} may be due to -NH bending deformation.

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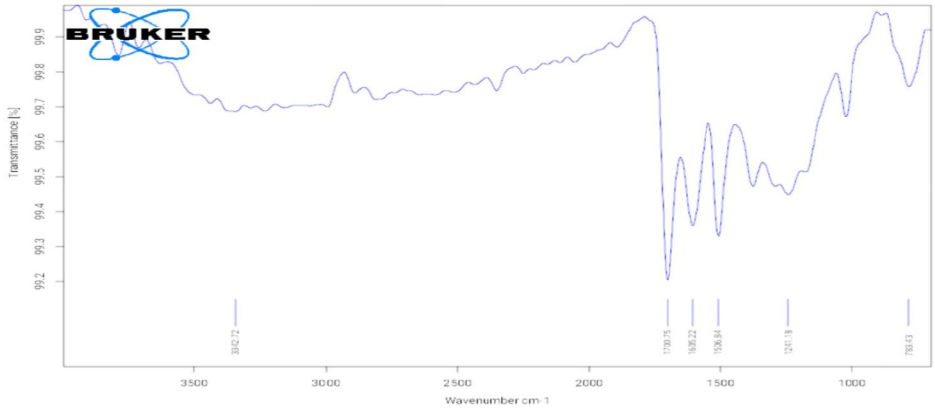

Fig.3.0. IR Spectrum of PAPDF Terpolymer

^{1}HNMR spectrum PAPDF is depicted in Fig. 3. It reveals that the signal at 2.5 (δ) ppm due to methylene proton Ar-CH_2-N linkage. The signal at 9.9 (δ) ppm is assigned to the COOH groups of pthalic acid ring. The signal at 3.5 (δ) ppm can be ascribed to -NH bridge. The multiplate signals observed in the range of 7.57 – 7.67 (δ) ppm indicates the presence of aromatic protons.

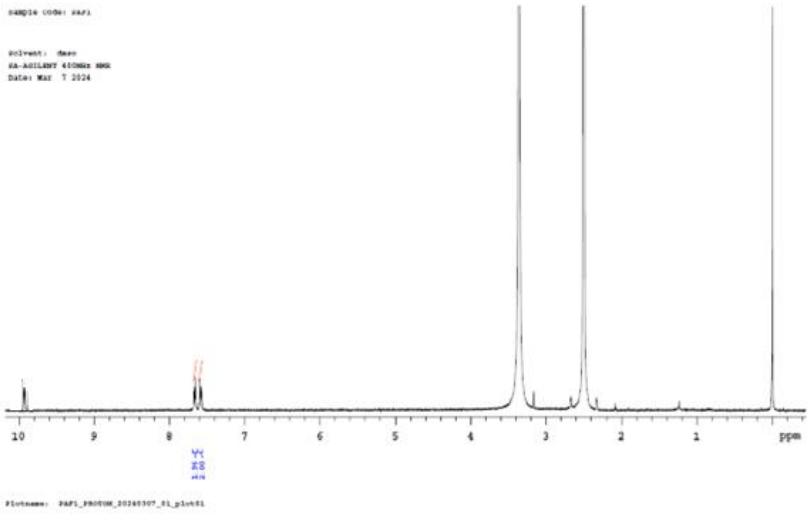

Fig.4.0. NMR Spectrum of PAPDF Terpolymer

3.3. Activation Energy By TGA Studies

Formula for calculating thermodynamic parameters using Freeman- Carroll mehod

Entropy Change (ΔS)

$$\text{Intercept} = \frac{\log kR}{h\Phi E} + \frac{\Delta S}{2.303R} \quad \text{---------- [3]}$$

Where,k = 1.3806 x 10^{-16} erg/deg/mole

R = 1.987 cal/deg/mole (8.314 J/K/Mol)

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h = 6.625 x 10^{-27} erg sec
ϕ = 0.166
ΔS = change in entropy
E = activation energy from graph

Free energy change (ΔF)

ΔF = ΔH - TDS ---------- [4]
Where, DH = Enthalpy change = Activation energy
T = Temperature in K
ΔS = Entropy change {from [5] used}

