

# Synthetic Resin. XII. Synthesis and Characterization of New Terpolymer Resins Derived from 8-Hydroxyquinoline 5-Sulfonic Acid, Melamine, and Formaldehyde

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**ABSTRACT:** Terpolymers 8-hydroxyquinoline 5-sulphonic acid–melamine–formaldehyde (8-HQ5-SAMF) were synthesized through the condensation of 8-hydroxyquinoline 5-sulfonic acid and melamine with formaldehyde in the presence of an acid catalyst. Four different terpolymers were synthesized with various molar proportions of the reacting monomers. The terpolymer resin compositions were determined on the basis of elemental analysis. The number-average molecular weights of these resins were determined by conductometric titration in a nonaqueous

medium; viscometric measurements in dimethyl sulfoxide were carried out to ascertain the characteristic functions and constants. Ultraviolet–visible, Fourier transform infrared, <sup>1</sup>H-NMR, and <sup>13</sup>C-NMR spectroscopy and thermogravimetric analysis were used to elucidate the structures. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 116: 1400–1407, 2010

**Key words:** degree of polymerization (DP); polycondensation; resins; structure; synthesis

## INTRODUCTION

The synthesis of new terpolymers attracts the attention of researchers because of their versatile utility in our day-to-day life. Resins of terpolymers, which form a special class of polymers, are widely known for their uses. Various modified phenol–formaldehyde resins have a large number of practical applications<sup>1–3</sup> as highly thermally conductive materials, excellent abrasives, and ion exchangers, and many other renowned applications are reported in the literature. Syntheses of various kinds of terpolymers with various organic compounds and their physicochemical properties and applications have been studied by various researches.<sup>4,5</sup> Jadhao et al.<sup>6</sup> synthesized and studied the ion-exchange properties of 2,2'-dihydroxybiphenyl, urea, and formaldehyde terpolymer resins. Extensive research work has been reported on terpolymer resins derived from phenolic compounds (amine, amide–trioxane, and formaldehyde from this department).<sup>7,8</sup> However, no work seems to have been carried out on the synthesis and physicochemical technique used for the characterization of newly synthesized terpolymer resins derived from 8-hydroxyquinoline 5-sulfonic acid (8-HQ5-SA) and melamine with formaldehyde. This article

presents the synthesis of these new terpolymer resins and their systematic structural study with the help of elemental and spectral studies and other various physicochemical techniques.

## EXPERIMENTAL

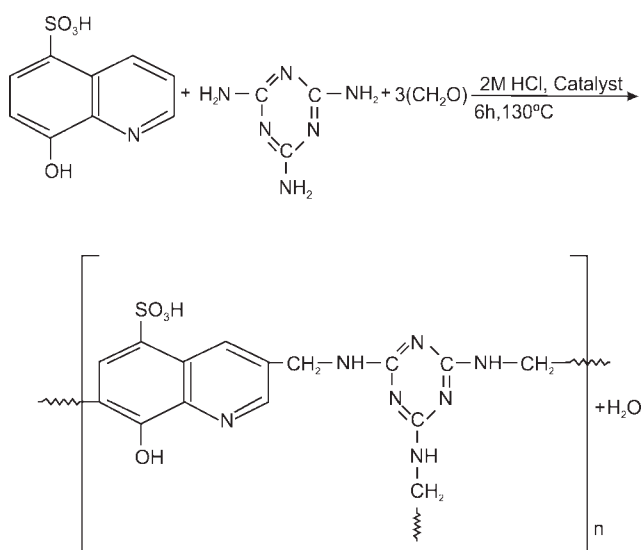
### Materials

The entire chemicals used in the synthesis of the various terpolymer resins were procured from the market and were analar-grade (Fulka Manufacturing Co., Ltd., Mumbai, India) or chemically pure grade, whenever required, were further purified by the standard procedure.

### Synthesis of an 8-HQ5-SAMF terpolymer resin (8-HQ5-SAMF-1)

The new terpolymer resin 8-Hydroxyquinoline 5-sulphonic acid–Melamine–Formaldehyde (8-HQ5-SAMF-1) was synthesized through the condensation of 8-HQ5-SA (2.43 g, 0.1 mol) and melamine (1.26 g, 0.1 mol) with 37% formaldehyde (11.1 mL, 0.3 mol) in a molar ratio of 1 : 1 : 3 in the presence of 200 mL of 2M HCl as a catalyst at 130°C ± 2°C for 6 h in an oil bath with occasional shaking to ensure thorough mixing. The separated terpolymers were washed with hot water and methanol to remove the unreacted starting materials and acid monomers. The properly washed resin was dried, powdered,

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**Figure 1** Reaction and suggested structure of a representative 8-HQ5-SAMF terpolymer resin.

and extracted first with diethyl ether and then with petroleum ether to remove any 8-HQ5-SA-formaldehyde copolymer that might be present along with 8-HQ5-SAMF terpolymer. The yellow, resinous product was immediately removed from the flask as soon as the reaction period was over and then purified. The reaction and suggested structure of 8-HQ5-SAMF-1 are shown in Figure 1.

