

A Study on Radiation Effects in Acrylonitrile-AMPS Copolymer

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Abstract

Radiation behavior of Acrylonitrile AMPS copolymer has been investigated by Electron Spin Resonance (ESR), Fourier Transform Infrared (FTIR) techniques. Temperature dependent ESR studies indicate that free radicals responsible for irradiated copolymer decay at around 100°C. Presence of macro-radicals and polyene radicals is detected. FTIR spectra of copolymer of unirradiated and irradiated are compared and chemical groups influenced by gamma irradiation are identified. The studies suggest that chain cleavage occur either on AMPS part or AN part depending on the co-monomers. DSC thermograms indicate that chemical transformations occur at higher temperatures.

Keywords: AN AMPS copolymer, gamma radiation, ESR spectrum, FTIR spectrum, DSC spectrum, free radicals

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INTRODUCTION

AMPS homopolymer and copolymers is important class of water soluble polymers. Due to the presence of sulfonic acid groups in their chemical structures, these polymers are usually water soluble and used for different purposes. Though different types of AMPS copolymers like acrylamide (AA)-AMPS, methacrylamide (MA)-AMPS, methyl acrylate-AMPS, have been extensively studied, such studies of acrylonitrile (AN)-AMPS copolymers have not received much attention. Acrylonitrile homopolymer is an interesting molecular system with specific applications. Acrylonitrile used as one of co-monomers and copolymers of Styrene-Acrylonitrile (SAN) copolymer have been investigated. The polar groups of AN are expected to interact with polar groups of AMPS system to produce copolymers with different characteristics. Though synthesis characteristic dielectric properties are investigated, radiation resistance of the system is not previously attempted. The present studies aims at this.

Aggour *et al.* have extensively investigated the thermal properties of AMPS systems [1, 2].

Thermal degradation of AMPS homopolymer has been reported by chromatographic technique. Various types of reaction mechanisms are proposed which led to the formation of cyclic imide groups at higher temperatures. Aggour *et al.* have also compared on thermal stabilities of homopolymer and copolymers of AMPS using thermogravimetric analysis [2]. Among the copolymer and homopolymer of AMPS investigated, thermal stability of copolymer is found to be more than that of homopolymer. Therefore copolymerization has improved thermal stability of AMPS systems.

Thermoluminescence studies of methyl methacrylate AMPS (MMAAMPS) copolymer have been recorded by Rao *et al.* during TSL techniques [3]. The observed TSL glow peak at 85°C is found to shift towards low temperatures, when compared to AMPS homopolymer indicating the presence of bulky groups of MMAAMPS copolymer. The TSL peak is reported to be associated with glass transition temperature of MMA-AMPS copolymer. Further, the glow peak shift to low temperature indicates that T_g of complex is

found to shift towards low temperatures with the increase of irradiation to doses.

Synthesis and characterization of ethyl methacrylate AMPS copolymer has been reported by Rao and Prasad [4]. For this, complex thermo-luminescence studies have been made by Rao [3]. The glow peak temperature is reported to be higher than that of MMA copolymer. Increase in glow peak maxima is due to the increase in molecular weight of copolymer. These authors have calculated trap parameters corresponding to glow peak.

Gamma irradiation effects in methacrylamide AMPS copolymer (MAAMPS) copolymer have been reported by Sridhar *et al.* and Rao *et al.* using ESR and FTIR techniques [5, 6]. These authors have exposed the MAAMPS copolymer (50:50) and (80:20) of compositions to gamma rays to different radiation doses and studied the nature of radiation products formed on radiolysis by electron spin resonance method. Complex ESR spectra are observed for both the copolymers. In order to analyze the ESR spectra, these authors have employed computer simulations. Based on the magnetic parameters during simulation of the ESR spectra, the authors have identified the free radicals formed on radiolysis. The studies indicate that irradiated MAAMPS 55 compose macro-radicals of the type $\sim\text{CH}_2-\dot{\text{C}}\text{H}-\text{CH}_2$ and methyl radicals (CH_3); whereas MAAMPS 82 contain component $\sim\text{CH}_2-\dot{\text{C}}-\text{CH}_2\sim$ and $\dot{\text{C}}\text{H}_3$. Component quintet is assigned to $\sim\text{CH}_2-\dot{\text{C}}-\text{CH}_2\sim$ and component quartet to be due to $\dot{\text{C}}\text{H}_3$ radicals. Therefore for copolymer with low AMPS composition, cleavage occurs on methacrylamide (MA) part producing free radicals on MA part, while increasing the composition of AMPS delete, gamma irradiation induces cleavages on AMPS part of copolymer producing macroradicals $\sim\text{CH}_2-\text{CH}-\text{CH}_2\sim$ and CH_3 (methyl) radicals.

