ISSN: 2096-3246
Volume 57, Issue 02, December, 2025

THERMAL DEGRADATION STUDIES AND ANTIMICROBIAL ACTIVITIES OF NOVEL SASF COPOLYMER FOR INDUSTRIAL APPLICATIONS

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Abstract

SASF copolymer was synthesized by polycondensation of Sulphanilic acid (0.2 mol), Semicarbazide (0.1 mol) and Formaldehyde (0.3 mol) in 500 mL RB flask using 1M hydrochloric acid as a catalyst. This mixture was kept for about 5 hours at 120^0C which finally converted into resinous product. To characterize the synthesized copolymer using different spectroscopic tool like FTIR spectra, proton NMR spectra, XRD, SEM, and TGA. Resulting product was then studied by various analytical techniques such as Elemental composition, molecular weight determination by non-aqueous conductometry titration method and solubility in organic and inorganic solvents. The morphology of copolymer was examined by scanning electron microscopy (SEM). The studies have been additionally reached out to non- isothermal thermogravimetric examination (TGA) for assurance of their method of disintegration and relative thermal stability. Actuation energy (Ea), order of reaction (n), and frequency factor (z) were determined by Sharp-Wentworth and Freeman-Carroll techniques. Actuation energy determined by Sharp Wentworth and Freeman-Carroll techniques are in close concurrence with one another. Activation energy of SASF copolymer was found to 15.6 kJ/mole and order of reaction was estimated as 0.82. SASF copolymer seen to be thermally stable and potent antimicrobial activity against four parasites.

Keywords: Copolymer, Characterization, Thermal parameter, antimicrobial action Polycondensation.

Introduction

Numerous infectious diseases are found as a result of increased action of microbes which in ultimately affect the safety and wellbeing of human life. Many infectious diseases remain untreated though substantial progress is observed in antimicrobial drugs. Antimicrobial polymers have adapted considerable attention in both academic as well as industrial research[1-3]. Along with it offers promising antimicrobial strategy for fighting against pathogens. Many proficient polymers acting firmly as antimicrobial have been reported. The number of FDA- approved polymers has risen in significant way over the past decades [4]. Due from past three decades above researches highlights the recent advances in antimicrobial polymers. Antimicrobial polymers can be classified as either passive or active based on their mechanism of action. From a material perspective, they are further

categorized into destined or leaking antimicrobials. These polymers find applications across diverse fields, including the medicine, food, and textile industries[5-8].

Thermal analysis of any polymer as a function of temperature under controlled heating condition is essential for the mode its applicability in diverse field such as lubricant, adhesives, ion exchange materials, catalyst, fire resistant materials and semiconductor[9-12]. Waste water analysis is one of the major issues in relation to toxic effect of pathogen on human being, agriculture and animals. Gurnule and his team synthesized copolymer using monomers and selectively remove toxic metal ion from wastewater[13]. Ion exchange process shows a vital character in wastewater purification, removal of specific metal ion as well as various industrial applications. Mandavgade S. K. studied ion exchange properties of copolymer 8-HQ-5-SACF-catechol-formaldehyde by employing Batch equilibrium method [14]. Due to special ability of thermally stable polymers to maintain structural integrity and performance under high temperature they have established as an important class of materials in the polymer discipline. A novel copolymer based on 2, 4-dihydroxypropiophenone, 1,5-diaminonaphthalene, and formaldehyde was synthesized by Gurnule et al [15] in which Thermal and kinetic parameters were examined along with synthesis.

The three-dimensional assembly materials that comprise hydrophilic polymer chains are known as hydrogel. With the important properties these are also useful in several fields. Many applications of hydrogel fail in various fields due to its low thermal stability, poor mechanical strength and low strain. Hybrid hydrogel designed with enhanced and exclusive characteristic has a boon with polymeric network and highly hydrated properties[16-17]. Various types of nanoparticles along with different polymeric networks form Nano composite hydrogels such as Carbon-based nanomaterial. Many Nano composite hydrogels has been established by the polymer Nano technology. Due to its biocompatible and ecofriendly assets it is used in biomedical fields such as drug delivery, antimicrobial applications and tissue engineering [18-19]. As useful applications against the microorganisms, also being ecofriendly and non-toxic nature Ag nanoparticles can be directed to biomedical field. Due to said properties these are used in wound dressing and antibacterial actions [20].