The terpolymer was purified by dissolution in an 8% aqueous sodium hydroxide solution, filtered, and reprecipitated by the 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-1 thus obtained was filtered, washed several times with hot water, dried in air, powdered, and kept in a vacuum desiccator over silica gel. The yield of the terpolymer resin was found to be 75%.

Similarly, other terpolymer resins (8-HQ5-SAMF-2, 8-HQ5-SAMF-3, and 8-HQ5-SAMF-4) were synthesized through the variation of the molar proportions of the starting materials (i.e., 2 : 1 : 4, 3 : 1 : 5, and 4 : 2 : 7, respectively) with little variation of the experimental conditions. The sample yield (%) and reaction details are tabulated in Table I.

### Characterization of the terpolymer resins

The viscosity was determined with a Taun-Fuoss viscometer (Laxminarayan Institute of Technology, Nagpur, India) at six different terpolymer concentrations ranging from 1.00 to 0.031% in dimethyl sulfoxide (DMSO) at 30°C. The intrinsic viscosity ( $[\eta]$ ) was calculated from relevant plots of the Huggins and Kraemer equations:

**TABLE I**  
Synthesis and Physical Data of the 8-HQ5-SAMF Terpolymer Resins

Terpolymer	Reactant				Yield (%)	Color	mp (K)	Elemental analysis (%)					
	8-HQ5-SA (mol)	Melamine (mol)	Formaldehyde (mol)	2M HCl as catalyst (mL)				C	H	N	S	Found (Calcd)	Found (Calcd)
8-HQ5-SAMF-1	0.1	0.1	0.3	200	75	Yellow	418	43.19 (44.22)	3.36 (4.10)	23.35 (24.00)	6.98 (7.86)	Found (Calcd)	Found (Calcd)
8-HQ5-SAMF-2	0.2	0.1	0.4	200	80	Yellow	422	45.51 (46.58)	3.15 (3.72)	16.77 (17.39)	9.15 (9.95)	Found (Calcd)	Found (Calcd)
8-HQ5-SAMF-3	0.3	0.1	0.5	200	75	Yellow	427	47.62 (48.55)	2.90 (3.58)	13.86 (14.56)	10.50 (11.09)	Found (Calcd)	Found (Calcd)
8-HQ5-SAMF-4	0.4	0.2	0.7	200	80	Yellow	432	48.10 (48.70)	2.68 (3.32)	10.79 (11.36)	11.25 (11.80)	Found (Calcd)	Found (Calcd)

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

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

where  $\eta_{\text{sp}}$  is the specific viscosity,  $\eta_{\text{rel}}$  is the relative viscosity,  $K_1$  is the Huggins' constant and  $K_2$  is the Kraemmer's constant, and  $C$  is the concentration. The number-average molecular weight ( $M_n$ ) values were determined by conductometric titration in a nonaqueous medium such as DMSO with ethanolic KOH as a titrant. From the graphs of the specific conductance against the milliequivalents of the base, the first and last breaks were noted, from which the degree of polymerization (DP) and  $M_n$  were calculated for each terpolymer resin.

Ultraviolet-visible (UV-vis) spectra of all terpolymer resins were recorded in DMSO (spectroscopic-grade) on a Shimadzu double-beam spectrophotometer (UV-Visible Spectrometer) (Shimadzu Scientific Instruments, Kyoto, Japan) in the range of 200–850 nm at the Sophisticated Analytical Instrumentation Facility of Punjab University (Chandigarh, India). IR spectra of four 8-HQ5-SAMF terpolymer resins were recorded on a PerkinElmer 983 spectrophotometer (Mono Instrument Service Ltd, Ontario, Canada) in KBr pellets in the wavenumber region of 4000–400  $\text{cm}^{-1}$  at the Sophisticated Analytical Instrumentation Facility. Nuclear magnetic resonance (NMR) spectra of newly synthesized terpolymer resins were scanned on a Bruker Advanced 400 NMR spectrometer (London, United Kingdom) with DMSO- $d_6$  at the Sophisticated Analytical Instrumentation Facility. A  $^{13}\text{C}$ -NMR spectrum was also recorded with the Bruker 400-MHz spectrometer to identify the carbon linkage at NCL (Pune, India). Thermogravimetric analysis of all terpolymer resins was carried out with a PerkinElmer TGS-II thermogravimetric analyzer at a heating rate of 10°C/min up to 800°C. The spectral data and characterization matched the probable structure of the terpolymers, as shown in Figure 2.

