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THERMAL DEGRADATION AND KINETIC ANALYSIS OF A SULPHANILIC ACID–MELAMINE–FORMALDEHYDE COPOLYMER FOR HIGH-PERFORMANCE INDUSTRIAL APPLICATIONS

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ABSTRACT

The SAMF copolymer was prepared by the polycondensation of Sulphanilic acid, Melamine, and formaldehyde in a 1:1:4 molar ratio in the presence of 1 M HCl as a catalyst. Structural characterization of the synthesized copolymer was carried out using several physicochemical techniques, namely Ultraviolet–Visible spectroscopy, X-ray diffraction (XRD), Fourier-transform infrared (FTIR) spectroscopy, and proton nuclear magnetic resonance (^{1}H NMR) spectroscopy. The number-average molecular weight of the SAMF copolymer was determined by employing the non-aqueous conductometric titration method. Viscosity studies performed in DMF solvent indicated that the polymer solution exhibited normal behavior. The surface characteristics and morphological features of the copolymer were examined using scanning electron microscopy (SEM). Furthermore, kinetic parameters such as activation energy and reaction order (n), together with thermodynamic parameters including entropy change, Gibbs free energy change, apparent entropy, and frequency factor, were estimated employing the Freeman–Carroll and Sharp–Wentworth methods. The activation energy values derived from both methods were found to be in close agreement, whereas the reaction order was determined to be 0.90.

Keywords: Copolymer, Characterization, Activation energy, Kinetic parameter, Thermal studies.

INTRODUCTION

A copolymer is a macromolecular material formed through the polymerization of two or more distinct monomeric units incorporated within the same polymeric chain. In contrast to homopolymers, which are derived from a single type of monomer, copolymers can be structurally modified to exhibit diverse physical, chemical, thermal, and mechanical characteristics depending on the nature, composition, and sequence distribution of the constituent monomers. Owing to these tunable properties, numerous copolymers have been synthesized, characterized, and utilized in applications such as ion-exchange resins, packaging materials, adhesives, and coating technologies [1–3].

Polymer metal complexes represent an important class of hybrid materials in which metal ions or metal clusters interact coordinatively with functional groups embedded within the polymer backbone. These complexes integrate the adaptability and easy processability of polymeric matrices with the catalytic, electronic, optical, and magnetic attributes imparted by metal species. Due to their multifunctional nature, copolymers and their metal complexes have attracted considerable attention for a broad spectrum of scientific and industrial applications [4–7].

Polymers possess remarkable flexibility, toughness, corrosion resistance, strength, rigidity, and long-term durability, making them highly suitable for diverse applications in aerospace, automotive, electrical and electronic devices, construction materials, sports equipment, and biomedical fields [8–10]. The thermal, physical, chemical, and mechanical characteristics of polymers are significantly influenced by temperature; therefore, the evaluation of their thermal stability is of considerable importance. Thermal analytical techniques such as Differential Scanning Calorimetry (DSC), Thermogravimetric Analysis (TGA), and Dynamic Mechanical Analysis (DMA) are extensively employed to determine transition temperatures, thermal resistance, and degradation behavior of polymeric materials.

Gurnule and co-workers investigated the thermal behavior of a copolymer synthesized from 2-amino-6-nitrobenzothiazole, oxamide, and formaldehyde [11]. Similarly, Nandekar and Gurnule synthesized a copolymer through the polycondensation of salicylic acid, thiosemicarbazide, and formaldehyde, and subsequently evaluated its thermal properties using the Freeman–Carroll and Sharp–Wentworth methods [12-13].

In recent years, bio-based terpolymers have gained substantial scientific interest owing to increasing environmental concerns associated with petroleum-derived plastics and the accumulation of non-biodegradable plastic waste. These polymers, derived from renewable natural resources, are designed to undergo biodegradation over time, thereby offering a sustainable alternative to conventional synthetic plastics [14]. By integrating different monomeric components into a single polymeric framework, they obtained materials possessing both functional versatility and desirable mechanical characteristics. These findings suggest that naturally derived polymeric materials can significantly reduce environmental burden while also providing potential applications in biomedical devices and biodegradable packaging materials [15–16].

