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RESEARCH ARTICLE

Synthesis, Characterization and Thermal Behaviour Studies of Terpolymer Resin Derived From 8-Hydroxyquinoline-5-Sulphonic Acid and Anthranilic Acid

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Abstract: *Background*: Terpolymer have versatile applications in adhesives, electroluminescent devices, packaging, coating materials, ion exchanger and hardener for epoxy resin.

***Objective*:** In the present investigation, our main aim was to synthesize terpolymer using 8-hydroxyquinoline-5-sulphonic acid, anthranilic acid and formaldehyde (8-HQ-5-SAAF) *via* the polycondensation method and to evaluate thermal properties of terpolymer.

***Materials and Methods*:** 8-Hydroxyquinoline-5-sulphonic acid, anthranilic acid and formaldehyde were used for synthesis. Terpolymer was obtained by employing the polycondensation method.

***Results*:** TGA analysis shows that the synthesized terpolymer resin was thermally stable. The activation energy calculated by Freeman-Carroll and Sharp-Wentworth methods has been found to be in good agreement with each other. The low value of frequency factor and negative entropy value indicate that the thermal decomposition would be a slow reaction.

***Conclusion*:** The thermal energy of activation calculated by using Sharp-Wentworth and Freeman-Carroll methods has been found to be nearly identical. The terpolymer 8-HQ-5-SAAF starts degradation at high temperature, showing that this polymer resin is thermally stable.

Keywords: 8-hydroxyquinoline-5-sulphonic acid, anthranilic acid, morphology, spectral study, terpolymer, thermal analysis.

1. INTRODUCTION

Copolymers are high molecular weight compounds made up of small repeating units, which are held together by strong covalent bonds. They find versatile applications like packaging, adhesives, coating materials, electroluminescent devices, hardener for epoxy resin, ion exchangers [1-3]. For several decades, the synthesis of polymer-metal complexes has attracted great attention due to their engrossing properties such as high thermal ability and antimicrobial actions [4, 5]. Terpolymer is a polymer derived from two (or more) monomeric species, as opposed to homopolymers where only one monomer is used. Thermal degradation study of terpolymer has become a subject of recent interest, which primarily adopts thermal stability and process capability. A large number of thermally stable polymers have been prepared and their thermo gravimetric property studied, which showed many applications such as semiconductors [6], high dielectric constant for energy-storage capacitors [7].

To blend polymers having plentiful practical applications, there is a need to examine the effect of temperature on the polymers in order to establish their thermal stability. The degradation of polymer under air or inert atmosphere at increasing temperatures delivers valuable data about the types of compounds formed [8]. Bobde *et al*. prepared a terpolymer by condensation of the monomers 2, 2'-biphenol, ethylenediamine and formaldehyde in 1:1:2 molar proportions in the presence of 2M HCl as a catalyst. Activation energy and thermal stability were calculated by Sharp-Wentworth and Freeman-Carroll method [9]. Kukade and co-workers synthesized terpolymer resin by green synthesis approach, condensing thymol and guanidine hydrochloride with formaldehyde [10]. Thermoanalytic and kinetics study of terpolymer resin prepared from P-Cresol-Oxamide-Formaldehyde has also been conducted [11]. Sorption studies of heavy metal ions by salicylic acid, formaldehyde and catechol have also been carried out [12]. Synthesis of terpolymer

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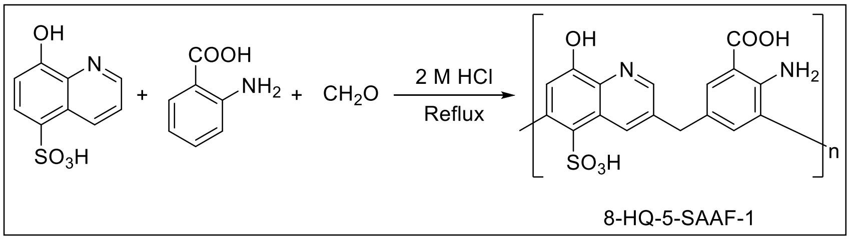

Fig. (1). Synthesis sequence of 8-hydroxyquinoline-5-sulfonic acid-formaldehyde terpolymer resin.