## RESULTS AND DISCUSSION

All the newly synthesized and purified 8-HQ5-SAMF terpolymer resins were found to be yellow. The copolymers were soluble in solvents such as dimethylformamide (DMF), DMSO, and tetrahydrofuran but insoluble in almost all other organic solvents. The melting points of these terpolymers were determined with an electrically heated melting point apparatus and were found to be in the range of 418–432 K. These resins were analyzed for carbon, hydrogen, and nitrogen contents. The details of the synthesis of the copolymers along with the color,

melting point, yield, and elemental analysis are incorporated into Table I. The copolymer used in this investigation was prepared with the reaction presented in Figure 1.

The  $M_n$  values of these copolymers were determined by conductometric titration in a nonaqueous medium with standard potassium hydroxide (0.05M) in absolute ethanol as a titrant. The results are presented in Table II. The specific conductance was plotted against the milliequivalents of ethanolic KOH required for neutralization of 100 g of each copolymer. There were several breaks before the complete neutralization of all phenolic hydroxyl groups. The first break in the plot was the smallest break, and we assumed that this corresponded to a stage in titration when an average of one phenolic hydroxyl group of each chain was neutralized.

From the plot, the first and final breaks were noted. The average DP values and hence the  $M_n$  values of all the copolymers were determined with the following formula:

$$\text{DP} = \frac{\text{Total milliequivalents of base required for complete neutralization}}{\text{Milliequivalents of base required for the smallest interval}} \quad (3)$$

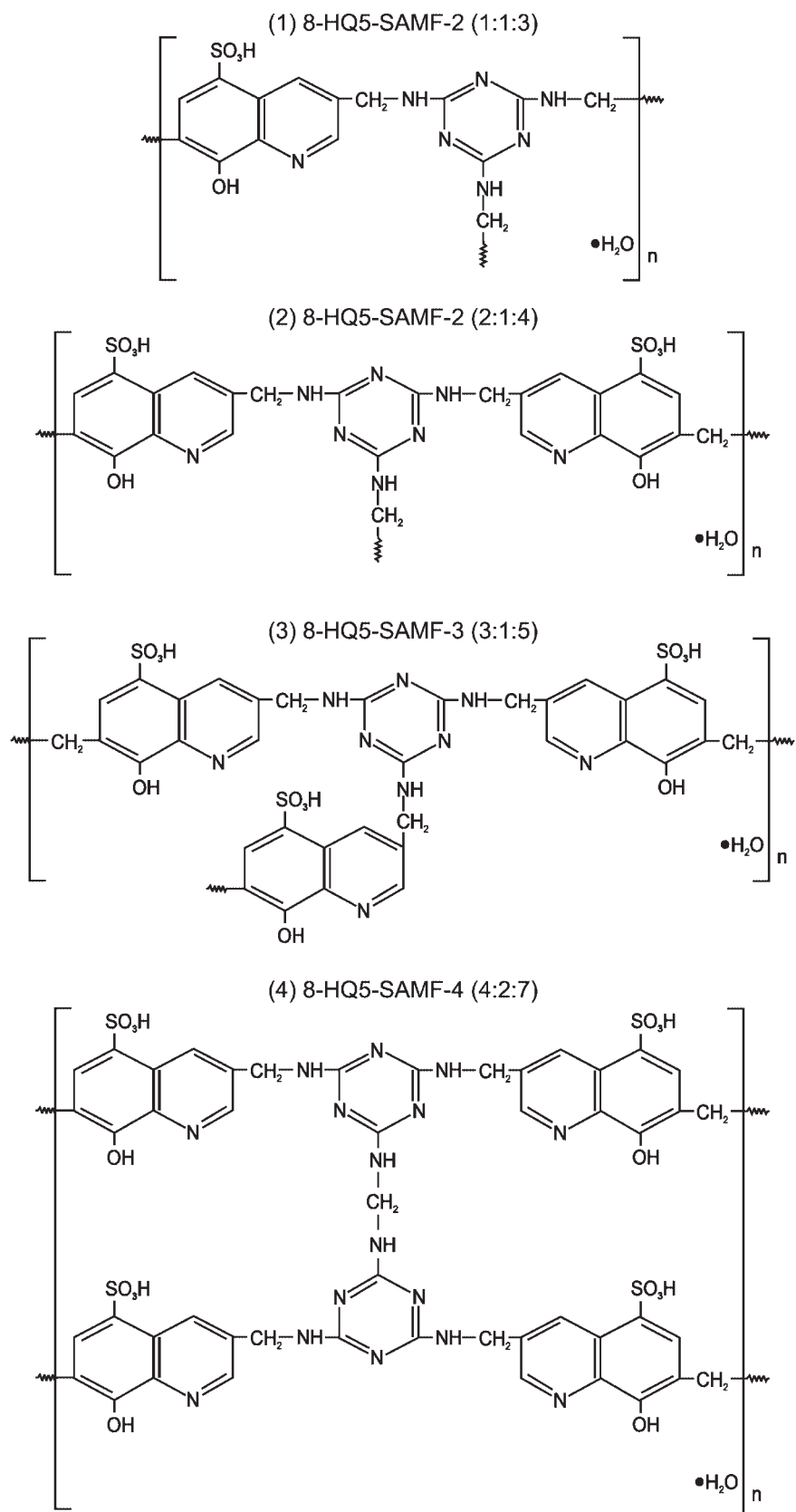
$$M_n = \text{DP} \times \text{Repeat unit weight} \quad (4)$$