Frequency Factor (Z)

$$B_{2/3} = \frac{\log Z E_a}{R\Phi}$$ ---------- [6]

$$B_{2/3} = \log 3 + \log[1 - 3\sqrt{1-\alpha}] - \log p(x)$$ -------- [7]

Where, Z = Frequency factor
B = Calculated from equation [8]
log p(x) = Calculated from Doyle's table corresponding to activation energy.
a = degree of transformation [a= w/W_c]
w = loss in weight at time t
Wc = maximum loss in weight on the TG curve

Apparent entropy (S*)

$$S^* = 2.303 \log \frac{Zh}{kT^*}$$ ----------[9]

Where, Z = from relation [10]
T* = Temperature at which half of the compound is decomposed from it total loss.

3.4. Thermogravimetric Analysis

PAPDF terpolymer was subjected to thermogravimetric analysis and the data was used to asses the degradation pattern . Thermal degradation behavior of synthesized terpolymer has been incorporated in Table.1.and decomposition pattern is shown in Fig.5. in temperature range from R.T.to 900 ^{0}C. Decomposition pattern of terpolymer shows three stage of decomposition after loss of water molecules. The temperature range of decomposition stage for terpolymer found to be 210 ^{0}C. It is observed from TG data of terpolymer resin that the sample lost 8.8 % of its weight when temperature was raised to 210 0C,. This weight loss may be due to loss of water of crystallization associated with terpolymer resin. Second decomposition step starts rom 210-290 ^{0}C which represents the loss of side chain attached to aromatic nucleus (13.99 % found and 14.04 % calculated),]. The third decomposition starts from 290-630 ^{0}C, corresponds to loss of 2COOH groups and $C_6H_6N_2$ units(53.36 % found and 54.27 % calculated). Fourth decomposition step shows the loss of side chain (furan ring and partial degradation of aromatic nucleus (93.72 % found and 94.21% calculated).

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Table 1: Results of Thermogravimetric Analysis of PAPDF Terpolymer Resin

Terpolymer	Temperature Range (^{0}C)	Stage of Decomposition (DTA Peak)	Species degraded	% mass loss	
				Found	Calc.
PAPDF	R.T. – 210 ^{0}C	First	Loss of $2H_2O$ molecule	8.8 %	
PAPDF	210 – 290 ^{0}C	Second	Loss of side chain attached to aromatic nucleus	13.99 %	14.04 %
PAPDF	290 – 630 ^{0}C	Third	Loss of 2COOH groups and $C_6H_6N_2$ units	54.36 %	54.27 %
PAPDF	630 – 830 ^{0}C	Fourth	Loss of side chain (Furan ring and partial degradation of aromatic nucleus)	93.72 %	94.21 %

Table.2 :Thermo-analytical Data And Decomposition Temperature For PAPDF Terpolymer

Terpolymer	Decompo. Temp T (^{0}C)	Half Decomp Temp (^{0}C)	Activation Energy kJ/mole		Kinetic Parameters by FC				
			FC	SW	ΔS (J)	ΔF (kJ)	Z (S^{-1})	S* (J)	n
PAPDF	210^{0}C	600	24.28	24.27	-153.23	56.33	48.24	-33.06	0.95

