

Research Article

Thermogravimetric and Spectroscopic Analysis of 8-Hydroxyquinoline 5-Sulphonic Acid-melamine-formaldehyde Polymer Resin-IV

Rajesh N. Singru

R.D College of Science, Aheri, Gadchiroli, Nagpur, Maharashtra 442705, India

Correspondence should be addressed to Rajesh N. Singru, rajeshsingru@in.com

Received 11 December 2011; Accepted 2 January 2012

Academic Editors: Y. Fang, E. Gallicchio, S. Hashimoto, and P. Trens

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The title terpolymer (8-HQ5-SAMF-IV) synthesized by the condensation of 8-hydroxyquinoline 5-sulphonic acid (8-HQ5-SA) and melamine (M) with formaldehyde (F) in the presence of acid catalyst and using varied 3 : 1 : 5 molar proportions of the reacting monomers. The synthesized terpolymer was characterized by different physicochemical techniques. The thermogravimetric analysis of terpolymer resin prepared in the present study has been carried out by nonisothermal thermogravimetric analysis technique in which sample is subjected to condition of continuous increase in temperature at linear rate. Thermal study of the resin was carried out to determine the mode of decomposition and thermal stability. Thermal decomposition curve was studied carefully with minute details. The Freeman-Carroll and Sharp-Wentworth methods have been used in the present investigation to calculate thermal activation energy and different kinetic parameter of the terpolymer resin. Thermal activation energy (E_a) calculated by two above mentioned methods, is in close agreement. The advantage of Freeman-Carroll method is to calculate both the order of reaction (n) and energy of activation in one single stage by keeping heating rate constant. By using data of thermogravimetric analysis, various thermogravimetric parameters like frequency factor (Z), entropy change (ΔS), free energy change (ΔF), and apparent entropy (S^*) have been determined using Freeman-Carroll method.

1. Introduction

The synthesized terpolymer resins, showing versatile applications and properties, attracted the attention of scientist and introduced the recent innovations in the polymer chemistry. These terpolymers can be used as high-energy material [1], ion-exchanger [2], semiconductors [3], antioxidants, fire proofing agent, optical storage data, binders, molding materials, and so forth. Literature survey reveals the chelation ion-exchange properties of 2, 4-dinitrophenylhydrazone of 2-hydroxyacetophenone-formaldehyde resin [4], and oximes of 2-hydroxyacetophenone-substituted benzoic acid-formaldehyde resin [5] for different metal ions. Thermogravimetric analysis of urea-formaldehyde polycondensate (UFPS) has been reported by Zeman and Tokarova [6]. Terpolymer resins having good thermal stability have enhanced the scope for development of some polymeric materials. The

study of the thermal degradation of terpolymer resins have recently become a subject of interest. Hong et al. studied the thermal decomposition behaviour of phosphorous containing copolyester [7]. In an earlier communication [8–11] from this department, numbers of studies on such terpolymers have been reported. However, no work seems to have been carried out on synthesis, characterization, and thermal stabilities of the terpolymer resins-IV from 8-hydroxyquinoline 5-sulphonic acid-melamine and formaldehyde. The present paper describes the composition of newly synthesized resin, its characterization by elemental analysis, average molecular weight (\bar{M}_n), determination of intrinsic viscosity in DMSO, FTIR Spectra, UV-Visible absorption spectra, NMR spectra, and thermal analysis by applying the Sharp-Wentworth and Freeman-Carroll methods. Energy of activation (E_a), kinetic parameter namely, Z , ΔS , ΔF , S^* , and order of reaction (n) were determined by applying the Freeman-Carroll method.

2. Experimental

2.1. Materials. The chemicals used in the synthesis of new terpolymer resin were procured from the market and were analar or Fulka or chemically pure grade. Whenever required, they were further purified by standard procedure.

2.2. Synthesis of 8-HQ5-SAMF-IV Terpolymer. The new terpolymer resin 8-HQ5-SAMF-IV was synthesized by condensing 8-hydroxyquinoline 5-sulphonic acid (0.4 mol) and melamine (0.2 mol) with 37% formaldehyde (0.7 mol) in a mol ratio of 4:2:7 in the presence of 2 M 200 mL HCl as a catalyst at $140^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for 6 h in an oil bath with occasional shaking to ensure thorough mixing. The separated terpolymer was washed with hot water and methanol to remove unreacted starting materials and acid monomers. The properly washed resin was dried, powdered, and then extracted with diethyl ether and then with petroleum ether to remove 8-hydroxyquinoline 5-sulphonic acid formaldehyde copolymer which might be present along with 8-HQ5-SAMF-IV terpolymer. The yellow colour resinous product was immediately removed from the flask as soon as reaction period was over and then purified. The reaction and suggested structure of 8-HQ5-SAMF-IV is shown in Figure 1.