The molecular weights of the copolymers increased with an increase in the 8-HQ5-SA content. This observation agreed with the trend observed earlier by others.<sup>9,10</sup>

Viscosity measurements were carried out at 300 K in freshly triple-distilled DMSO with a Tuan-Fuoss viscometer at six different concentrations ranging from 1.00% to 0.031%. The reduced viscosity versus the concentration was plotted for each set of data. The intrinsic viscosity  $[\eta]$  was determined from the corresponding linear plots (Fig. 3).  $K_1$  and  $K_2$  were determined.

According to the aforementioned relations, the plots of  $\eta_{\text{sp}}/C$  and  $\ln \eta_{\text{rel}}/C$  against  $C$  were linear with slopes of  $K_1$  and  $K_2$ , respectively. By the extrapolation of linear plots to the zero concentration, intercepts on the viscosity function axis provided the  $[\eta]$  values in both plots. The  $[\eta]$  values obtained from both plots were found to closely agree with each other. The calculated values of constants  $K_1$  and  $K_2$  (Table II) in most cases favorably satisfied the relation  $K_1 + K_2 = 0.5$ .<sup>11</sup> It was observed that a copolymer with a higher  $M_n$  value had a higher value of  $[\eta]$ , and this is in good agreement with the earlier work of other researchers.<sup>11,12</sup>

The UV-vis spectra (Fig. 4) of all the 8-HQ5-SAMF terpolymer samples in pure DMSO were

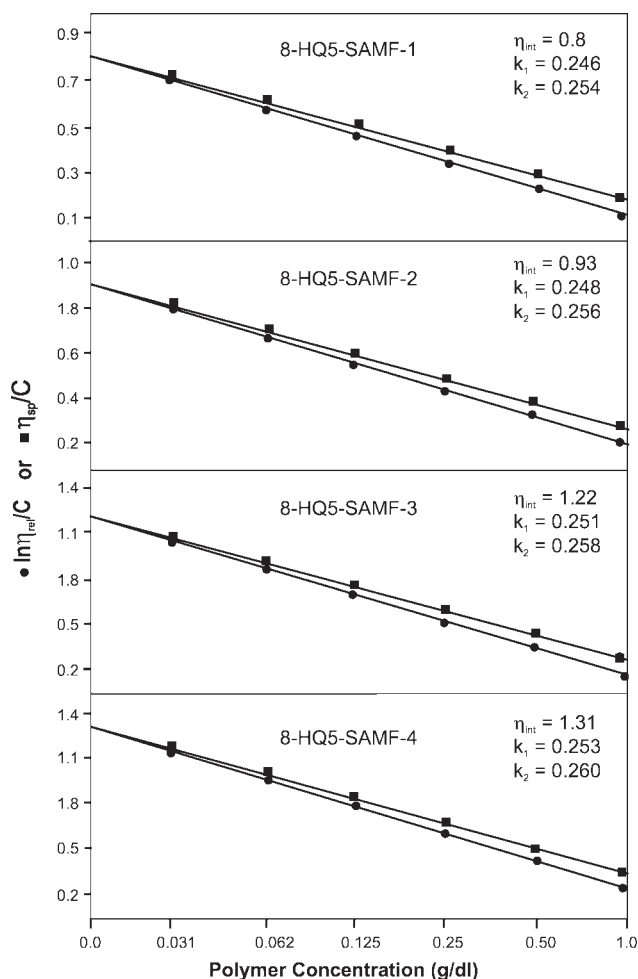


**Figure 2** Suggested structures of the terpolymer resins: (1) 8-HQ5-SAMF-1, (2) 8-HQ5-SAMF-2, (3) 8-HQ5-SAMF-3, and (4) 8-HQ5-SAMF-4.