Researchers investigated a bio-based plastic incorporated with silver nanoparticles to evaluate its antibacterial efficiency. The developed material exhibited significant antimicrobial activity, particularly against Escherichia coli, indicating that silver nanoparticles can disrupt bacterial cell membranes and inhibit microbial growth. Similar studies have also reported polymeric systems possessing remarkable antimicrobial activity against several pathogenic microorganisms. Gurnule and co-workers synthesized metal complexes of terpolymers and examined their antimicrobial activity against Staphylococcus aureus, Escherichia coli, and Klebsiella pneumoniae [17]. A copolymer based on anthranilic acid, urea, and formaldehyde, which demonstrated promising antimicrobial properties [18]. Gurnule and his research group also developed polymeric materials capable of efficiently removing toxic metal ions from wastewater systems [19]. Furthermore, Chetana Kohad together with W. B. Gurnule reported that specifically engineered polymers exhibited luminescent behavior under particular light irradiation conditions, which was strongly influenced by the molecular arrangement within the polymeric structure [20]. Considerable research efforts are currently focused on the synthesis of advanced polymeric materials possessing enhanced properties and multifunctional applications in everyday life. Among these materials, polymer resins constitute an important class of polymers known for their excellent electrical conductivity, high thermal stability, and superior abrasion resistance, thereby expanding their potential industrial applications [21–23].

The present investigation was undertaken to synthesize a novel copolymer through the polycondensation reaction of Sulphanilic acid, Melamine, and formaldehyde. These monomeric constituents were carefully selected because of their unique structural and functional characteristics. The synthesized copolymer was subsequently characterized using various physicochemical and spectroscopic techniques to elucidate its structural features. In addition, thermal studies were performed to evaluate the thermal stability and decomposition behavior of the material.

Comprehensive characterization further provided valuable insight into the composition, surface morphology, and physicochemical properties of the newly synthesized copolymer.

EXPERIMENTAL METHODS

Materials

From Central Scientific Company in Nagpur came sulphanilic acid - extra pure, by Loba Chemie Pvt. Ltd. - alongside melamine, also extra pure from the same supplier. Formaldehyde arrived too, sourced under Qualigens Thermo Fisher Scientific India Pvt. Ltd. Each substance went straight into use, untouched by additional cleaning steps.

Synthesis of SAMF Co-polymer

A copolymer was synthesized by the polycondensation reaction of Sulphanilic acid, Melamine, and formaldehyde using a 1:1:4 molar ratio, respectively. The reaction mixture was heated in an oil bath maintained at approximately 120 °C with continuous stirring for 5 hr in the presence of 1 M hydrochloric acid as a catalyst. During the course of the reaction, a pale cream-colored rigid copolymer was gradually formed, indicating successful condensation between the monomeric units.

After completion of the reaction, the mixture was poured into ice-cold water and kept overnight to ensure complete precipitation of the copolymer. The obtained product was successively washed with hot water, methanol, and acetone to remove unreacted monomers, low molecular weight fractions, and other soluble impurities. These purification steps effectively eliminated residual side products and unwanted materials. The stepwise synthetic route of the copolymer is illustrated in Fig:1.

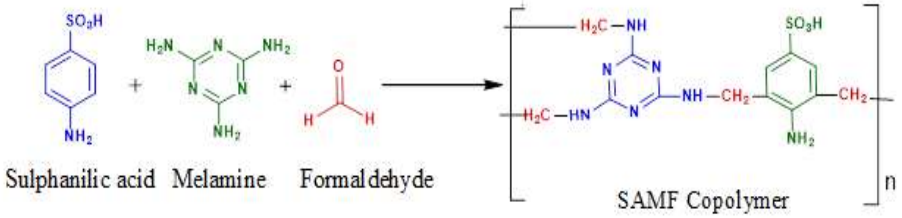

Fig: 1 Formation of SAMF copolymer

Physicochemical Properties and Element Finding of SAMF Copolymer

Elemental analysis of the synthesized copolymer was carried out using a Vario EL III elemental analyzer to evaluate the percentage composition of carbon, hydrogen, nitrogen, and sulfur. The crystallographic characteristics of the copolymer were examined through X-ray diffraction (XRD) analysis using a Bruker D8 Advance A25X diffractometer, which provided insight into the amorphous and crystalline nature of the material.The functional groups associated with the copolymer structure were identified by Fourier-transform infrared (FTIR) spectroscopy using a Thermo Nicolet iS50 spectrophotometer within the wavenumber range of 4000–400 cm^{-1}.

Additional structural characterization was performed through proton nuclear magnetic resonance (^{1}H NMR) spectroscopy employing DMSO-d_6 as the solvent on a Bruker Avance III 400 MHz spectrometer. The surface morphology and microstructural characteristics of the synthesized copolymer were further investigated using scanning electron microscopy (SEM) with a JEOL JSM-6390LV microscope operated at an accelerating voltage of 15 kV. All analytical and spectroscopic studies were conducted at the Sophisticated Test and Instrumentation Centre, Cochin University of

Science and Technology, Kochi, Kerala, India.