The terpolymer was purified by dissolving in 10% aqueous sodium hydroxide solution, filtered, and reprecipitated by gradual dropwise addition of ice-cold 1:1 (v/v) concentrated hydrochloric acid/distilled water with constant and rapid stirring to avoid lump formation. The process of reprecipitation was repeated twice. The terpolymer sample 8-HQ5-SAMF-IV thus obtained was filtered, washed several times with hot water, dried in air, powdered, and kept in vacuum desiccator over silica gel. The yield of the terpolymer resin was found to be 76%. The elemental analysis is tabulated in Table 1.

3. Characterization

The viscosities were determined using Taun-Fuoss viscometer at six different concentrations ranging from 1.00 to 0.031% of terpolymer in DMSO at 30°C . The intrinsic viscosity η was calculated by relevant plots of the Huggins' equation and Kraemmer's equation:

$$\frac{\eta_{\text{sp}}}{C} = [\eta] + K_1 [\eta]^2 C, \quad (1)$$

$$\frac{\ln \eta_{\text{rel}}}{C} = [\eta] + K_2 [\eta]^2 C. \quad (2)$$

The number average molecular weight (\bar{M}_n) was determined by conductometric titration in nonaqueous medium such as dimethylsulphoxide (DMSO) using ethanolic KOH as a titrant. From the graph of specific conductance against milliequivalents of base, first and last break were noted from which degree of polymerisation ($\bar{D}\bar{P}$) and the number average molecular weight (\bar{M}_n) has been calculated for terpolymer resin.

Electron absorption spectrum of terpolymer resin was recorded in DMSO (spectroscopic grade) on Shimadzu double beam spectrophotometer in the range of 200 to 850 nm. An infrared spectrum of 8-HQ5-SAMF-IV terpolymer resin was recorded on Perkin-Elmer-983 spectrophotometer in KBr pallets in the wave number region of $4000\text{--}400\text{ cm}^{-1}$. A nuclear magnetic resonance (NMR) spectrum of newly synthesized terpolymer resin has been scanned on Bruker Advanced 400 NMR spectrometer using DMSO- d_6 . TGA of terpolymer resin has been carried out by using Perkin-Elmer TGS-II Thermogravimetric Analyzer at heating rate of 10°C per minute up to 800°C .

4. Results and Discussion

The newly synthesized and purified 8-HQ5-SAMF-IV terpolymer resin was found to be yellow in colour. The terpolymer is soluble in solvents such as DMF, DMSO, and THF while insoluble in almost all other organic solvents. The melting point of the terpolymer was determined by using electrically heated melting point apparatus and is found to be in 392 K. This resin was analyzed for carbon, hydrogen, nitrogen, and sulphur content. The details of elemental analysis are incorporated in Table 1. The terpolymer which has been used in the present investigation was prepared by the reaction given in Figure 1.

The number average molecular weight (\bar{M}_n) of the terpolymer has been determined by conductometric titration method in nonaqueous medium and using standard potassium hydroxide (0.05 M) in absolute ethanol as a titrant. The result is presented in Table 1. The specific conductance was plotted against milliequivalents of ethanolic KOH required for neutralization of 100 gm of each terpolymer. There are several breaks before the complete neutralization of all phenolic hydroxyl groups. The first break in the plot was the smallest break and assumed that this corresponds to a stage in titration when an average one phenolic hydroxy group of each chain was neutralized.

From the plot (Figure 2) the first and last break was noted. The average degree of polymerization ($\bar{D}\bar{P}$) and hence the number average molecular weights (\bar{M}_n) of terpolymer has been determined using the following formula [12, 13]:

$(\bar{D}\bar{P}) = \text{Total milliequivalents of base required for complete neutralisation} / \text{Milliequivalents of base required for smallest interval}$
 $(\bar{M}_n) = (\bar{D}\bar{P}) \times \text{Repeat unit weight.}$

The intrinsic viscosity $[\eta]$ was determined by the corresponding linear plots. Huggins' and Kraemmer's constants were determined by expressions 1 and 2.