**TABLE II**  
Molecular Weight Determination, Viscometric Data, and IR Frequencies for the 8-HQ5-SAMF Terpolymer Resins

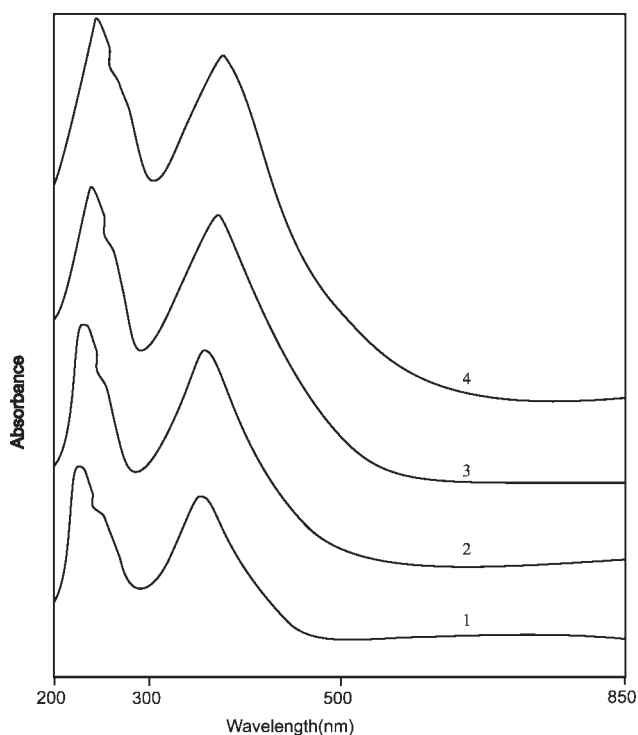
Copolymer	Empirical formula of the repeat unit	Empirical weight of the repeat unit (g)	DP	$M_n$	$[\eta]$ (dL/g)	$K_1$	$K_2$	$K_1 + K_2$	Wave number (cm <sup>-1</sup> )	Important IR frequencies	
										Assignments	
8-HQ5-SAMF-1	C <sub>15</sub> H <sub>17</sub> N <sub>7</sub> O <sub>3</sub> S	407	13.00	5291	0.80	0.246	0.254	0.500	3506–3508 <sub>b, st</sub> 3391–3402 <sub>b, st</sub> 3080–3102 <sub>b, st</sub> 1556–1626 <sub>b, st</sub> 1421–1502 <sub>b, st</sub> 1385–1387 <sub>sh, st</sub> 1228–1130 <sub>sh, st</sub> 1271–1273 <sub>sh, st</sub> 953–970 <sub>sh, wv</sub> 1045–1046 1182–1188 1320–1321	>OH phenol >NH linkage (amide/imides) Intramolecular hydrogen bonding Aromatic ring >CH <sub>2</sub> bending (scissoring) >CH <sub>2</sub> bending (twisting and wagging) >CH <sub>2</sub> (methyl bridge) >CH <sub>2</sub> (plane bending) 1,2,3,5-Substitution in benzene skeleton	
8-HQ5-SAMF-2	C <sub>24</sub> H <sub>23</sub> N <sub>8</sub> O <sub>3</sub> S <sub>2</sub>	631	14.25	8991	0.93	0.248	0.256	0.504			
8-HQ5-SAMF-3	C <sub>35</sub> H <sub>31</sub> N <sub>9</sub> O <sub>13</sub> S <sub>3</sub>	887	14.50	12774	1.22	0.251	0.258	0.509			
8-HQ5-SAMF-4	C <sub>49</sub> H <sub>47</sub> N <sub>16</sub> O <sub>17</sub> S <sub>4</sub>	1259	15.50	19514	1.31	0.253	0.260	0.513			

b = broad; m = medium; sh = sharp; st = strong; w = weak.



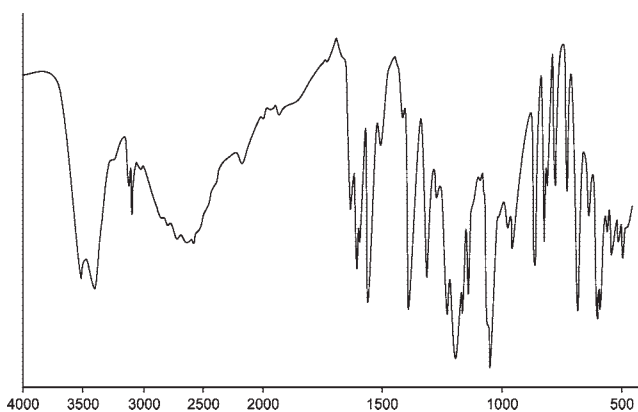
**Figure 3** Viscometric plots of the terpolymer resins: (1) 8-HQ5-SAMF-1, (2) 8-HQ5-SAMF-2, (3) 8-HQ5-SAMF-3, and (4) 8-HQ5-SAMF-4.