RESULTS AND DISCUSSION

The newly synthesized SAMF copolymer was obtained as a light cream-colored solid material. The copolymer exhibited good solubility in dimethyl sulfoxide (DMSO), while remaining insoluble in common organic solvents such as ethanol, methanol, acetone, chloroform, and benzene. This solubility behavior may be attributed to the presence of polar functional groups such as $–NH_2$ and $–SO_3H$, which enhance interactions with highly polar aprotic solvents like DMSO. The synthesized copolymer showed an overall yield of 82%, indicating the effectiveness of the condensation polymerization method employed for its preparation.

Elemental analysis was carried out to determine the percentage composition of carbon, hydrogen, nitrogen, and sulfur present in the copolymer. The experimentally obtained values were found to be in close agreement with the theoretically calculated data, confirming the successful incorporation of all three monomeric units into the polymeric backbone. The empirical formula $C_{13}H_{14}N_7SO_3$ corresponded well with the proposed repeating unit structure of the SAMF copolymer, having a molecular weight of approximately 348 g mol^{-1}. These findings further indicate that the synthesized copolymer retained its expected structural integrity during the polymerization process [24–25].

Table 1. Percentage Elemental Composition and Empirical Formula of SAMF Copolymer

Synthesized polymer	Experimental and Theoretical Carbon (%)	Experimental and Theoretical Hydrogen (%)	Experimental and Theoretical Nitrogen (%)	Experimental and Theoretical Sulfur (%)	**Empirical Formula of Copolymer Repeat Unit**	Empirical Formula Weight (g mol^{-1})
SAMF	37.95 (42.62)	2.79 (3.82)	23.30 (26.77)	4.05 (4.96)	$C_{13}H_{14}N_7SO_3$	348

Evaluation of Molecular Weight through Non-Aqueous Conductometric Titration

Chemists measured the average size of molecules in the new SAMF material by a special electrical test in alcohol-free liquid. They picked DMF to dissolve the sample that allows tracking during analysis clearly. A solution made of pure ethanol and potassium hydroxide at one-tenth molarity was added slowly. This slow addition revealed how much base was needed to shift the balance. From beginning to end, each step leaned on electric signals changing as drops flowed into the stirred container. What emerged was Mn - an outcome pulled directly from watching resistance drop moment by moment.

In this method, the electrical conductivity of the solution was continuously monitored during the gradual addition of alkali. The conductometric titration curve obtained by plotting conductance against the volume of KOH exhibited two distinct inflection points, corresponding to the initiation and completion of the neutralization process. The degree of polymerization (DP) was determined from the quantity of KOH consumed and the known weight of the copolymer sample. Subsequently, the number-average molecular weight (Mn) of the copolymer resin was calculated using the degree of polymerization and the molecular weight of the repeating unit according to the following relation:

$$Mn = \frac{m\ x\ 1000}{\Delta V\ x\ N}$$

Where

- m = mass of polymer sample (g),
- ΔV = volume difference between first and last break (mL)
- N = normality of KOH solution (eq/L).

This technique provides a reliable estimation of Mn since conductometric titration in a non-aqueous medium eliminates complications arising from polymer insolubility and side reactions in aqueous systems.

$$DP = \frac{Total\ miliequivalent\ of\ the\ base\ required\ for\ last\ break}{Miliequivalent\ of\ the\ base\ required\ for\ first\ break}$$

Mn = DP x Empirical formula weight of the repeating unit

Table 2: Determination of Molecular weight (Mn) of SAMF copolymer

Copolymer	Initial Stage of Neutralization	Completion of Neutralization Process	Degree of polymerization	Empirical formula weight	Number average molecular weight
SAMF	36	956	26.5	348	9222

Fourier Transform Infrared (FTIR) Spectral Analysis

The FTIR spectrum of the synthesized SAMF copolymer was recorded at the Sophisticated Test and Instrumentation Centre, Kochi, Kerala, India, using a Thermo Scientific Nicolet iS50 FTIR spectrophotometer. Spectral analysis was carried out within the wavenumber range of 4000–400 cm^{-1} to identify the characteristic functional groups and to verify the structural features of the synthesized copolymer. The obtained FTIR spectrum displayed several prominent absorption bands, confirming the successful incorporation of Sulphanilic acid, Melamine, and formaldehyde units into the copolymer backbone:

- A broad peak at 3201 cm^{-1} was observed which indicates the presence of $–NH_2$ groups from sulphanilic acid. This peak is affected by hydrogen bonding inside the polymer [26].
- A peak near 2111 cm^{-1} is related to C–H bonds of $–CH_2$ groups, shows the formation of methylene linkages during the process of polymerization [27] .
- The sharp peak at 1613 cm^{-1} confirms the presence of C=N bonds from the melamine unit in the copolymer [28].
- Peaks at 1007, 886, and 812 cm^{-1} indicate the presence of the triazine ring, confirming melamine incorporation into the polymer structure.
- The peak at 1494 cm^{-1} is due to vibrations of $–CH_2$ groups, again supporting the formation of methylene bridges from formaldehyde.
- A strong peak at 1032 cm^{-1} corresponds to the S=O bond stretching of sulphanilic acid.