resin from resorcinol, thiourea and formaldehyde was conducted by Karunakaran *et al* for thermal and chelate ion-exchange properties of terpolymer for Fe^{3+}, Co^{2+}, Cu^{2+}, Pd^{2+}, Zn^{2+} and Cd^{2+} ions [13]. Singru *et al.* reported thermal, electrical, and photoluminescence properties of terpolymer resin derived from p-Cresol and melamine with formaldehyde [14]. Terpolymer resin synthesized from anthranilic acid, salicylic acid and formaldehyde suggested that terpolymer resin was thermally stable and low-frequency factor values indicated slow decomposition reaction [15]. Masram and colleagues prepared terpolymer resin from salicylaldehyde, ethylene diamine and formaldehyde and showed that the terpolymer had a more ordered structure and involved slow decomposition reaction which was supported by the low frequency factor values [16]. DSC and TGA were used to evaluate the thermal properties and degradation parameters of terpolymer [17]. The terpolymer resin obtained from 8-hydroxyquinoline, anthranilic acid with formaldehyde and prepared its chelates with some transition metals. The thermal stability and kinetic parameters were calculated. The results show that polychelates possess higher thermal stability, low activation energy than the corresponding terpolymer ligand [18]. The terpolymer resin derived from o-toludine, semicarbazide, formaldehyde and its composite with activated charcoal has been prepared and its ion exchange and kinetic parameters were evaluated [19]. The non-isothermal decomposition of terpolymer resin obtained from 2, 4-dihydroxypropiophenone, 1, 5-diaminonapthalene-formaldehyde has been studied by Gurnule *et al.* [20]. Several terpolymers were synthesized by condensation of 4-hydroxybenzaldehyde oxime, formaldehyde and chloro, bromo, methoxy or methyl substituted acetophenones and their thermal stability was investigated by using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) [21]. The thermal degradation parameters of terpolymer resin synthesized from p-hydroxybenzoic acid-semicarbazide and formaldehyde were determined by Freeman-Carroll and Sharp-Wentworth's method. The results reveal that terpolymers were thermally stable and the decomposition reaction obeyed first order kinetics [22].

The present paper describes the synthesis and characterization of terpolymer resins obtained from 8-hydroxyquinoline-5-sulphonic acid and anthranilic acid with formaldehyde. From thermogram, the thermal energy of activation and kinetic parameters such as apparent energy, frequency factor, free energy change, and entropy change were evaluated by using Sharp-Wentworth and Freeman-Carroll methods.

2. MATERIALS AND METHODS

2.1. Materials

Entire synthetic chemicals purchased from the Central Scientific Company Nagpur were analytically and chemically pure. 8-Hydroxyquinoline-5-sulfonic acid (Sigma Aldrich, India), anthranilic acid (SRL Chemicals, Mumbai, India) and formaldehyde (S.D. Fine Chemicals) were used as received.

2.2. Synthesis of the Terpolymer Resin

2.2.1. Condensation of 8-Hydroxyquinoline-5-Sulfonic Acid and Anthranilic Acid with Formaldehyde

The newly synthesized terpolymer 8-HQ-5-SAAF was orchestrated by gathering 8-hydroxyquinoline-5-sulfonic acid, anthranilic acid and formaldehyde within the sight of 2 molar HCl as a catalyst in 1:1:2 molar extent of monomers at a temperature of 125°C in an oil bath for around 5 hrs. After completion of the reaction, dark brown colour solid compound was obtained. The product obtained was extricated with diethyl ether to expel 8-hydroxyquinoline-5-sulfonic acid, formaldehyde terpolymer which may be available as 8-HQ-5-SAAF terpolymer. It was then additionally cleansed by dissolving in 8% NaOH and then separated. Afterward, the terpolymers were reprecipitated by drop savvy expansion of 1:1 (v/v) conc. HCl and water with consistent and quick blending to maintain a strategic distance from the lumping formation. The filtrate was dried, powdered and kept in a vacuum desiccator over silica gel. The reaction sequence has been delineated in Fig. (**1**).

3. CHARACTERIZATION

3.1. Analytical and Physicochemical Studies

The elemental analysis was carried out by using Perkin Elmer 789N QP-2010 instrument. The UV-Visible analysis was obtained using UV-Visible Absorption Spectrometer Shimadzu, Japan. Shimadzu RL -200N spectrometer in KBr pallets with the wavenumber region of 4000-400 cm^{-1} used for scanning FTIR spectrum of the terpolymer resin. The proton NMR spectrua were recorded in DMSO-d6 solvent using Bruker 400 MHz spectrometer. All the analytical and spectral studies for the newly synthesized terpolymer were carried out at the Indian Institute of Technology, Mumbai.