recorded in the region of 200–850 nm at a scanning rate of 100 nm/min and at a chart speed of 5 cm/min. All four 8-HQ5-SAMF terpolymer samples displayed two characteristic bands at 360–370 and 240–260 nm. These observed positions for the absorption bands had different intensities. The more intense band at 240–260 nm was due to the allowed transition ( $\pi \rightarrow \pi^*$ ) of the quinoline ring, which readily attained coplanarity and shoulder merging (loss of fine structure), and also was due to chromophore groups such as  $\text{>C=C}$ ,  $\text{>C=N}$  and  $-\text{SO}_3\text{H}$  groups, and the less intense band at 360–370 nm may have been due to the forbidden transition ( $n \rightarrow \pi^*$ ) in  $\text{>C=N}$  and  $-\text{SO}_3\text{H}$  groups. Thus, the  $\pi \rightarrow \pi^*$  transition indicates the presence of aromatic nuclei, and the  $n \rightarrow \pi^*$  transition indicates the presence of  $-\text{NH}$  and  $-\text{OH}$  groups. The bathochromic shift from the basic values (230 and 310 nm) may have been due to the combined effect of conjugation (due to chromophores) and phenolic hydroxyl groups as well as  $-\text{NH}$  groups (auxochromes). The hyperchromic effect was due to the presence of  $-\text{OH}$  and  $-\text{NH}$



**Figure 4** UV-vis spectra of the terpolymer resins: (1) 8-HQ5-SAMF-1, (2) 8-HQ5-SAMF-2, (3) 8-HQ5-SAMF-3, and (4) 8-HQ5-SAMF-4.

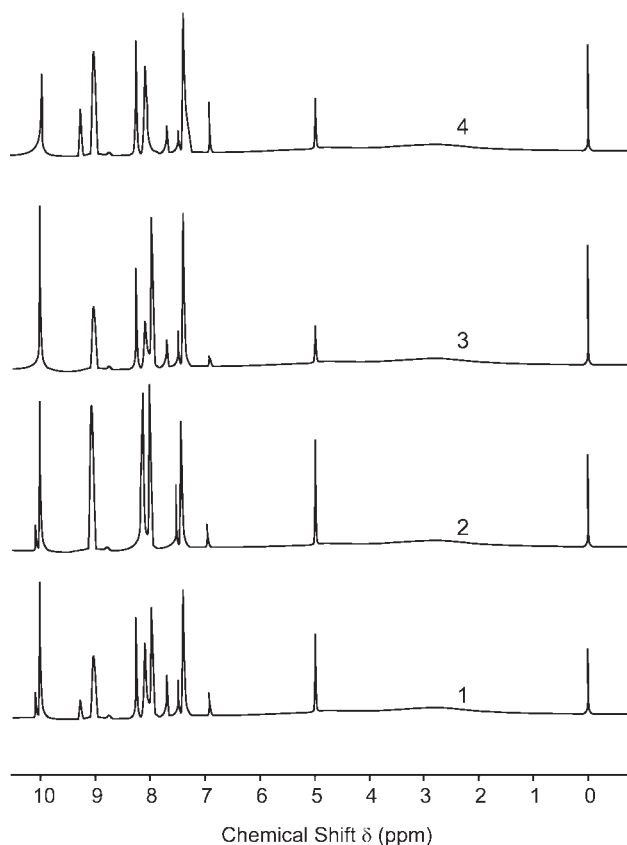
groups, which acted as auxochromes.<sup>13</sup> From the spectra of 8-HQ5-SAMF terpolymer resins, it was observed that the  $\epsilon_{\max}$  (the wavelength of absorption where intensity is maximum) value gradually increased in the order of 8-HQ5-SAMF-1 < 8-HQ5-SAMF-2 < 8-HQ5-SAMF-3 < 8-HQ5-SAMF-4. The increasing order of  $\epsilon_{\max}$  values may have been due to the introduction of more and more aromatic ring and auxochrome phenolic —OH and —NH groups in the repeated unit of the terpolymer resins, it may have been due to distortion of the chromophore brought about by ring strain or by steric hindrance, or it may have been due to an increase in the possi-



**Figure 5** IR spectrum of the 8-HQ5-SAMF-1 terpolymer resin.

bility of the interaction of nonbonding electrons to the  $\pi$ -electron system. This observation was found to be in good agreement with the proposed structures of the terpolymer resins (Fig. 2).