- Peaks at 1148 cm^{-1} and 1095 cm^{-1} are linked to sulfonate group vibrations [29].

Among those distinct peaks, each tied to a specific building block - Sulphanilic acid, melamine, yet also formaldehyde - their joint appearance confirms integration within the structure. Evidence from the readings in Table 3 backs up the existence of anticipated chemical features; meanwhile, what the full infrared pattern looks like appears in Figure 3.

Table 3: Fourier Transform Infrared spectral analysis of SAMF copolymer

Observed frequencies (cm^{-1})	Assigning groups	Expected frequencies (cm^{-1})
3270 (b,st)	N-H stretching for amino group	3500-3200
2111 (m)	-C-H stretching for CH_2 group	2500-2200
1613 (m)	C=N stretching of melamine ring	1600-1500
1494	-CH_2 bending and wagging	1500-1300
1032	S=O symmetric stretching	1080-1040
1123,1176	S=O Asymmetric stretching	1230-1170
812,886,1007	Triazine ring of melamine	1000-800

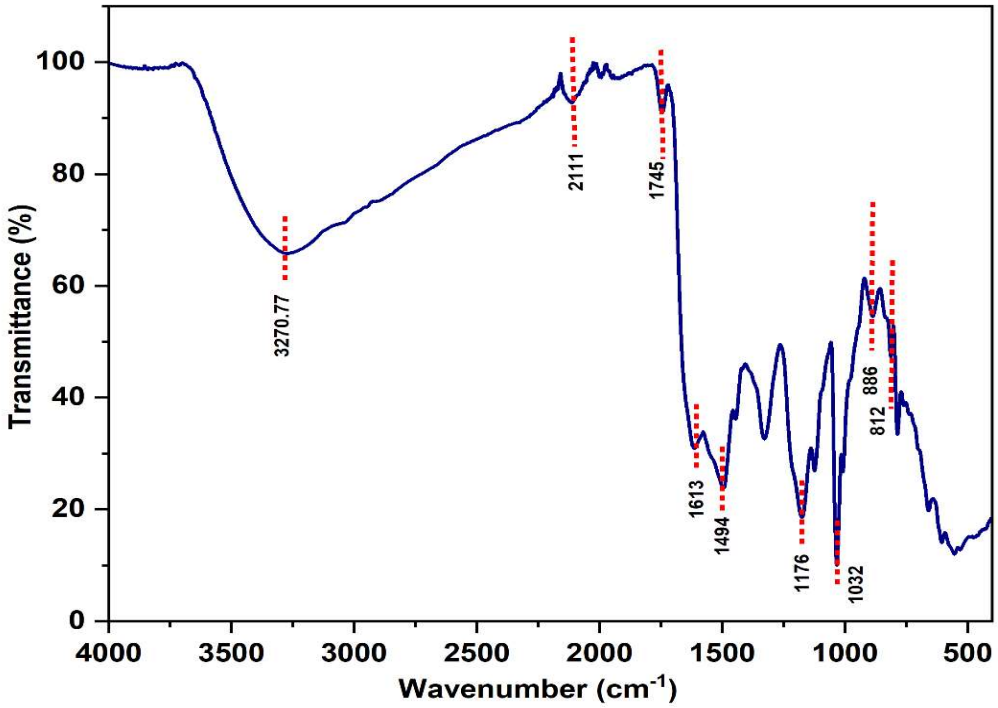

Figure 2: Fourier Transform Infrared spectra of SAMF copolymer

X-ray Diffraction (XRD) Analysis

The X-ray diffraction (XRD) analysis of the synthesized SAMF copolymer was carried out to investigate its structural organization and degree of crystallinity. The obtained diffraction pattern indicated the presence of both crystalline and amorphous regions within the copolymer matrix, suggesting a partially ordered molecular arrangement. The appearance of distinct diffraction peaks

along with broad diffuse regions confirmed the coexistence of ordered domains and disordered polymeric networks in the material.

A broad halo observed in the lower diffraction angle region is characteristic of the amorphous nature of the copolymer, indicating irregular chain packing and the absence of a well-defined crystalline lattice. Such disordered arrangements generally arise from cross-linked polymeric structures and heterogeneous chain alignment, where uniform molecular orientation is restricted. These findings demonstrate that the SAMF copolymer possesses a semi-crystalline nature with both structured and randomly oriented regions distributed throughout the polymer framework.