With regard to AAAMPS copolymers, gamma irradiation effects on AAAMPS 73 copolymer have been reported by Reddy *et al.* using ESR technique [7]. Presence of macroradicals has been postulated. Rao *et al.* have reported composition, radiation dose and temperature dependent gamma irradiation effects in

AAAMPS copolymers of 50:50, 60:40 and 70:30 compositions [8]. The ESR spectra observed by these authors is assigned to macroradicals of the type $\sim\text{CH}_2-\text{CH}-\text{CH}_2$ and methyl ($\dot{\text{C}}\text{H}_3$) radicals. The macroradicals might have been formed by cleavage of CONH_2 groups on acrylamide part. Both the events might result in formation of macro-radicals. The free radicals produced in AMPS part of the copolymer produce $\text{CH}_2\text{SO}_3\text{H}$ radicals which convert to methyl radicals (CH_3) during subsequent reaction [8]. Further, it is observed that the increase in composition of AMPS content produces more number of free radicals, when compared to the less content of AMPS. The FTIR studies also indicate a decrease in concentration of sulfonic acid groups on γ -irradiation. Therefore sulfonic acid groups are primary sites of radiation attack, proceeded by cleavage of total side groups on AMPS part of the copolymer producing macroradicals.

ESR spectra of gamma irradiated AAAMPS copolymer of 50:50 compositions have been recorded in the temperature range of 300–420 K. The spectra observed at RT (300 K) possess more number of hyperfine lines, while the spectra at high temperature possess less hyperfine structure. The ESR spectra observed at 420 K is a singlet and beyond this temperature, the ESR signal totally vanished. Disappearance of ESR signal with temperature is thought to be associated with recombination of free radicals.

Indira *et al.* have investigated the influence of bulky gunidine methyl methacrylate (GMA) groups on degradation characteristics of AMPS complexes [9]. They have reported on radiation induced changes on GMAAMPS copolymer by ESR spectroscopy. An ESR spectrum different to that of AAAMPS and MAAMPS is observed. The spectrum is a multiplet with hf splitting of 23G-11G and spread of 120G. Intensity distribution of the spectrum is reported to be complex. Therefore these authors have analyzed the observed spectra of by computer simulation. As such, the spectrum is simulated to be a superposition of macroradicals and component triplet, and a difference singlet. The components are assigned to different types of free radicals produced on gamma irradiation of GMA AMPS

copolymer. The spectrum of higher doses is appeared to be resolved and possesses more number of hyperfine lines, when compared to the low dose spectra. These component assigned spectra are $\sim\text{CH}_2\text{-}\dot{\text{C}}\text{H-CH}_2\sim$. FTIR spectra of unirradiated and irradiated GMA AMPS copolymer have been reported by these authors and the studies indicate the cleavage of sulfonic acid groups. Therefore GMA AMPS copolymer has also undergone radiation induced cleavages like other AMPS system.

Rao *et al.* have used Bloch analysis to evaluate activation energy associated with the decay of free radicals of AAAMPS complex [10]. Since the observed ESR spectra of irradiated polymers/copolymers decayed with temperature, the radical decay is thought to be associated with by the nature of constituent polymer matrix. Polymer with rigid matrix facilitates slow decay; while the amorphous medium/elastic nature of polymer contributes to fast decay of free radicals. Decay in amorphous region is associated with less value of activation energy (E_a) while decay in rigid matrices usually have higher value of E_a . Therefore depending on the E_a value, nature of polymer matrix can be ascertained. Based on these concepts, these authors have recorded the ESR spectra of MAAMPS 55 complex at different temperatures. The hyperfine splitting, intensity distribution and spread of the spectrum gradually decreased with increase of temperature and finally spectrum disappeared above 420 K. Based on line width analyses, the intermediate and fast decay line widths are used to calculate the correlation time (τ). A plot of $\log(1/\tau)$ against inverse of temperature ($1/T$) is drawn. Slope of the straight line directly gives the value of activation energy (E_a) in 40.96 kJ/mole. The E_a value of AAAMPS is found to be 2.86 kJ/mole.