According to the above relations, the plots (Figure 3) of η_{sp}/C and $\ln \eta_{\text{rel}}/C$ against C were linear with slopes of K_1 and K_2 , respectively. By extrapolating linear plot to zero concentration, intercepts on the viscosity function axis give $[\eta]$ value in both plots.

Values of intrinsic viscosity obtained from both plots have been found to be closed agreement with each other. The calculated values of the constants K_1 and K_2 in most cases satisfy the relation $K_1 + K_2 = 0.5$ favorably [14]. It was observed that terpolymer having higher (\bar{M}_n) shows

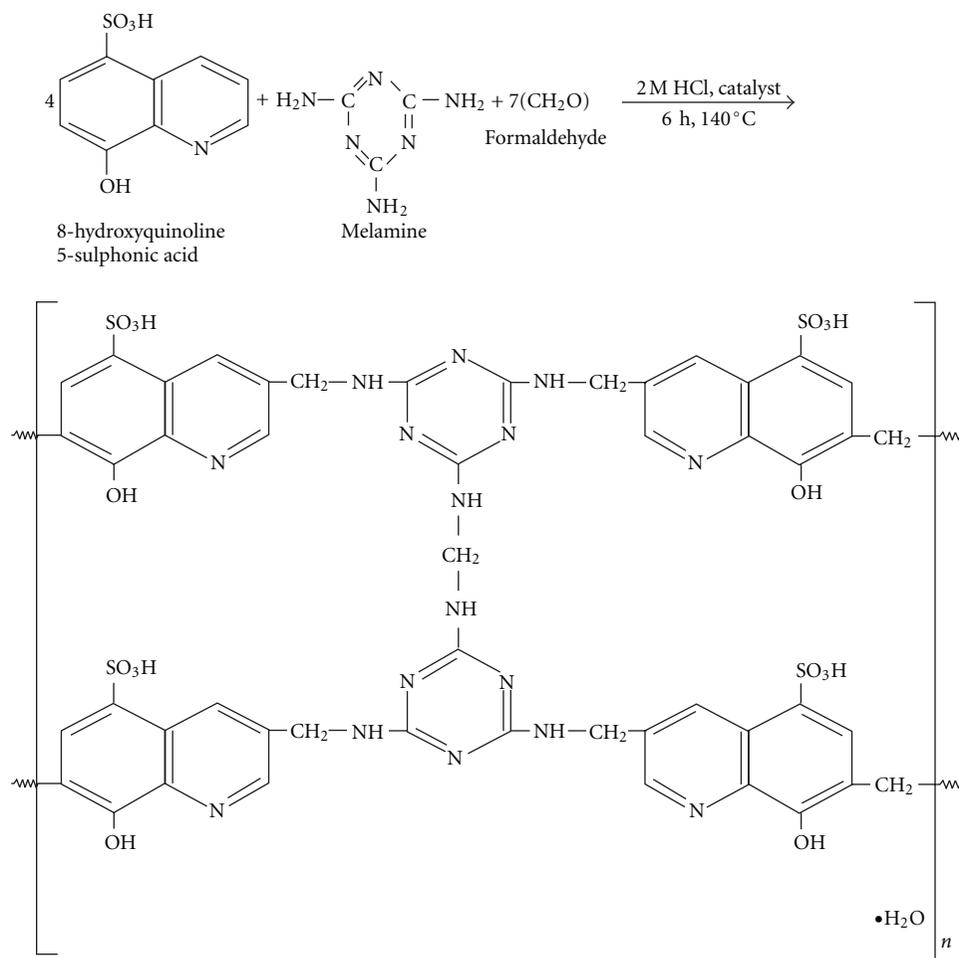


FIGURE 1: Reaction and expected structure of 8-HQ5-SAMF-IV terpolymer resin.

TABLE 1: Elemental analysis, molecular weight determination, and intrinsic viscosity of 8-HQ5-SAMF-IV terpolymer resin.