Among these signals, distinct spikes stood out - narrow yet intense - marking spots where the material formed rigid, structured zones. Where chains lined up neatly, repeating patterns emerged, possibly guided by how triazine rings settled into place. Arranged segments could just as well come from Sulphanilic acid groups fitting tightly together, creating pockets of order within the bulk. A peak here, a gentle slope there - Figure 3 reveals how the SAMF copolymer mixes disordered regions with sharp ordered zones through its XRD signature [30–31].

Half the peak width looks wider, which hints at some parts of the polymer holding structure while others do not. Sharp spikes show areas locked in place. A spread-out central bump means much of it stays loose, so the mix ends up neither fully solid nor entirely random. This pattern points toward a blend rather than pure form. Crystalline pockets sit inside an otherwise chaotic polymer framework, according to XRD data on the SAMF copolymer. Because of this mix, stiffness comes from structured zones while bendable qualities emerge from messy ones.

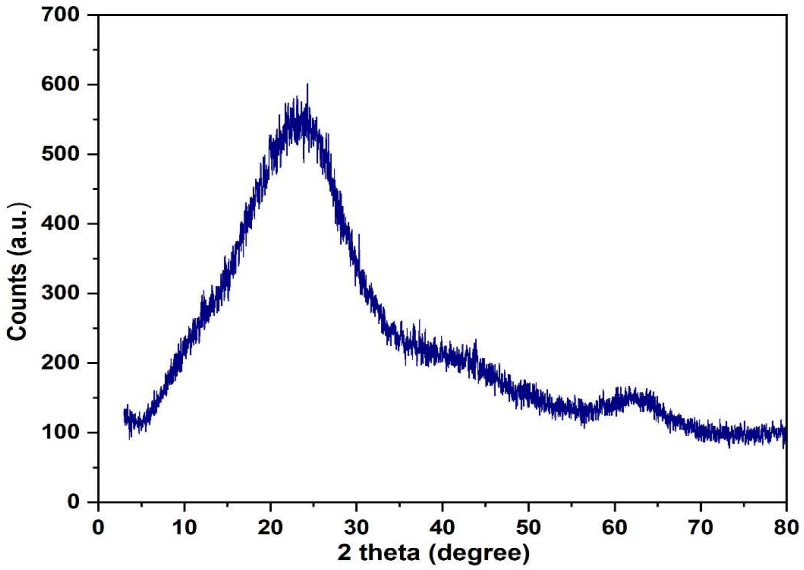

Figure 3. XRD Spectrum of the Synthesized SAMF Copolymer

Scanning Electron Microscopy Analysis

The SEM images of the SAMF copolymer were recorded at different magnifications ranging from ×500 to ×6000, and the images are presented in Figure 4. The surface morphology observed

through SEM analysis provided useful information about the internal arrangement and structural nature of the copolymer material. The SEM micrographs showed that the surface of the copolymer was not smooth or uniform in appearance. Instead, it contained round-shaped clusters, rough edges, irregular patches, and small pores distributed over the surface.

These observations indicate that the copolymer possesses both crystalline and amorphous regions within its structure. Some areas appeared more ordered and tightly packed, while other regions were loosely arranged and less organized. The presence of small pores and uneven gaps may be due to irregular packing of polymer chains during the polymerization process. Fringe-like patterns observed in some regions suggested the existence of boundaries between ordered and disordered zones inside the material.

At higher magnifications such as ×3000 and ×6000, the SEM images clearly revealed fine surface textures and closely packed regions along with shallow depressions. The partial crystalline behavior may be attributed to the regular arrangement of melamine triazine units and methylene bridges formed during the reaction with formaldehyde. The coexistence of rigid and flexible regions gives the copolymer both structural stability and flexibility.

Overall, the SEM analysis confirmed that the SAMF copolymer has a heterogeneous surface morphology with both ordered and disordered structures. These SEM findings were found to be in good agreement with the results obtained from XRD and FTIR studies, further supporting the successful formation of the copolymer structure.