Since temperature radiation dose and composition dependent changes in MAAMPS systems are available in literature, Reddy has reported in this regard [11]. MAAMPS copolymer in different composition is taken and exposed to gamma irradiation to different radiation doses; and ESR spectra are recorded at different temperatures. ESR spectra of MAAMPS 55 possess number of hyperfine lines with complicated shapes whereas

hyperfine structure and spectral resolution increased at lower compositions. ESR spectra at lower composition i.e. MAAMPS 82 is reported to posses contribution from quintet-quartet combination which is assigned to $\sim\text{CH}_2\text{-}\dot{\text{C}}\text{-CH}_2\sim$ and CH_3 radicals assumed to be produced due to cleavage of CONH_2 groups on methacrylamide part. While on the AMPS part, only methyl propane sulfonic acid groups are thought to be cleaved due to gamma irradiation. ESR spectra are recorded at different temperatures for MA55 and MA82 copolymers. The ESR signal intensity is found to fall off with temperature for all compositions. The temperature at which the ESR signal totally vanishes is called radical decay temperature (T_R). The study on unirradiated and irradiated AMPS systems indicates that cleavage of sulfonic acid groups is the main event of gamma irradiation.

Dielectric spectroscopy is used to study molecular relaxation in AN-AMPS copolymer and two peaks at 5 and 100°C are observed [12]. The low temperature peak is correspond to β -relaxation, while the high temperature peak is assigned to α -relaxation of copolymer. Cross linked AN-AMPS hydro-gels are used for removal of cadmium ions [13].

Scope of the Present Studies

Although reports on acrylate, acrylamide copolymers of AMPS system are reported in literature, such studies on acrylonitrile AMPS systems have not been attempted. Acrylonitrile group is considered to have polar nature but it is highly insoluble in water, whereas AMPS groups are also having polar nature but having hydrophilic character. Therefore a complex made up of these two different monomers is expected to have an intermediate character. This type of complex has not been reported in literature.

Modification of chemical structures of polymers, copolymers blends and copolymers is one of the important aspects. In this context, the author attempts gamma irradiation induced modification of ANAMPS copolymer. In order to investigate free radicals produced on gamma irradiation and ascertain chemical changes, electron spin resonance (ESR) and Fourier transform infrared (FTIR) techniques are employed.

Bloch analysis is applied to evaluate activation energy associated with radical recombination occurred due to thermal heating of irradiated ANAMPS copolymer. The values are compared with the other AMPS and AN copolymer systems. In order to apply this analysis, evaluation of accurate values of line widths is essential. For this purpose, computer simulations are employed to simulate the ESR spectra of irradiated complex. The simulation technique includes evaluation of magnetic parameters which includes line width etc. If line widths and hyperfine coupling constants are known; the value of correlation time is calculated. By plotting $(1/T)$ on X-axis and $\log(1/\tau)$ on Y-axis, a straight line is obtained. Slope of the straight line gives the value of activation energy.

EXPERIMENTAL WORK

ANAMPS copolymer in the form of powder is used in the present studies. Synthesis and characterization of complex is described by Narender *et al.* [12]. The copolymer has shown characteristic FTIR absorption bands of both the polymers indicating the formation of complex. Gamma irradiation is performed using a cobalt 60 gamma source which delivers a radiation dose of 15 K. Gy/hr. Gamma irradiations are carried in normal atmospheric conditions at room temperature. Radiation dose administered to the polymer is controlled by time of exposure of the sample. ESR spectra of unirradiated and irradiated complex have been recorded on Varian E line spectrometer operating at X-band frequencies and 100 kHz modulation. The spectrometer is fitted with accessories to record variable temperature ESR spectra. Fourier transform infrared (FTIR) spectra of non-irradiated and irradiated complex have been recorded on PERKIN ELMER spectrophotometer. For this purpose, the complex is made into the pellet form along with potassium bromide having a thickness of 2 mm.

The pellets are placed in the window of spectrometer placed in front of infrared source. Thermograms of complex have been measured on TA instruments. 10 mg sample is kept in aluminium pan and empty pan is kept as a reference. Throughout the experiment, nitrogen gas is flushed in order to avoid oxidation of sample. A heating rate of 10°C/min is maintained.

RESULTS

ESR Studies

Unirradiated copolymer has not shown any ESR signal. ESR spectrum of gamma irradiated ANAMPS copolymer is shown as curve 1 in Figure 1. The spectrum is a multiplet with hyperfine splitting of 23G and spread of 13G. Intensity distribution of spectrum is not followed any particular pattern. The spectra observed at 310, 330, 350 and 370 K are shown as curves 1, 2, 3 and 4 in Figure 2. By heating the irradiated copolymer to a temperature of 400 K, the ESR signal totally vanished. Spectral parameters corresponding to different temperatures are as given in Table 1.