Empirical formula of repeat unit	Empirical weight of repeat unit (gm)	Average degree of polymerization (\overline{DP})	Average molecular weight (\overline{M}_n)	Elemental analysis				Intrinsic viscosity $[\eta]$ (dl/g)
				Percentage (%) of element				
				C	H	N	S	
				Found (Cal.)	Found (Cal.)	Found (Cal.)	Found (Cal.)	
$\text{C}_{55}\text{H}_{45}\text{N}_{11}\text{O}_{21}\text{S}_5$	1355	15.5	21002	48.15 (48.1)	2.68 (3.32)	10.79 (11.3)	11.25 (11.80)	1.31

higher value of $[\eta]$ which is in good agreement with earlier coworkers [14, 15].

The UV-Visible spectra (Figure 4) of the 8-HQ5-SAMF-IV terpolymer sample in pure DMSO was recorded in the region 200–850 nm at a scanning rate of $100 \text{ nm}\cdot\text{min}^{-1}$ and a chart speed of $5 \text{ cm}\cdot\text{min}^{-1}$. The 8-HQ5-SAMF-IV terpolymer sample gave two characteristics bands at 360–370 nm and 240–260 nm. These observed positions for the absorption bands have different intensities. The more intense band is due to $\pi \rightarrow \pi^*$ transition and the less intense is due to $n \rightarrow \pi^*$ transition. The $\pi \rightarrow \pi^*$ transition indicates the presence of aromatic nuclei, and $n \rightarrow \pi^*$ transition indicates presence of $-\text{NH}$ and $-\text{OH}$ group. The hyperchromic effect

is due to the presence of $-\text{OH}$ and $-\text{NH}$ groups, which act as auxochrome [16].

The IR (Figure 5) spectral studies revealed that the terpolymer gave broad absorption band appeared in the region $3506\text{--}3508 \text{ cm}^{-1}$ may be assigned to the stretching vibrations of phenolic hydroxyl ($-\text{OH}$) groups exhibiting intramolecular hydrogen bonding [17]. A sharp strong peak at $1556\text{--}1626 \text{ cm}^{-1}$ may be ascribed to aromatic skeletal ring. The bands obtained at $1210\text{--}1230 \text{ cm}^{-1}$ suggest the presence of methylene ($-\text{CH}_2$) bridge [18]. The 1, 2, 3, 5 substitution of aromatic benzene ring recognized by the sharp, medium/weak absorption bands appeared at $953\text{--}970$, $1145\text{--}1046$, $1182\text{--}1188$, and $1320\text{--}1321 \text{ cm}^{-1}$, respectively.

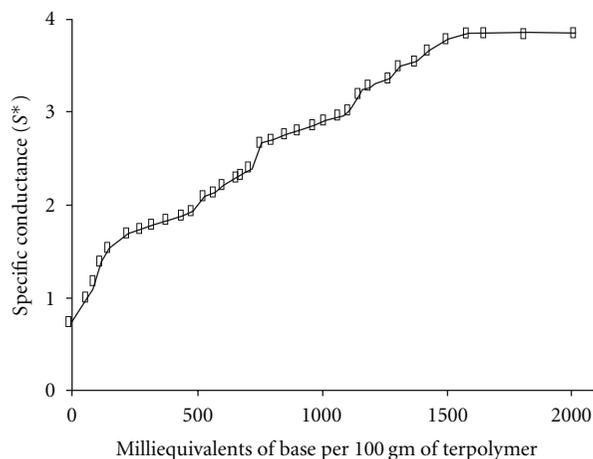


FIGURE 2: Conductometric titration curve of 8-HQ5-SAMF-IV resin.

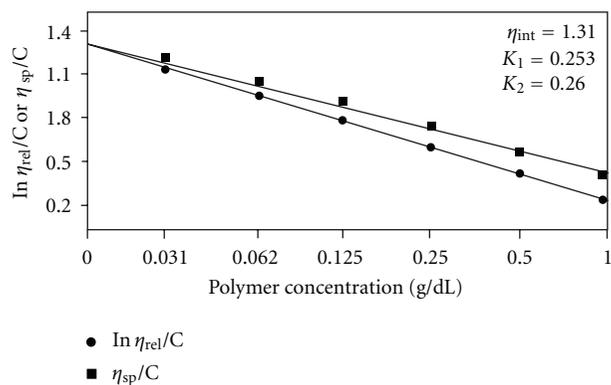


FIGURE 3: Viscometric curves of 8-HQ5-SAMF-IV terpolymer resin.