3.2. SEM Analysis

Scanning electron microscopic technique makes very useful data available about the surface characteristics of any

Table 1. Elemental analysis and empirical formula of 8-hq-5-saaf terpolymer resins.

Terpolymer Resins	% of C Observed (Cal.)	% of H Observed (Cal.)	% of N Observed (Cal.)	% of S Observed (Cal.)	Empirical Formula of Repeated Unit	Empirical Formula Weight
8-HQ-5SAA-F	54.96 (55.6)	4.03 (4.12)	6.76 (7.21)	8.06 (8.24)	$C_{18}H_{16}N_2O_6S$	388.39

material. The surface morphology was researched by a Hitachi (Model S-4800) scanning electron microscope for a terpolymer at different amplifications.

3.3. Thermogravimetric Analysis

Thermo-gravimetric analysis was researched by utilizing a thermal analyzer (DTG-60 DTA-TG Apparatus Shimadzu). The thermal stability of the terpolymer was evaluated by thermo-gravimetric analysis and differential thermal analysis. The sample was heated from RT-700°C under air at 10°C/min (Department of Chemistry R.T.M. Nagpur University, Nagpur).

4. RESULTS AND DISCUSSION

The newly blended terpolymer was found to be yellow in colour. The terpolymer is solvable in solvents like DMSO, DMF and THF while unsolvable in common other organic solvents. The melting point of 8-HQ-5-SAAF terpolymer resin is 190°C and the yield of terpolymer resin has been found to be 80%.

4.1. Elemental Analysis

The terpolymer 8-HQ-5-SAAF has been analyzed for carbon, hydrogen, nitrogen and sulphur content. Calculated and observed C, H, N & S elements are given in Table **1**. On the basis of the results, the empirical formula and the empirical weight of the single repeating unit of the terpolymer were found to be $C_{18}H_{16}N_2O_6S$ and 388.39 respectively, which is in good agreement with the calculated values.

4.2. FT-IR Spectra

The FTIR spectrum of the 8-HQ-5-SAAF terpolymer is depicted in Fig. (**2**). A sharp band appearing at 3409-3501cm^{-1} may be attributed to the stretching vibrations of phenolic hydroxyl (-OH) groups of -COOH present in the aromatic ring displaying intramolecular hydrogen holding [23]. A peak at around 1271 cm^{-1} may be allocated to C-N stretching the Ar-NH_2 group [24]. The 1,2,3,5 tetra substitution of the aromatic benzene ring is identified from the bands appearing in the region 1200-800 cm^{-1}. The band at 1620 cm^{-1} is due to -C=O stretching vibrations [25]. The band displayed at 1190-1228 cm^{-1} suggests the presence of a methylene bridge (-CH_2) bridge [26]. A sharp and weak band appearing at 1271-1314 cm^{-1}attributed to Ar-CH_2-Ar bridge in the polymer.

4.3. ^{1}H NMR Spectra

The ^{1}H NMR Spectral analysis of 8-HQ 5-SAAF is displayed in Fig. (**3**), while the NMR spectrum of 8-HQ 5-SAAF is recorded in DMSO-d_6 solvent. The powerful signal at 9.1 ppm is ascribed to the -OH of the quinoline ring. A singlet appearing at 8.6 ppm assigned to the -OH of Ar-COOH exhibiting intramolecular hydrogen with the proton of -NH_2 of Ar-NH_2. The multiple peaks obtained in between the region 7.3-8.3 ppm, are the expected protons of aromatic ring [27]. An intense signal appeared at 2.4 ppm due to the methylene proton of Ar-CH_2-Ar bridge [28]. The sharp singlet appearing at 9.9 ppm is attributed to the -SO_3H group [29].

4.4. Scanning Electron Microscopy (SEM)

The surface morphology of 8-HQ-5-SAAF terpolymer resin has been established by scanning electron microscopy. The photograph of the 8-HQ-5-SAAF terpolymer shows a spherulites structure with a profound increase. The crystals are minor in the superficial zone being less closely packed with high sponginess. Also, the spherulites morphology of resin exhibits a crystalline structure of resin having deep corrugation, which is clearly visible in SEM images of resin.