ESR spectrum observed at RT (Curve 1 in Figure 1) is simulated with the component spectra shown in Figure 6. Curve 1 in Figure 2 is component multiplet and curve 2 the component singlet. The superposition of curve 1 and 2 results in curve 2 in Figure 7, i.e., ESR spectrum of ANAMPS copolymer at RT. Similarly, the ESR spectra at other temperatures are (310, 330, 350 and 370 K) also simulated using the component spectra are as shown in Figure 8.

Fourier Transform Infrared Studies

FTIR spectra of non-irradiated and gamma irradiated ANAMPS copolymer are shown as curve 1 and curve 2 in Figure 3. Absorption bands corresponding to various functional groups are as listed in Tables 2 and 3.

Table 1: Spectral Parameters of Irradiated ANAMPS Copolymer at Different Temperatures.

S. No.	Temperature (K)	Number Lines	Splitting (G)	Spread (G)
1	300	6	20	110
2	310	5	20	110
3	330	5	20	90
4	350	4	15	100
5	370	1	Singlet	40

Table 2: FTIR Absorption Bands Observed for Unirradiated ANAMPS Copolymer.

Sl. No	Band Position (cm ⁻¹)	Intensity	Assignment
1	3450	Strong	OH vibration of sulfonic acid groups
2	2933	Medium	CH ₂ /CH ₃ group vibrations
3	2430	Medium	C≡N vibration
4	1650	Medium	CH ₃ asymmetric group
5	1350	Medium	CH ₂ /CH ₃ group
6	1260	Medium	CH ₂ /CH ₃ /CH group
7	1125	Weak	CH ₂ /CH group
8	1050	Strong	SO ₃ H group vibration

Table 3: FTIR Absorption Band Affected by Gamma Irradiation.

S. No.	Absorption Band (cm ⁻¹)	Shift in Peak Position	Interpretation
1	3500	Low wavelength shift of 5 cm ⁻¹	Decrease in intensity of SO ₃ H groups
2	1600	Low wavelength shift of 10 cm ⁻¹	Decrease in C=O functional group
3	1050	Low wavelength shift of 5 cm ⁻¹	Decrease in sulfonic acid groups

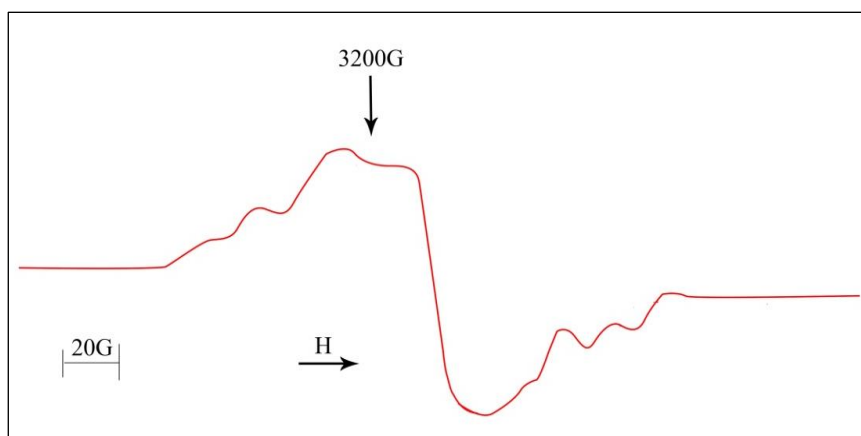


Fig. 1: ESR Spectrum of ANAMPS Copolymer at RT.