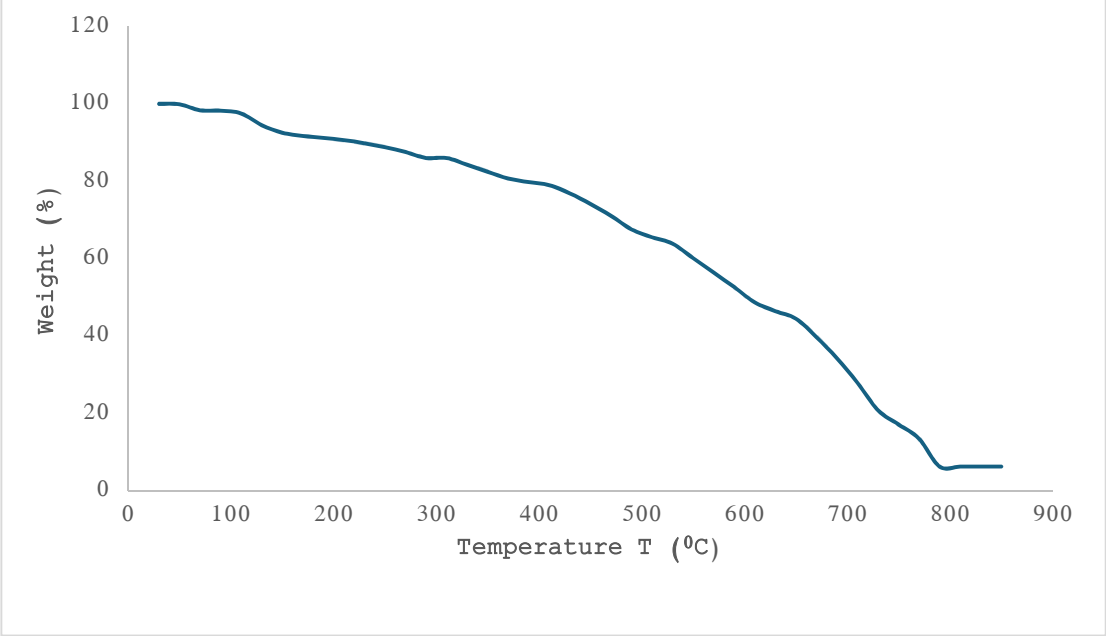

Fig. 5.0 : PAPDF (TGA THERMOGRAM)

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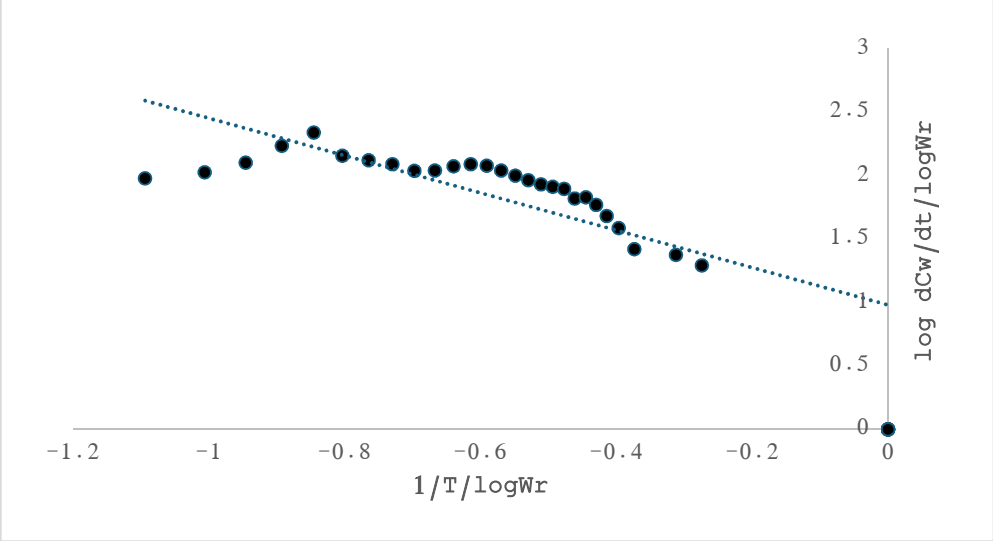