The above evidences indicate the resin, more or less showing an amorphous character with less closely packed surface having deep pits [30]. Thus, the terpolymer exhibits amorphous nature and shows higher trade ability for metal ions. SEM analysis shows that the 8-HQ-5-SAAF terpolymer resin is crystalline, in addition to being shapeless or be crystalline and indistinct, as shown in Fig. (**4**).

4.5. Thermogravimetric Analysis

The thermogravimetric technique has been found to be the most advantageous and operative method to determine the thermal degradation pattern of a terpolymer. The thermogram is obtained by heating the resin sample in air at 10°C/min. The thermogram 8-HQ-5-SAAF is presented in Fig. (**5**) with the terpolymer exhibiting four degradation steps. The first step decay was slow and began from a temperature range of 40-140°C due to mass loss (4.21% found and 4.63% calculated) which highlighted loss of one crystalline water molecule. The terpolymer eliminated 39.43% of its weight in the second step, corresponding to -SO_3H,-OH,-COOH and -NH_2 groups. (39.9% found and 39.43% calculated). The third step began from 250°C to 480°C, corresponding to a mass reduction (73.1% found and 72.93% determined), which highlighted the corruption of aromatic nucleus and methylene bridges. The fourth step of debasement started at 480°C and ended at 700°C due to complete reduction of the whole molecule.