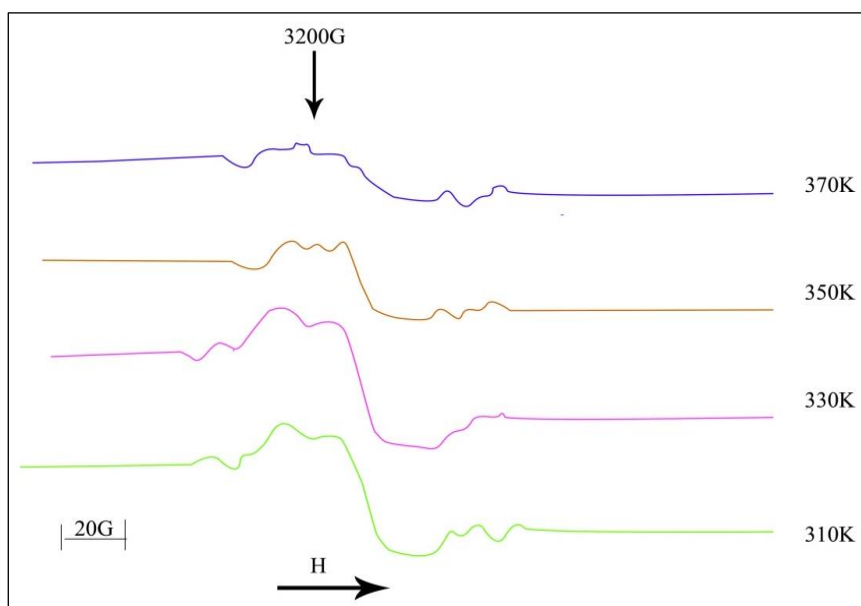


Fig. 2: ESR Spectrum of Irradiated ANAMPS Copolymer at Different Temperatures.

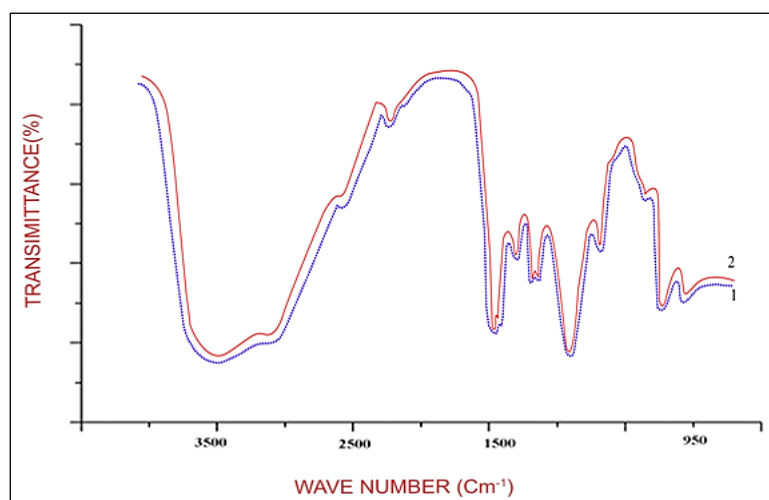


Fig. 3: FTIR Spectrum of ANAMPS Copolymer: (1) Unirradiated; (2) Irradiated.

Table 4: Magnetic Parameters of ANAMPS Copolymer at Different Temperatures.

Sl. No.	Temperature (K)	Relative Intensity Y_{maxi}	Line Width a_i	Centre of Spectrum X_{oi}	Hyperfine Splitting (G)		n_i	m_i
					A	B		
1	300	20	20	3215	23	26	2	5
2	310	10	20	3215	23	26	2	5
3	330	5	20	3215	23	26	2	5
4	350	2.5	20	3215	23	26	2	5
5	370	1.25	20	3215	23	26	2	5

Thermal Studies

Thermograms of unirradiated and irradiated ANAMPS copolymer are shown in Figures 4 and 5. DSC thermograms of unirradiated copolymer show an endothermic peak at 230°C and for the irradiated copolymer, the endothermic peak is at 250°C as observed. Park *et al* [14]. have observed an endothermic peak for PAN fibers at around 270°C and assigned the peak to due to chemical transformations [14]. Since the copolymer contains acrylonitrile groups, the copolymer is also expected to show similar behavior. Therefore the peak is thought to be associated with the chemical transformations occurring at high temperature. Due to crosslinking of copolymer on irradiation, the irradiated copolymer is expected show the peak at higher temperature.

DISCUSSION

ESR spectrum of irradiated AMAMPS at RT is as shown in curve 1 in Figure 1. Since the intensity distribution and hyperfine intervals are not in a particular order, computer simulations are employed to analyze the spectra. The method is based on generation of various component spectra assuming cleavage of various bonds/groups in the copolymer. The

generated component spectra are superposed in different ratios so as to yield the experimental spectrum to fit it in aspects like intensity and line position.

The magnetic parameters indicate the presence of one alpha proton and four equivalent, beta protons. Free radical of this configuration is of the type $\sim\text{CH}_2\text{-CH-CH}_2\sim$. It is formed by the cleavage of side groups from the AMPS part of the copolymer. The other part of the broken chain may give component singlet shown as curve 2 Figure 7. Superposition of component 1, together with component 2 gives experimentally observed spectrum of RT. To have a comparison, the experimental and superposed spectra are shown as curve 1 and curve 2 in Figure 6. The method of simulation of ESR spectra is described by Rao *et al.* and Murthy *et al.* [15, 16]. The other values of magnetic parameters used to simulate the spectra are given in Table 4.