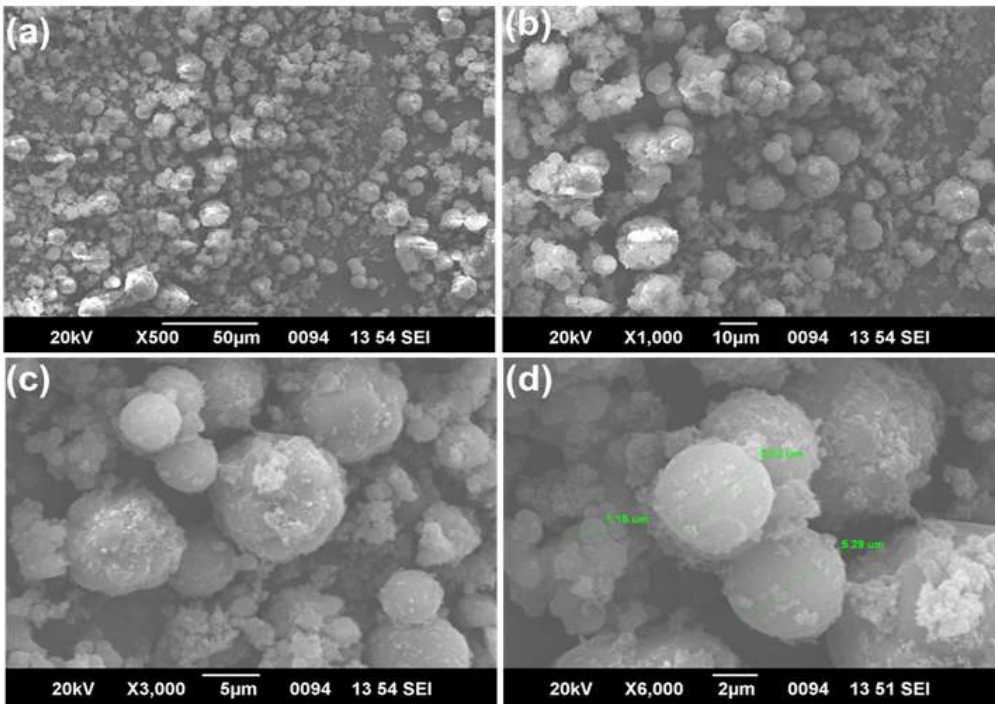

Figure 4: a) SEM image at 50µm (b) SEM image at10 µm(c) SEM image at 5 µm (d) SEM image at 2 µm

Structural Elucidation by ^{1}H NMR Spectroscopy

The ^{1}H NMR test was used to study the structure of the SAMF copolymer and understand how hydrogen atoms are arranged in it. The analysis was done using a Bruker Avance III machine at STIC, Kochi, Kerala, in DMSO-d_6 solvent. Previous research studies were also used to help interpret the results.The main observation in the ^{1}H NMR spectrum are as follows:

- Signals between 7.11–7.75 ppm show the presence of benzene ring hydrogen from Sulphanilic acid in the polymer chain. These peaks help confirm how the aromatic rings are arranged in the structure.
- A weak signal near 9.2 ppm comes from the $–SO_3H$ group of sulfonic acid. This happens because the sulfonic group strongly attracts electrons and also forms hydrogen bonding inside the polymer.
- A peak at 1.96 ppm indicates the presence of $–CH_2–$ (methylene) bridges connecting aromatic rings with nitrogen atoms. This confirms that formaldehyde reacted successfully with Sulphanilic acid and melamine during polymer formation.

The ^{1}H NMR results confirm that the SAMF copolymer was formed successfully. The data supports the presence of sulphanilic acid, melamine units, and methylene bridges in the structure. These findings also match well with the FTIR and XRD analysis results, proving that the designed polymer structure was achieved.

Table 4: ^{1}H NMR analysis for SAMF copolymer

Observed chemical shift value in SAMF	Proton Environment Assignment	Characteristic Chemical Shift Range (δ, ppm)
2.1	Methylene protons of Ar-CH_2 moiety	2.0-3.0
3.1	Ar-CH_2-N moiety for methylenic proton	3.00-3.5
5.4	Proton in N-H bridging	5.00-8.00
7.1-7.75	Ar-H aromatic ring proton	6.0-8.5
9.2	Proton of acidic linkage SO_3H group	8.00-10.00