4.5.1. Sharp-Wentworth Method

In this method following equationis used derived by Sharpand Wentworth,

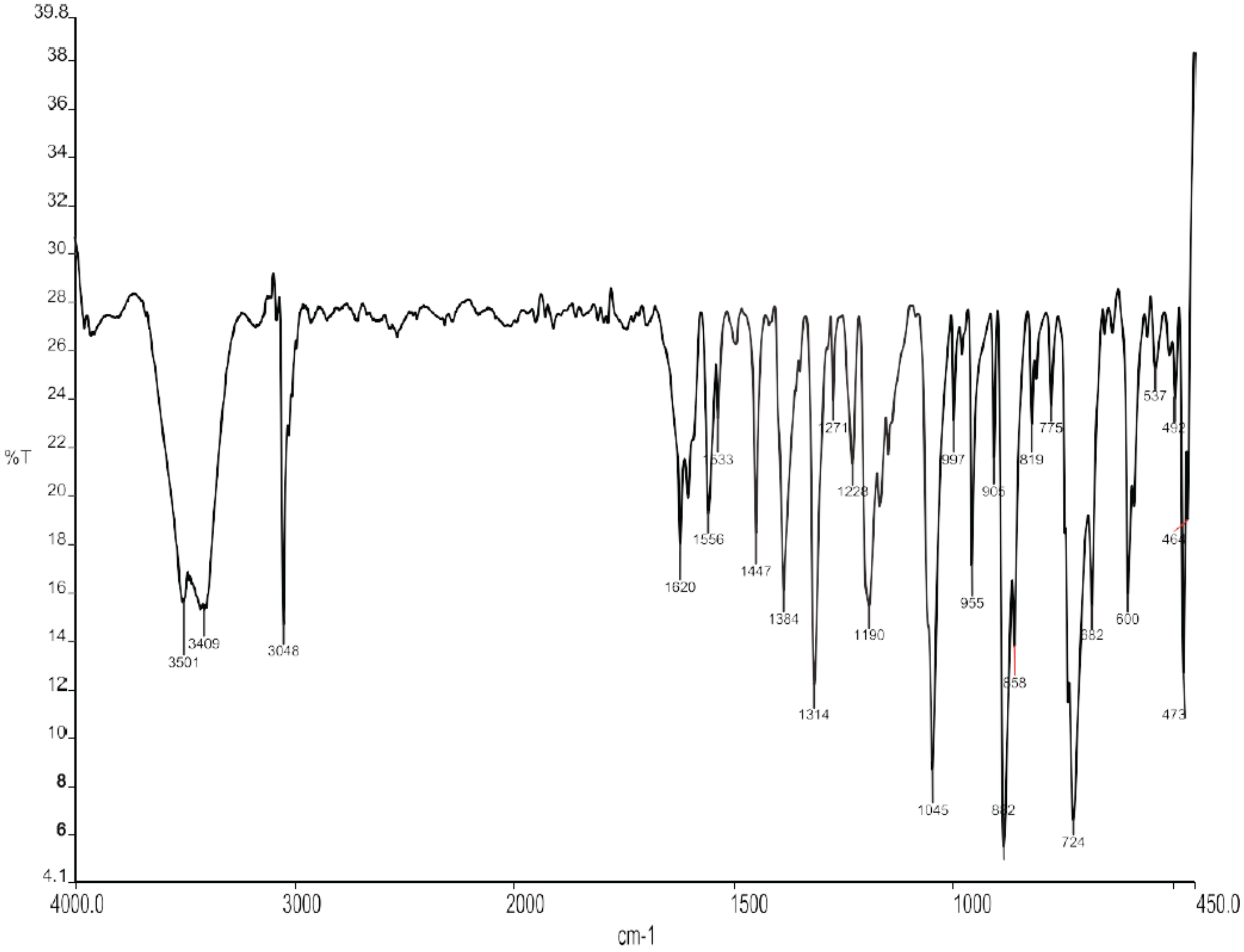

Fig. (2). Infrared spectra of 8-HQ-5-SAAF Terpolymer.

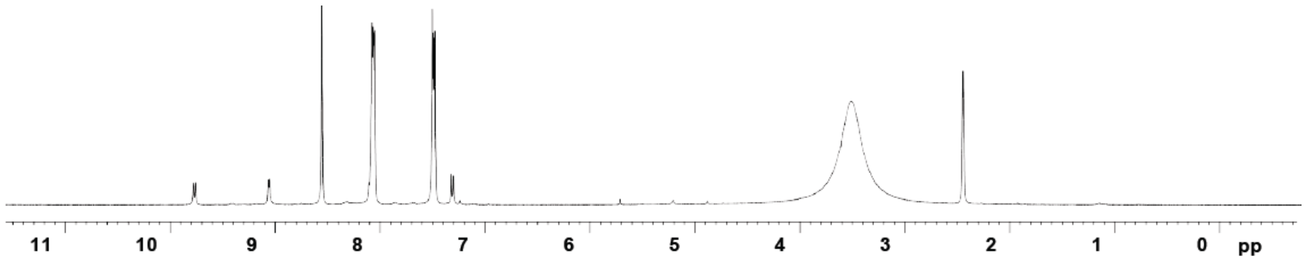

Fig. (3). H^1 NMR Spectrum of 8-HQ-5-SAAF terpolymer.

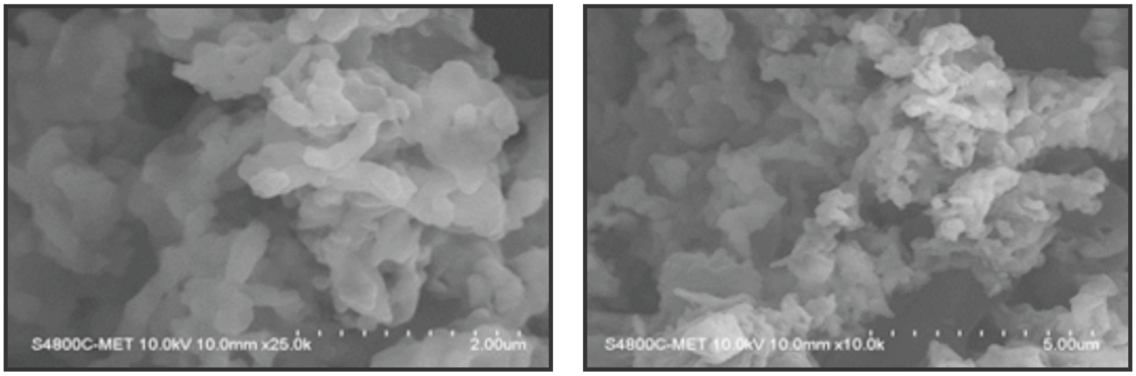

Fig. (4) contd…

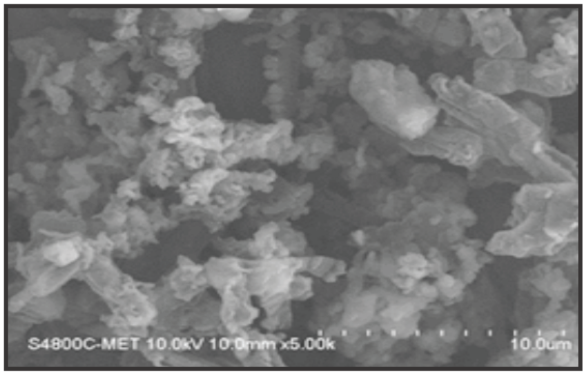

Fig. (4). SEM Images of 8-HQ-5-SAAF terpolymer. (*A higher resolution / colour version of this figure is available in the electronic copy of the article*).