The ESR spectra of irradiated ANAMPS copolymer at different temperatures are also simulated with almost same set of magnetic parameters i.e. n_i and m_i , but remaining values of magnetic parameters have been changed.

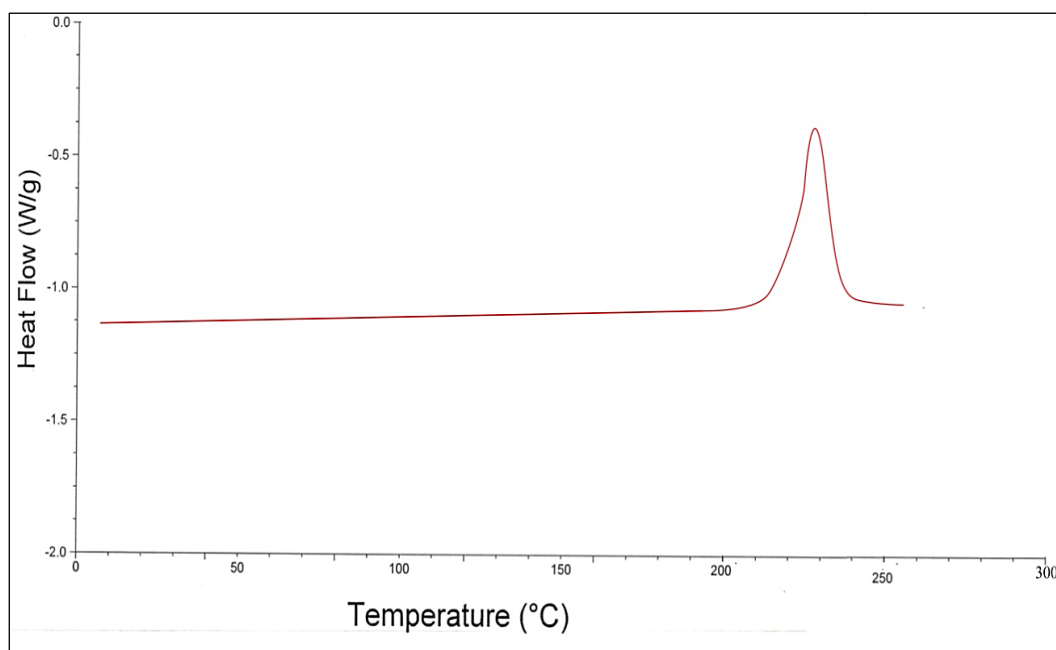


Fig. 4: DSC Thermogram of Unirradiated ANAMPS Copolymer.

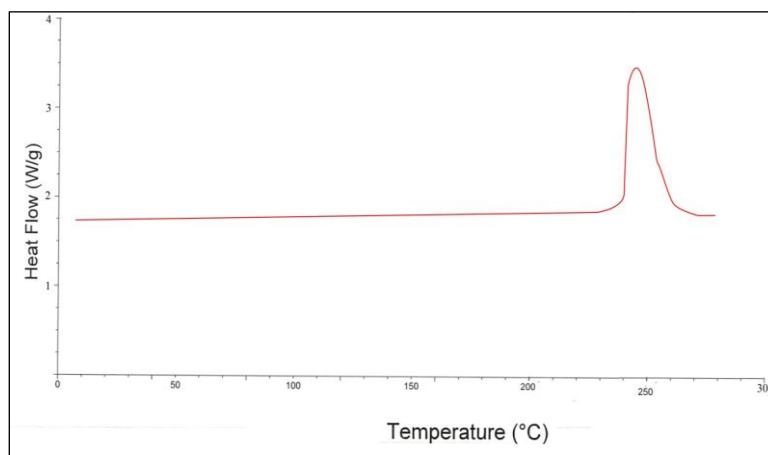


Fig. 5: DSC Thermogram of Irradiated ANAMPS Copolymer.