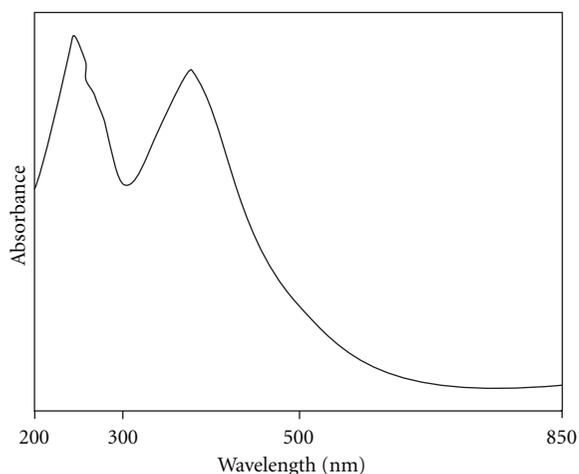


FIGURE 4: Uv-Visible spectrum of 8-HQ5-SAMF-IV terpolymer resin.

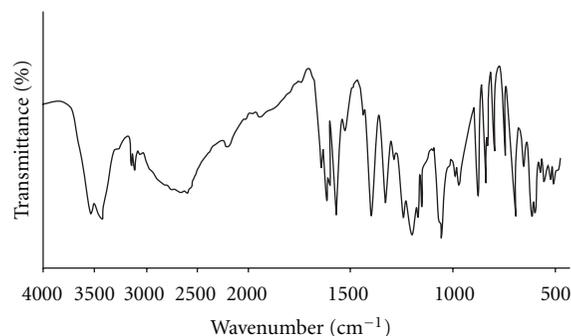


FIGURE 5: IR spectrum of 8-HQ5-SAMF-IV terpolymer resin.

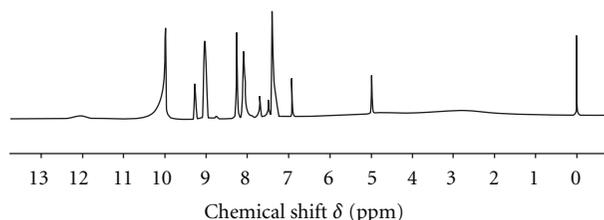


FIGURE 6: NMR spectrum of 8-HQ5-SAMF-IV terpolymer resin.

The presence of sharp and strong band at $3393\text{--}3402\text{ cm}^{-1}$ indicates the presence of --NH bridge. This band seems to be merged with very broad band of phenolic hydroxyl group.

The NMR spectrum (Figure 6) of 8-HQ5-SAMF-IV terpolymer was scanned in DMSO-d_6 solvent. The chemical shift (δ) ppm observed is assigned on the basis of data available in the literature [19, 20]. The singlet obtained in the region $4.98\text{--}4.96$ (δ) ppm may be due to the methylene proton of $\text{Ar-CH}_2\text{-N}$ moiety. The signal in the region of $7.35\text{--}7.41$ (δ) ppm is attributed to protons of --NH bridge. The weak multiplet signals (unsymmetrical pattern) in the region of $8.25\text{--}8.20$ (δ) ppm may be attributed to aromatic proton (Ar-H). The signal in the range at 9.02 to 9.06 (δ) ppm may be due to phenolic hydroxyl protons. The much downfield chemical shift for phenolic --OH indicates clearly the intramolecular hydrogen bonding of --OH group [20, 21]. The signals in the range of $9.96\text{--}9.92$ (δ) ppm are attributed to proton of $\text{--SO}_3\text{H}$ groups.

The polymer under study is terpolymer and, hence, it is very difficult to assign the exact structure. However, on the basis of the nature and reactive site of the monomers and taking into consideration the linear structure of other substituted phenol formaldehyde polymers and the linear branched nature of urea-formaldehyde polymers the most probable structure [22] of proposed for 8-HQ5-SAMF-IV terpolymer has been depicted in Figure 1, showing linear branch structure.

4.1. Thermogravimetry. Thermogravimetry of 8-HQ5-SAMF-IV terpolymer resin has been carried out using Perkin-Elmer thermogravimetric analyser. A brief account of thermal behavior of 8-HQ5-SAMF-IV terpolymer is given in Figures 7–9.