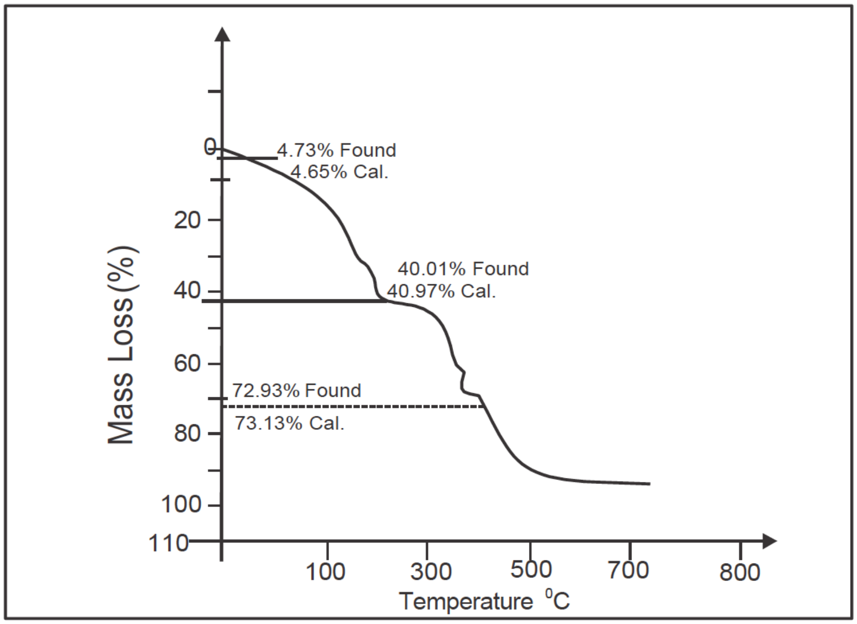

Fig. (5). Thermogravimetric Curve of 8-HQ-5-SAAF tempolymer.

$$\log[(dc/dT)/(1-c)]=\log(A/\beta)-[Ea/2.303R].1/T \qquad (1)$$

where,

dc/dT=rate of change off raction of weight withch angeint emperature

β=linear heating rated T/dt.

The graph between (logdc/dt)/(1-c)vs1/T obtained gives the straight line from which energy of activation (Ea) is calculated from its slope (Fig. **6**).

where β Is the conversion attimet, Risthegas constant

T is the absolute temperature.

4.5.2. Freemann-Carroll Method

The Free man and Carroll expression is:

$$[\Delta\log(dw/dt)]/\Delta\log Wr=(-E/2.303R).\Delta(1/T)/\Delta\log Wr+n \qquad (2)$$

where, dw/dt=rate of change of mass with time.

Wr = Wc-W

Wc=weight loss at completion of reaction.

W=fraction of weight loss at time t.

Ea=energy of activation

n=order of reaction.

A graph between the terms $[\Delta\log(dw/dt)]/\Delta\log W_r V_s \Delta (1/T)/\Delta\log W_r$ were plotted which gives as traight line from the slope we obtained energy of activation (Ea) and intercepton Y-axis as order of reaction (n) Fig. (**7**).

The change inentropy (S), frequency factor (z), apparent-entropy (S*) can also be calculated by further calculations [31, 32].