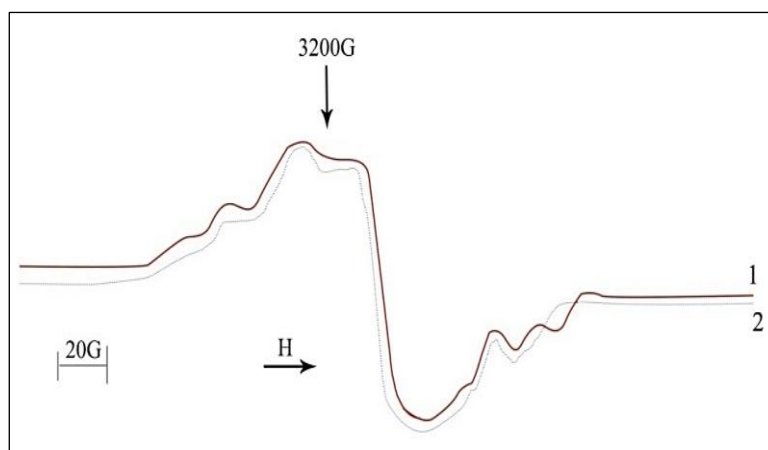


Fig. 6: ESR Spectrum of ANAMPS Copolymer. (1) Experimental; (2) Simulated Superposed Spectrum.

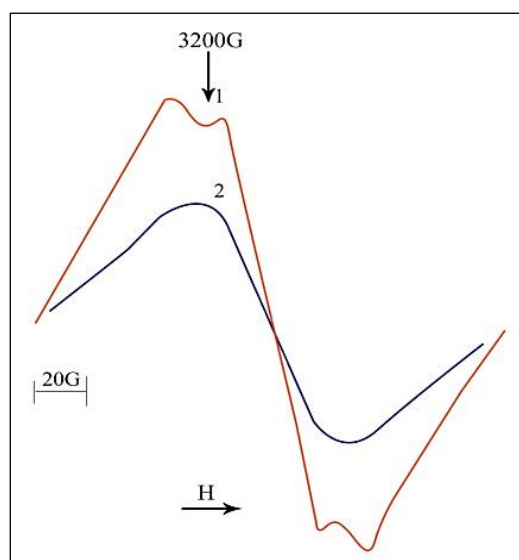


Fig. 7: Component Spectra of Irradiated ANAMPS at RT.

Curve 1: RT; Curve 2: Singlet.

This indicates that chemical constitution of free radical remains almost same, i.e., $\text{CH}_2\text{-}\dot{\text{C}}(\text{CONH})\text{-CH}_2$, but a change in physical substrate of copolymer might have occurred to cause different hyperfine interactions. Due to a variation in hyperfine interactions, the spectra observed at different temperatures are different. The simulated spectra are shown in Figure 8. Curve 1, 2, 3 and 4 represent the component spectra simulated at 310, 330, 350 and 370 K respectively.

The free radicals produced in ANAMPS completely disappear at around 380 K which called as radical decay temperature (T_R).

The value of T_R of ANAMPS is much less than AAAMPS and MAAMPS complexes. As the AN groups are considered to have more polar nature than AA and MA groups, complexation of AN with AMPS resulted a complex with more polar nature. Due to the increase in

polarity, chain flexibility increases causing the chains to react, resulting in faster decay of free radicals.

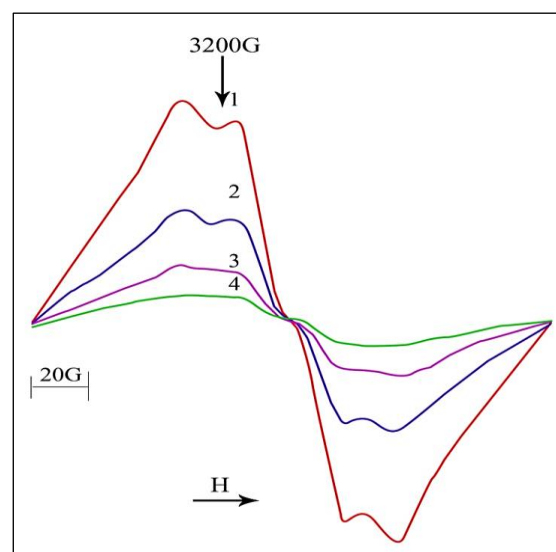


Fig. 8: Component Spectra of ANAMPS at Different Temperatures. Curve 1: 310 K; Curve 2: 330 K; Curve 3: 350 K; and Curve 4: 370 K.

Area under the ESR spectrum represents the free radical concentration. As such, ESR intensities at different temperatures are calculated and a plot of ESR intensity against temperature is drawn as shown in Figure 9.

Activation energy associated with free radical decay is calculated using Bloch analysis. To apply Bloch analysis, the values of correlation times are to be measured at different temperatures. The method of calculating relaxation times is described by Venkateswarao [10]. The values are listed in Table 5. Activation energy is calculated from the slope of $1/T\text{-}\log(1/\tau)$ straight line shown in figure 10. the value is estimated to be around 40.96 kJ/mole.