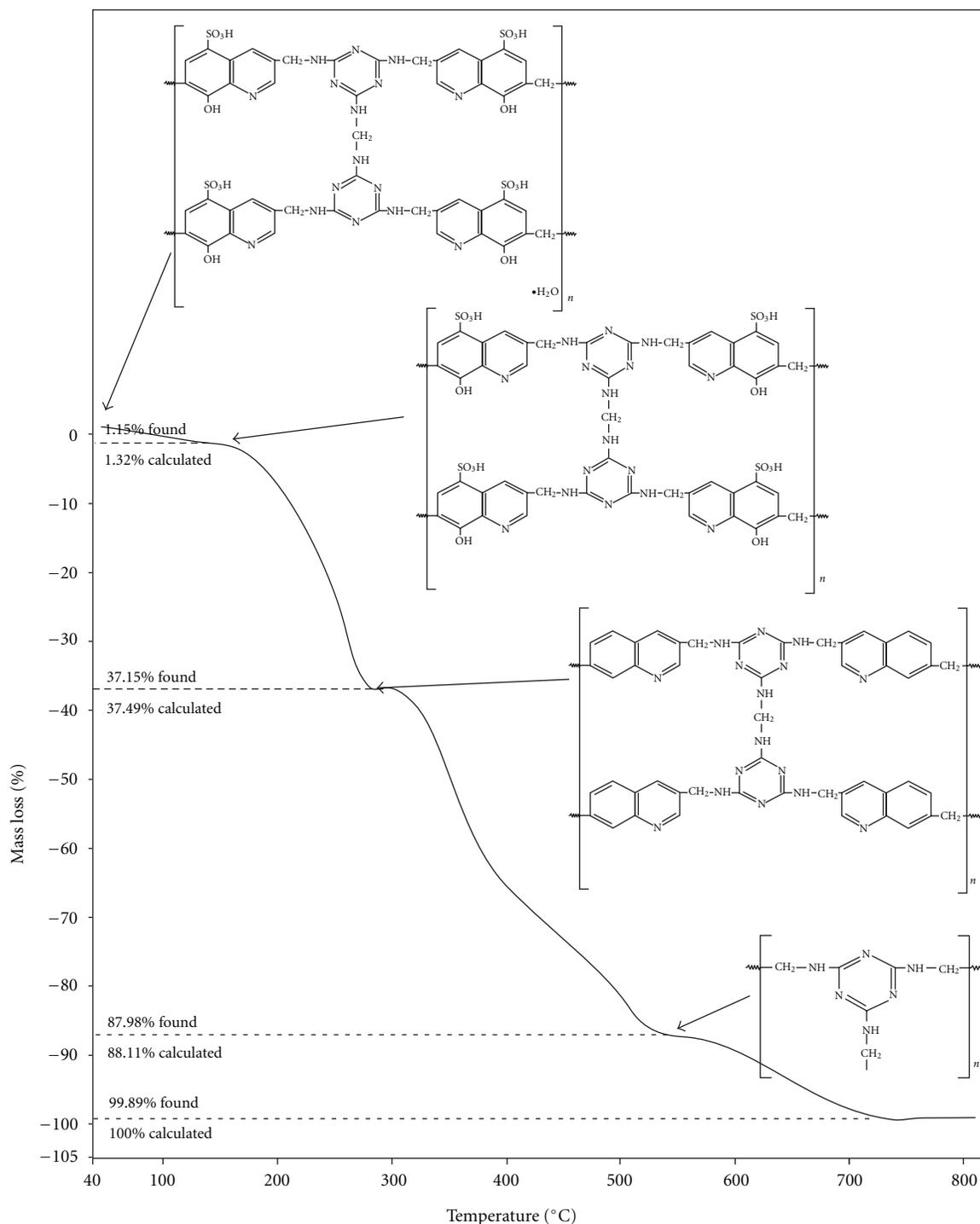


FIGURE 7: Decomposition pattern of 8-HQ5-SAMF-IV terpolymer resin.

TABLE 2: Results of thermogravimetric analysis of 8-HQ5-SAMF-IV terpolymer resin.

Terpolymer resin	Half decomposition temp. (K)	Activation energy (KJ/mol)		Entropy change $-\Delta S$ (J/degree)	Free energy change ΔF (KJ/mol)	Frequency factor Z (sec ⁻¹)	Apparent entropy S* (J)	Order of reaction found (n)
8-HQ5-SAMF-IV	653	FC	SW	-160.9	109.7	780	-19.1	0.98
		36.8	35.5					

FC = Freeman-Carroll, SW = Sharp-Wentworth.