By using thermogravimetric data and then employing the Sharp-Wentworth method, the thermal activation energies (Ea) were calculated and are presented in Table **2**. Which is in good agreement with the activation energy calculated by Freeman-Carroll method Fig. (**8**). Also, thermodynamic parameters such as entropy change (ΔS), apparent entropy change (S*), and frequency factor (Z) are calculated. From

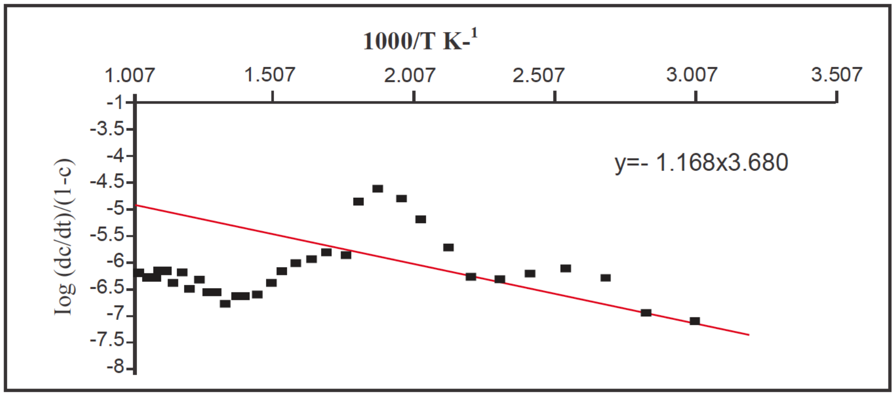

Fig. (6). Sharp - Wentworth plot of 8-HQ-5-SAAF terpolymer.

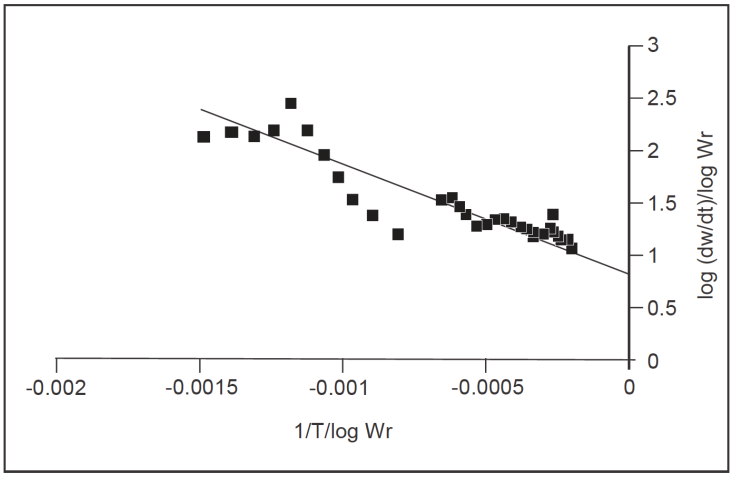

Fig. (7). Thermal activation energy plot of 8-HQ-5-SAAF terpolymer.

Table 2. Kinetic parameters of terpolymer.

Terpolymer	Activation Energy(Ea), KJ mol-1		Entropy Change (ΔS), (J)	Free Energy Change (ΔF), (KJ)	Frequency Factor (Z) (sec-1)	Apparent Entropy Change (S*)	Order of Reaction (n)
	F-C	S-W					
8-HQ-5-SAAF	20.08	22.36	-224.85	126.43	548	-60.5	0.8

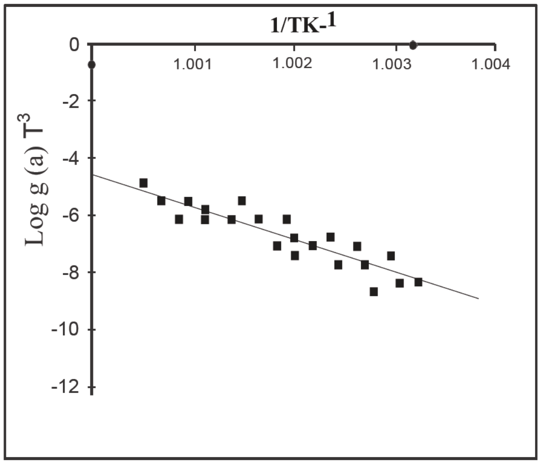

Fig. (8). Freeman - Carroll plot of 8-HQ-5-SAAF terpolymer.

the low value of frequency factor, the decomposition reaction is classified as slow, which is further supported by the negative value of the entropy change. A few points do not drop on the straight-line graphs, which points out that the reaction does not obey the first-order kinetics correctly.

Change of Entropy:

$$\text{Intercept} = [\log KR/h\phi E] + S / 2.303\, R \qquad (3)$$

where, K = 1.3806 x 10-16 erg/deg/mole, R = 1.987 Cal/deg/mole h = 6.625 x 10-27 erg sec, ϕ = 0.166

S = Change in entropy, E = Activation energy from graph.