Table 5: Parameters Used in Bloch Analysis of Irradiated AN ANPS Copolymer.

S. No.	Temperature (K)	$1/T \times 10^{-3}$	Relaxation Time (τ)	$1/\tau$	$\log(1/\tau)$
1	300	3.333			
2	310	3.195	0.00135	73.6	1.867
3	330	3.003	0.0132	75.7	1.879
4	350	2.833	0.0127	78.7	1.896
5	370	2.681	0.0126	79.3	1.899

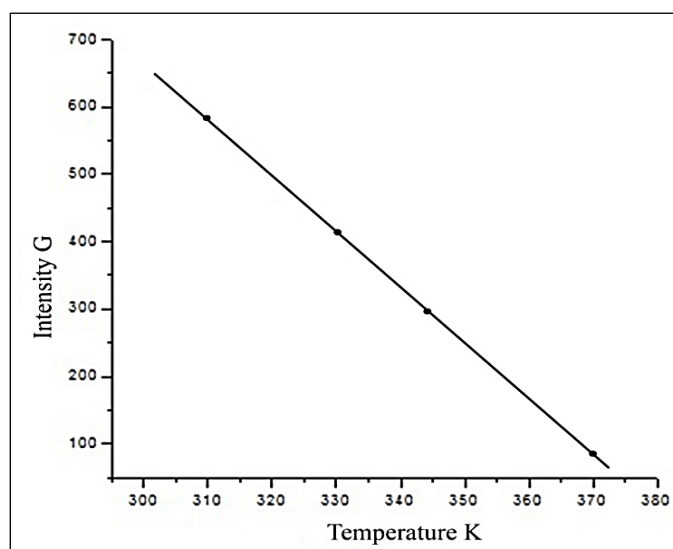


Fig. 9: Variation of ESR Intensity against Temperature.

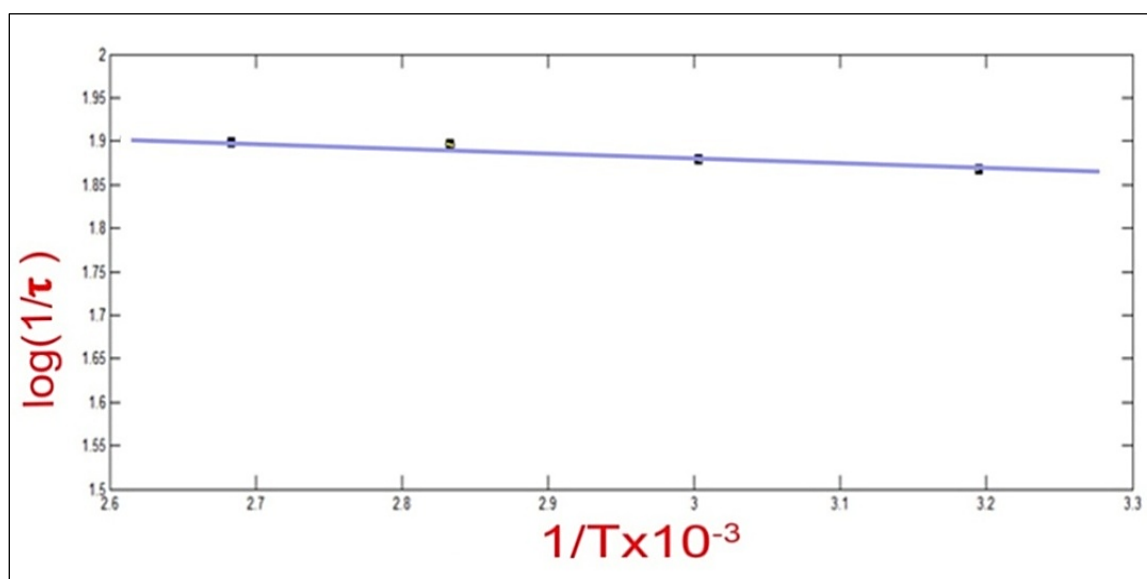


Fig. 10: Plot of Inverse of Temperature versus Log of Inverse of Relaxation Time.

CONCLUSION

Irradiation of ANAMPS copolymer results in formation of macroradicals of the type $\sim\text{CH}_2\text{-}\dot{\text{C}}\text{H-CH}_2\text{-}$, assuming cleavage of side groups on AMPS part. By incorporating high polar groups like nitrile groups, degradation occurs mainly on AMPS part only. When compared to other AMPS copolymers, decay of free radicals occurs at a lower temperature due to the polar nature of both the constituent polymers.

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