The IR spectral data are tabulated in Table II, and IR spectra of all four 8-HQ5-SAMF terpolymers are depicted in Figure 5. The IR spectral studies revealed that all these terpolymers gave rise to nearly the same spectral patterns. A broad absorption band appearing in the region of  $3506\text{--}3508\text{ cm}^{-1}$  could be assigned to the stretching vibrations of phenolic hydroxyl (—OH) groups exhibiting intramolecular hydrogen bonding.<sup>14</sup> A sharp, strong peak at  $1556\text{--}1626\text{ cm}^{-1}$  could be ascribed to an aromatic skeletal ring. The bands obtained at  $1210\text{--}1230\text{ cm}^{-1}$  suggest the presence of a methylene (—CH<sub>2</sub>) bridge.<sup>15</sup> The 1,2,3,5-substitution of the aromatic benzene ring, recognized by sharp, medium/weak absorption bands, appeared at  $953\text{--}970$ ,  $1145\text{--}1146$ ,  $1182\text{--}1188$ , and  $1320\text{--}1321\text{ cm}^{-1}$ , respectively. The presence of a sharp and strong band at  $3393\text{--}3402\text{ cm}^{-1}$  indicated the presence of a —NH bridge. This band seemed to have merged with the very broad band of the phenolic hydroxyl group.



**Figure 6** NMR spectra of the terpolymer resins: (1) 8-HQ5-SAMF-1, (2) 8-HQ5-SAMF-2, (3) 8-HQ5-SAMF-3, and (4) 8-HQ5-SAMF-4.

TABLE III  
<sup>1</sup>H-NMR Spectral Data for the 8-HQ5-SAMF Terpolymer Resins

Observed $\delta$ (ppm)				Nature of the assigned proton	Expected $\delta$ (ppm)
8-HQ5-SAMF-1	8-HQ5-SAMF-2	8-HQ5-SAMF-3	8-HQ5-SAMF-4		
4.98	4.95	4.96	4.96	Methylene proton of the Ar—CH <sub>2</sub> —N moiety	3.00–3.5
7.39	7.37	7.37	7.41	Proton of the —NH bridge	5.00–8.00
8.25	8.19	8.22	8.20	Aromatic proton (Ar—H)	6.2–8.5
9.02	9.07	9.04	9.06	Proton of the phenolic —OH involved in intramolecular hydrogen bonding	8.00–10
9.96	9.91	9.95	9.92	Proton of the SO <sub>3</sub> H group	9–10

The NMR spectra of all four 8-HQ5-SAMF terpolymers were scanned in the solvent DMSO-*d*<sub>6</sub>. The spectra are presented in Figure 6, and the spectral data are listed in Table III. From the spectra, it is revealed that all the 8-HQ5-SAMF terpolymers gave rise to different <sup>1</sup>H-NMR spectral patterns because each 8-HQ5-SAMF terpolymer possessed a set of protons with different electron environments. The observed chemical shift [ $\delta$  (ppm)] was assigned on the basis of data available in the literature.<sup>16</sup> The singlet obtained in the region of 4.98–4.96 ppm ( $\delta$ ) may have been due to the methylene proton of the Ar—CH<sub>2</sub>—N moiety. The signals in the region of 7.35–7.41 ppm ( $\delta$ ) could be attributed to protons of the —NH bridge. The weak multiplet signals (unsymmetrical pattern) in the region of 8.25–8.20 ppm ( $\delta$ ) could be attributed to an aromatic proton (Ar—H). The signals in the range of 9.02–9.06 ppm ( $\delta$ ) could be due to phenolic hydroxyl protons. The far downfield  $\delta$  value for phenolic —OH indicates clearly the intramolecular hydrogen bonding of the —OH group.<sup>17,18</sup> The signals in the range of 9.96–9.92 ppm ( $\delta$ ) could be attributed to protons of —SO<sub>3</sub>H groups.

<sup>13</sup>C-NMR spectra display signals arising from all carbon atoms and hence provide direct information about the carbon skeleton. The <sup>13</sup>C-NMR spectrum of the 8-HQ5-SAMF-1 terpolymer resin is shown in Figure 7, and the peak positions have been assigned according to the literature.<sup>19</sup> The <sup>13</sup>C-NMR spectrum shows the corresponding peaks at 151.00, 121.7, 136.8, 129.2, 134.6, 133.4, 136.2, 137.4, and 149.2 ppm with respect to C<sub>1</sub> to C<sub>9</sub> of the aromatic quinoline ring (Fig. 1). Peaks at 165.0, 163.2, and 161.00 ppm are due to C<sub>1</sub> to C<sub>3</sub> of the aromatic melamine ring. The peak at 115.5 ppm confirms the presence of the —C—NH group in the resin. The peak at 60.2 ppm may be due to the presence of the —C—OH group. The peak at 41.8 ppm may be assigned to the —CH<sub>2</sub> bridge in the terpolymer. The <sup>13</sup>C-NMR spectra clearly established the linear structure of the terpolymer synthesized from 8-HQ5-SA, melamine, and formaldehyde (Fig. 1).