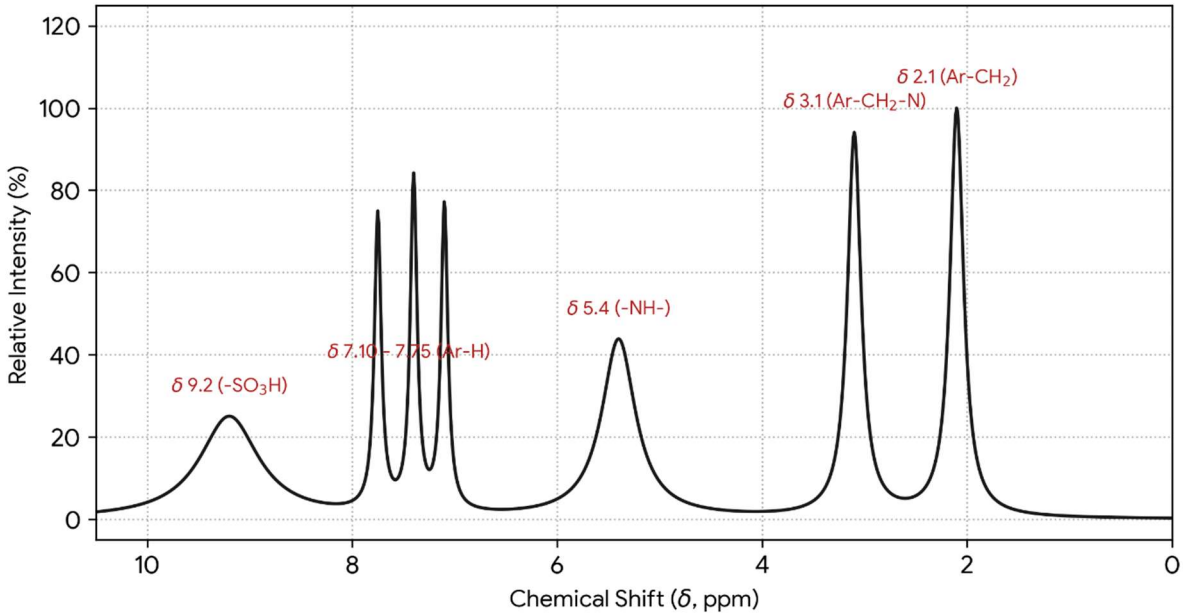

Figure 5: ^{1}H NMR spectra of SAMF Copolymer

Thermo-kinetic behavior of SAMF copolymer

Heat movement analysis of SAMF plastic material is important to reveal where it can be used. The point at which it turns glassy (Tg), along with the temperture where it falls apart (Td), shows if it survives manufacturing, keeps shape, works in glues, circuit parts, tough coatings [36–37]. The SAMF copolymer exhibited good thermal stability, as confirmed by TGA, which showed a multi-step degradation pattern associated with its aromatic and cross-linked structure. The relatively high decomposition temperature reflects the strong interactions imparted by sulphanilic acid and melamine units.

Thermogram is obtained by heating copolymer sample at 20^0C per minute in air and thermogram of SAMF copolymer is depicted in figure-6 with three degradation steps. Initial mass loss occur at the temperature range 60^0C-100^0C (4.91% calculated and 5.17 found) may be due to loss of water molecule from the polymer sample. The first stage degradation starts from 360^0C to 560^0C (31.42% calculated and 32.5% found) which may be due to loss of side chain SO_3H, NH_2 groups. Second stage degradation takes place from 560^0 C to 690^0C (66.9% calculated and 67.04% found) and half decomposition temperature is found to be 450^0C.

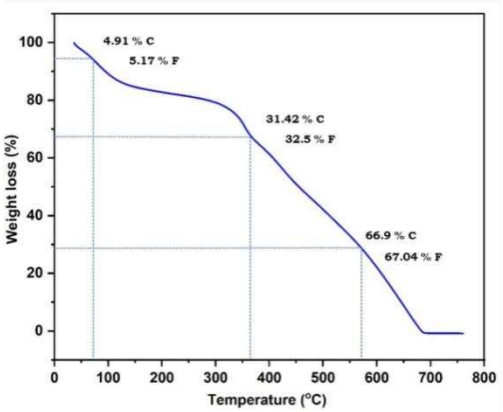

Figure 6: TGA curve of SAMF copolymer

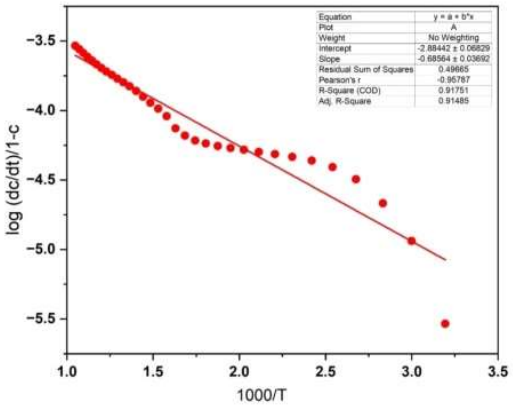

Figure 7: S-W plot of SAMF Copolymer

Sharp-Wentworth Method

The Sharp–Wentworth method provides an expression for calculating the activation energy (Ea), entropy change (ΔS), and free energy change (ΔF). A plot of log[(dc/dt)/(1–C)] versus 1/T yields a straight line, from which the activation energy is determined from the slope (Figure 6). Sharp-Wentworth expression is given as:

$$\frac{\log\left(\frac{dc}{dt}\right)}{(1-C)} = log \propto/\beta = Ea/2.303RT$$

Where β is the linear heating rate
dc/dt is rate of change of weight with temperature
T is absolute temperature

Feeman - Carroll Method

The Freeman–Carroll method is a kinetic analysis technique widely used in the thermal analysis of polymers, particularly for studying their decomposition behavior through thermogravimetric analysis (TGA). This method enables the determination of key kinetic parameters associated with polymer degradation.These parameters indicate the thermal stability of a polymer and its decomposition behavior under heat. Starting at high heat, weight shifts show up through TGA traces. The changes per degree matter the most as a slice of breakdown links directly to how fast mass drops. Temperature steps reveal speed patterns hidden in the slope.