Fig 6.0 : Thermal Activation Energy Plot of PAPDF Terpolymer Resin

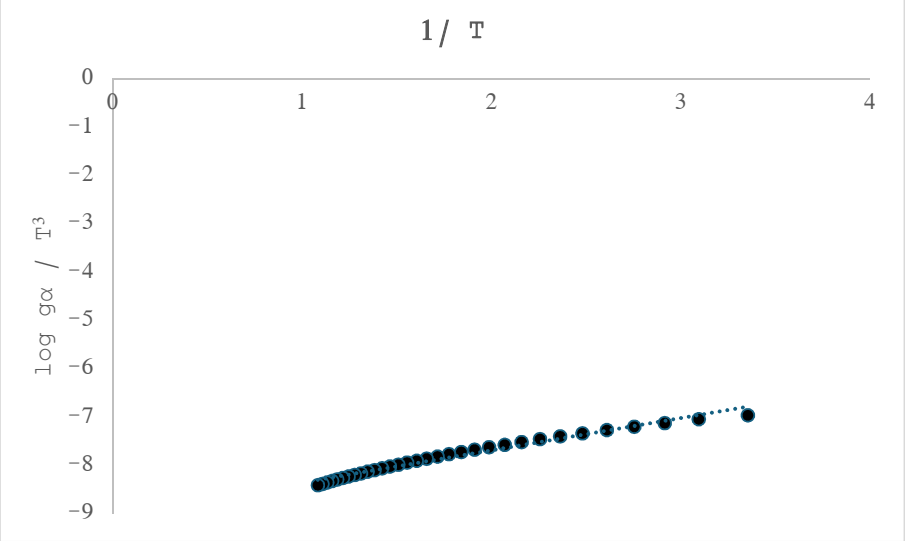

Fig. 7.0 : Freeman-Carroll plot of PAPDF Terpolymer Resin

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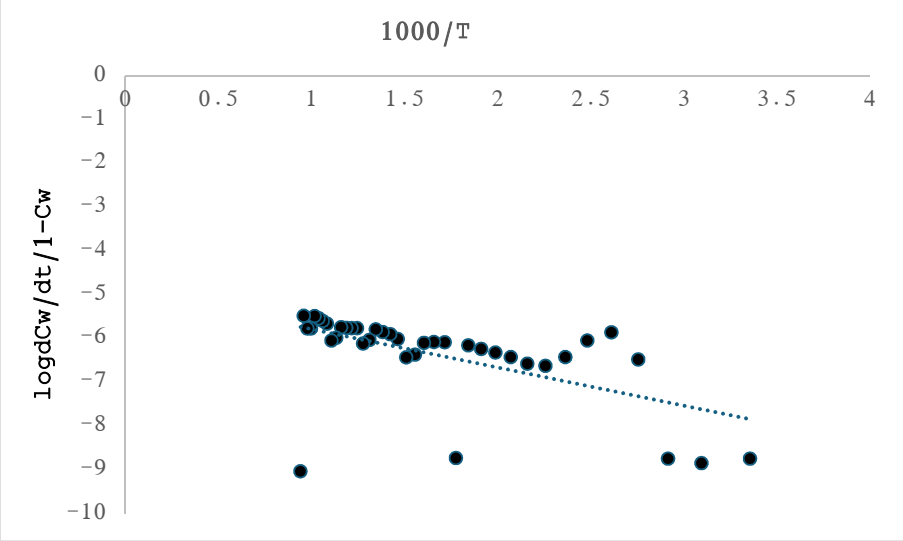

Fig. 8.0 : Sharp-Wentworth plot of PAPDF Terpolymer Resin

3.4.3.Kinetics of thermal decomposition by the Freeman-Carroll Method and Sharp Wentworth Method.

The observed thermal stability of terpolymer may be due to the stronger intermolecular hydrogen bonding present in polymer which may be attributed due to the presence of water of crystallization. From the thermal decomposition data thermal activation energy and thermogravimetric parameters of terpolymer have been calculated. These values are incorporated in [Table.1.] The activation energy calculated by using Sharp-Wentworth [Fig.6] and Freeman Carroll method[Fig.7] are in good agreement with each other [Table.2.]. From the data of [Table 2.], it can be observed that the negative value of entropy indicates that the activated polymer has more order structure than reactants, abnormal low value of frequency factors indicates the decomposition of terpolymer can be classified as slow reaction. Decomposition of terpolymer resin is known not to obey first order kinetics perfectly.

IV. CONCLUSION

Synthesized terpolymer resin PAPDF has been confirmed by ^{1}HNMR, IR-spectral studies. The values of kinetic parameter calculated from the Freeman - Carroll methods are in good agreement with the values obtained from the Sharp Went worth method. Thermogram of targeted terpolymer shows four degradation Steps.

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