Thermogravimetric analysis is a very useful method for assessing the thermal stability of polymeric resins.<sup>20</sup> Thermogravimetric analysis data for the terpolymer is presented in Table IV, and a thermogram was recorded in the temperature range of 40–800°C; it is shown in Figure 8. The 8-HQ5-SAMF-1 terpolymer resin exhibited a three-stage decomposition process after the loss of one water molecule in the temperature range of 40–120°C (a found mass loss of 4.40% vs a calculated mass loss of 4.46%). The first stage of decomposition started at 120–280°C, with a mass loss of 29.00% found and with a mass loss of 28.50% calculated, which may have been due to the removal of one sulfonic group (—SO<sub>3</sub>H) and hydroxyl group (—OH) attached to the aromatic quinoline ring. In the second stage, the decomposition started at 280–450°C, involving a found mass loss of 60.00% and a calculated mass loss of 59.95%, which corresponded to the mass loss due to the aromatic quinoline ring. The third stage of decomposition started at 450°C, and the complete decomposition of the terpolymer took place at 800°C, involving 99.61% of the mass loss found and 100% of the mass loss calculated for the terpolymer sample. The half-decomposition temperature of the terpolymer resin is also presented in Table IV.

The polymers under study were terpolymers, so it was very difficult to assign their exact structures. However, on the basis of the nature and reactive site of the monomers and taking into consideration the linear structures of other substituted phenol–formaldehyde polymers and the linear branched nature of

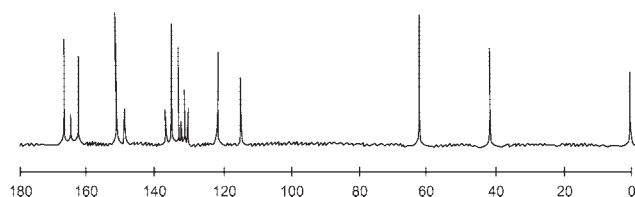


Figure 7 <sup>13</sup>C-NMR spectrum of the 8-HQ5-SAMF-1 terpolymer resin.

TABLE IV  
Thermogravimetric Analysis Data for the 8-HQ5-SAMF-1 Terpolymer Resin

Terpolymer resin	Mass loss (%)						Decomposition temperature range (°C)	Half-decomposition temperature (°C)	Complete decomposition temperature (°C)
	300°C	400°C	500°C	600°C	700°C	800°C			
8-HQ5-SAMF-1	48.59	63.62	75.28	95.25	99.18	99.61	40–800°C	310°C	800°C

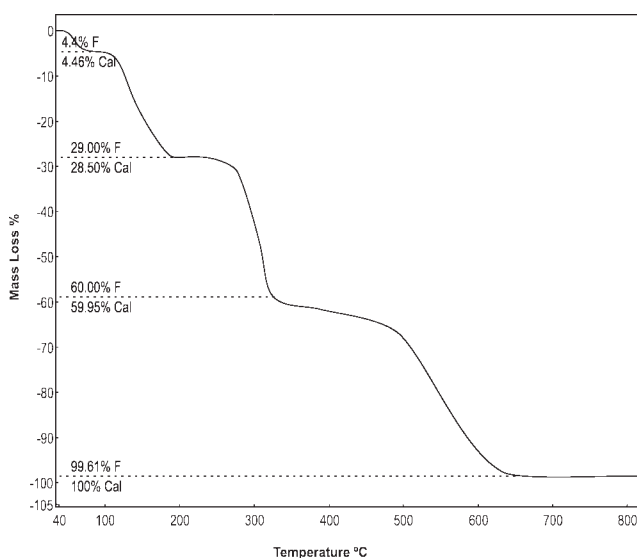


Figure 8 Decomposition pattern of the 8-HQ5-SAMF-1 terpolymer resin.

urea–formaldehyde polymers, we present the most probable structures proposed for the 8-HQ5-SAMF terpolymers in Figures 1 and 2.

### CONCLUSIONS

The terpolymer 8-HQ5-SAMF, based on the condensation reaction of 8-HQ5-SA, melamine, and formaldehyde in the presence of an acid catalyst, was prepared with various molar proportions of the reacting monomers. 8-HQ5-SAMF was yellow, partly soluble in methanol, completely soluble in DMF and DMSO, and insoluble in almost all other common solvents. On the basis of elemental analysis, UV–vis, Fourier transform infrared, and NMR spectroscopy studies, the proposed structures of the 8-HQ5-SAMF terpolymers were determined.

The terpolymer resins can be used as cationic ion exchangers for the separation of transition-metal ions. The resins can also be used for the removal of toxic metals. The resins can form polychelates with certain metal ions. Therefore, they can be used for the separation of metal ions from mixtures.

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