The Freeman–Carroll equation is:

$$\Delta \log\left(\frac{dw}{dt}\right) \div \Delta logWr = \left[-\frac{Ea}{2.303}\right] \times \frac{\Delta\, 1}{T} \div \Delta \log Wr + n$$

where:

- dw/dt = fraction decomposed (Rate of change of weight with time)
- Wr = difference between weight loss at completion of reaction at time ‘ t’
- T = temperature (K)
- R = gas constant

- n = order of reaction
- Ea = activation energy

By plotting a graph between Δlog(dw/dt)/ΔlogWr and 1/T logWr, the slope and intercept of the line provide the order of reaction and activation energy, respectively. For the SAMF copolymer [40-43] the activation energy was found to be 12.27 kJ mol^{-1}. Using thermodynamic data and applying the Sharp–Wentworth method, the calculated activation energies (Ea), presented in Table 5, were in good agreement with those obtained from the Freeman–Carroll method (Figures 8 and 9). The low value of the frequency factor indicates a slow decomposition process, which is further supported by the negative entropy change [38–39]. However, a few data points deviated slightly, suggesting that the reaction does not strictly follow first-order kinetics [44-47].

Table 5. Kinetic parameter of SAMF copolymer

Copolymer	Activation Energy (KJ/m)		Entropy change	Free energy change	Frequency factor	Apparent entropy	Order of reaction
	S-W	F-C					
SAMF	13.12	12.27	1.078 J	11.78 kJ	229.3	-61.7602	0.90

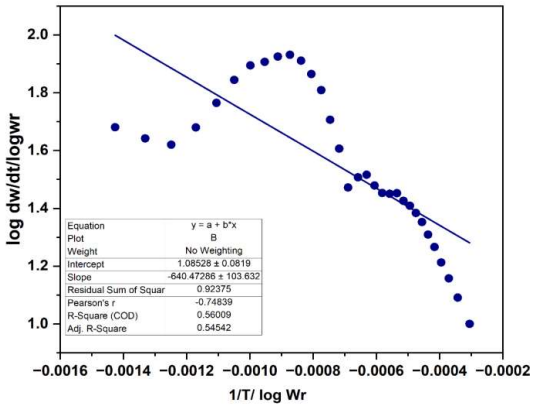

Figure 9: F-C plot of SAMF copolymer

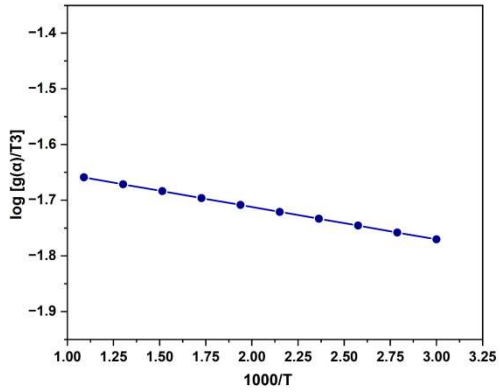

Figure 8: F-C plot of SAMF copolymer

CONCLUSION

The SAMF copolymer was synthesized through the polycondensation of Sulphanilic acid, Melamine, and formaldehyde in the presence of 1 M HCl as a catalyst at 120 °C for 5 h. The successful formation of the copolymer was confirmed through elemental analysis, FTIR spectroscopy, and ^{1}H NMR spectral studies. Surface morphological analysis by SEM revealed the presence of both ordered and disordered regions within the polymer matrix, which was further supported by the XRD results indicating the semi-crystalline nature of the copolymer. Collectively, these characterization techniques provided valuable insight into the relationship between the structural features and physicochemical behavior of the synthesized material, thereby highlighting its significance in polymer science research. Thermogravimetric analysis (TGA) demonstrated that the SAMF copolymer possesses appreciable thermal stability. The activation energy values calculated using different kinetic methods were found to be in close agreement with one another, confirming the reliability of the thermal analysis. Furthermore, the low frequency factor and negative

entropy values obtained from the Freeman–Carroll method suggested that the thermal degradation process of the copolymer proceeds through a comparatively slow reaction mechanism.

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