Free Energy Change:

$$\Delta F = \Delta H - T\Delta S \qquad (4)$$

where, ΔH = Enthalpy Change = activation energy

T = Temperature in K

S = Entropy change from (i) used.

Frequency Factor:

Bn= Log ZEa / ϕR (5)

B1 = log [ln 1 /1-α] - log P (x) (6)

Where, Z = frequency factor, B = calculated from equation (6)

Log P(x) = calculated from Doyle's table corresponding to activation energy.

Apparent Entropy Change:

S* = 2.303 log Zh / KT* (7)

Z = from relation (5)

T* = temperature at which half of the compound is decayed from its total loss.

4.6. UV-Visible Spectra

The UV-Visible Spectra of 8-HQ-5-SAAF terpolymer are shown in Fig. (**9**). The UV-Visible spectra were recorded in pure DMSO in the region 190-800 nm at a checking rate of 100 nm min^{-1}. The terpolymer samples exhibit three absorption maxima in the regions 240-270 nm, 310-380 nm and at 400-450 nm. The observed positions of the absorption bands have different intensities. More intense band 240-270 nm is due to ($\pi \rightarrow \pi^*$) allowing the transition of conjugation in the aromatic benzene ring, which readily attains coplanarity and shoulder merging (loss of fine structure) and also due to chromophore groups like >C=C, >C=N, which are in conjugation with an aromatic nucleus. The less intense band at 310-380 nm may be due to ($n \rightarrow \pi^*$) transition which indicates the presence of auxochromic substituents -C=N and $-NH_2$ groups in the terpolymer. The bathochromic shift from the basic value viz. 230 nm to 310 nm may be due to the combined effect of conjugation (due to chromophore) and phenolic hydroxyl group as well as $-NH_2$ groups (auxochrome). The presence of $-NH_2$ (auxochromes) is responsible for hyperchromic shift *i.e*. Σmax higher values [33].

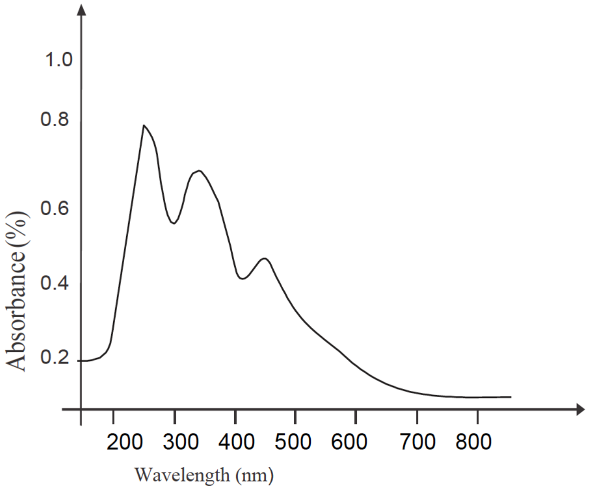

Fig. (9). UV -Visible spectra of 8-HQ-5-SAAF.

CONCLUSION

The terpolymer synthesized from starting materials 8-hydroxyquinoline-5-sulphonic acid, anthranilic acid and formaldehyde in the presence of an acid catalyst at 120°C. The structure of the terpolymer was confirmed by UV-VISIBLE, FT-IR and ^1H NMR spectral studies. From TGA curves, the thermal activation energy calculated by using Sharp-Wentworth and Freeman-Carroll methods was found to be nearly identical and for the reaction was found to be nearly one. TGA curve reveals that terpolymer had better thermal stability. The low-frequency factor and negative entropy values evaluated from Freeman-Carroll method indicate that the thermal decomposition is categorized as a slow reaction.

ETHICS APPROVAL AND CONSENT TO PARTICIPATE

This data is original and not submitted before anywhere for publication. Both the researchers have given consent to participate in publication.

HUMAN AND ANIMAL RIGHTS

No Animals/Humans were used for studies that are base of this research.

CONSENT FOR PUBLICATION

Not applicable.

AVAILABILITY OF DATA AND MATERIALS

All the data are included in the manuscript.

FUNDING

None.

CONFLICT OF INTEREST

The authors declare no conflict of interest, financial or otherwise.

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