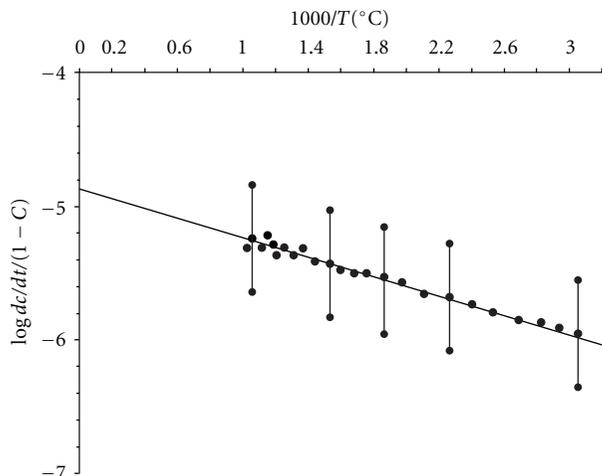


FIGURE 8: Sharp-Wentworth plot of 8-HQ5-SAMF-IV terpolymer resin.

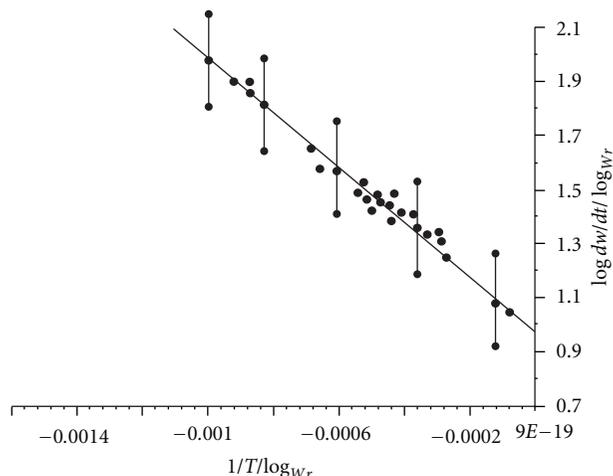


FIGURE 9: Freeman-Carroll plot of 8-HQ5-SAMF-IV terpolymer resin.

4.2. TG of 8-HQ5-SAMF-IV Terpolymer. Decomposition pattern of 8-HQ5-SAMF-IV terpolymer resin has been shown in Figure 7. Examination of thermogram found that the terpolymer under investigation is resistance to elevated temperature. From TG, the thermal degradation has been studied in four temperature ranges (40–180°C, 180–300°C, 300–560°C, 560–800°C). The initial weight loss of 1.15% found and 1.32% calculated at 40–180°C may be attributed due to removal of water in the form of moisture retained in the sample during synthesis. In the first stage of thermal degradation (180–300°C), four hydroxyl groups and four sulphonic groups become activated due to increasing thermal vibration in the molecule, so recross linking occurs and develops a strain in the macromolecule, increasing instability and losses in the fine structure of the sample, resulting in a loss of four hydroxyl and sulphonic groups (37.18% found and 36.98% calculated). In the second stage of thermal degradation (300–560°C), due to

increasing temperature the strain in the molecule increases which increases instability more, and causes the mass loss corresponding to 87.98% found and 88.11% calculated, equivalent to the decomposition of four aromatic quinoline rings. In the third stage of thermal decomposition (560–800°C) the strained in the macromolecule suffer high cross-linking and depolymerization occurs. This causes the weight loss of about 99.89% found and 100% calculated and may be due to degradation of 1, 3, 5 triazine ring with its side chain. The rest of the part after complete degradation is the residue, which was found negligible.

In the present investigation Sharp-Wentworth and Freeman-Carroll methods have been used to determine the kinetic parameters of 8-HQ5-SAMF-IV terpolymer sample.

4.3. Sharp-Wentworth Method. In this method the following expression is used:

$$\log \left[\frac{dc/dt}{\Delta(1-c)} \right] = \log \left(\frac{A}{\beta} \right) - \frac{E_a}{2.303R} - \frac{1}{T}, \quad (3)$$

where β is the linear heating rate. The graph of $\log[(dc/dt)/\Delta(1-c)]$ versus $1/T$ has been plotted. The graph is a straight line with E_a as slope and A as intercept. The linear relationship confirms that the assumed order (n) = 1 is correct.

4.4. Freeman-Carroll Method. In this method the following expression is used:

$$\frac{\Delta \log(dw/dt)}{\Delta \log W_r} = \left(-\frac{E_a}{2.303} R \right) - \frac{\Delta(1/T)}{\Delta \log W_r} + n, \quad (4)$$

where (dw/dt) = rate of change of weight of terpolymer sample with respect to time $W_r = W_c - W$, where W_c is the weight loss at the completion of the terpolymer reaction or at definite time and W is the total weight loss upto time t .

T is the temperature, R is the gas constant, and n is the order of reaction.

Hence the graph of $(\Delta \log(dw/dt))/(\Delta \log W_r)$ versus $\Delta(1/T)/(\Delta \log W_r)$ should give on Y axis ($x = 0$) an intercept for the value of n , the order of reaction, and the slope $m = -E_a/2.303R$. The detailed procedure is clearly laid out for one representative sample as an illustration.

A plot of percentage mass loss versus temperature has been shown in Figure 7 for 8-HQ5-SAMF-IV terpolymer. From this curve, initial and half decomposition temperatures were determined (Table 2), which can explain the thermal stability of the polymer, neglecting the degree of decomposition (Table 2).

Using thermal decomposition data and applying the Sharp-Wentworth method Figure 8, activation energy is calculated which is in agreement with the activation energy calculated by Freeman-Carroll method, Figure 9 [22]. Thermodynamic parameters such as entropy change (ΔS), free energy change (ΔF), frequency factor (Z), and apparent entropy (S^*) calculated on the basis of thermal activation energy are given in Table 2. Using equations given below, all thermodynamic parameters have been calculated:

(i) Entropy change

$$\text{Intercept} = \log \frac{KR}{h\Phi E} + \frac{\Delta S}{2.303R}, \quad (5)$$

where, $K = 1.3806 \times 10^{-16}$ erg/deg/mole, $R = 1.987$ cal/deg/mole, $h = 6.625 \times 10^{-27}$ erg sec, $\Phi = 0.166$, ΔS = change in entropy, E = activation energy from graph.

(ii)

$$\Delta F = \Delta H - T\Delta S \quad (6)$$

where, ΔH = Enthalpy change = Activation energy, T = Temperature in K, ΔS = Entropy change {from (i) used}.

(iii) Frequency factor

$$B_{2/3} = \frac{\log ZE_a}{R\Phi}, \quad (7)$$

$$B_{2/3} = \log 3 + \log [1 - 3\sqrt{1 - \alpha}] - \log p(x), \quad (8)$$

where, Z = Frequency factor, B = Calculated from (8), $\log p(x)$ = Calculated from Doyle table corresponding to activation energy.

(iv) Apparent entropy change:

$$S^* = 2.303R \log \frac{Zh}{RT^*}, \quad (9)$$

where, Z = from relation (7), T^* = Temperature at which half of the compound is decomposed from its total loss.

By using the data of the Freeman-Carroll method, various thermodynamics parameters have been calculated (Table 2). From the abnormally low values of frequency factor, it may be concluded that the decomposition reaction of 8-HQ5-SAMF-IV terpolymer resin can be classed as a "slow" reaction. There is no other obvious reason [23, 24].

Fairly good straight line plots are obtained using two methods. This is expected since the decomposition of terpolymer is known not to obey first-order kinetics perfectly [23, 24].

5. Conclusions

- (1) A terpolymer 8-HQ5-SAMF-IV, based on the condensation reaction of 8-hydroxyquinoline 5-sulphonic acid-melamine-formaldehyde in the presence of acid catalyst, was prepared.
- (2) As the degradation of the terpolymer under investigation started at high temperature which indicates that the terpolymer 8-HQ5-SAMF-IV is thermally stable at elevated temperature.
- (3) Low value of frequency factor may be concluded that the decomposition reaction of 8-hydroxyquinoline 5-sulphonic acid-melamine-formaldehyde terpolymer resin-IV can be classified as "slow reaction".

Acknowledgments

The authors are grateful to the director of LIT, RTM Nagpur University, Nagpur, and the Head of the Department of Chemistry, LIT, RTM Nagpur University, Nagpur, for providing laboratory facility. They are also thankful to the director of SAIF, Punjab University, Chandigarh.

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