An antimicrobial polymer is a special type of material that can stop germs like bacteria and fungi from growing on surfaces. It works because it contains certain chemical groups that can attack and damage the outer covering of these germs. When the covering is damaged, the germs cannot grow or spread. In 2025, Hatu Gmedhin and his team [21] developed polyacrylamide-based polymers with the Influence of end-group functionality and composition on the antifungal activity. Today, fungal infections that do not respond to medicines are becoming a serious health problem around the world. Because of this, scientists are working on new synthetic polymers that can act as antifungal materials. Within above context, copolymer resins that represent a remarkable class of polymers widely used for their diverse applications like photoluminescent material and thermal resistivity [22].

In recent studies we marked to produce novel copolymer SASF (Sulphanilic acid, Semicarbazide and Formaldehyde) by means of selected monomers. A number of analytical and spectral tools were applied for characterization. An antimicrobial activity of copolymer was established using Agar-Agar method and thermal degradation pattern was studied by Sharp-Wentworth and Freeman-Carroll Method.

Experimental

Materials

Sulphanilic acid and Semicarbazide utilized in the current examination of pure grade immaculateness were bought from Sigma Aldrich Chemicals. Formaldehyde (37%) was bought from S.D. Fine Chemicals, India. Every one of the pre-owned solvents like N,N-dimethylformamide, dimethyl sulfoxide, tetrahydrofuran, DMF, $(CH_3)_2CO$, and diethyl ether were secured from Merck, India.

Synthesis of SASF Copolymer

SASF copolymer was synthesized by polycondensation polymerisation of sulphanilic acid, semicarbazide with formaldehyde in 2M HCl as catalyst in the molar proportion 3:1:5 at temperature 120°C in an oil bath with incidental shaking, to guarantee exhaustive blending for around 4 hrs. The brown coloured shaded resinous mass was obtained. The resinous mass obtained was washed with hot refined water and methanol to eliminate the overabundance of sulphanilic acid formaldehyde copolymer which may be available alongside SASF copolymer. The appropriately washed copolymer dried, powdered and afterward extracted with diethyl ether and afterward with petrol ether. The brown coloured shading resinous item was promptly taken out from the flask as before long as response period was finished and afterward cleaned. The response and proposed scheme of reaction of SASF is displayed in Fig. 1.

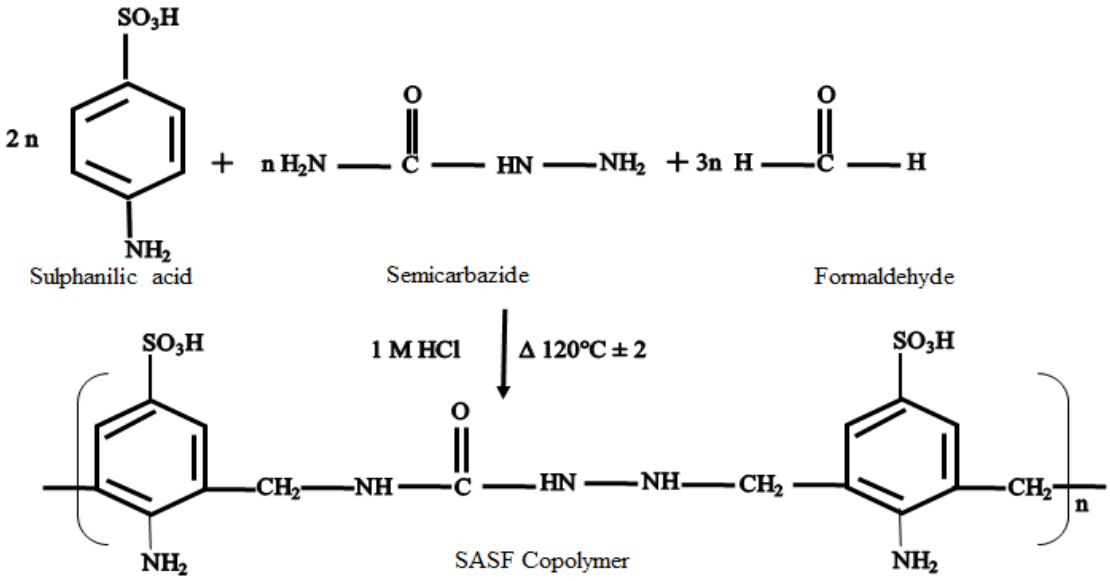


Figure 1: SASF copolymer preparation

Analytical and Physico-Chemical Studies

The basic investigation was done on Elemental Vario EL III Carlo Erba 1108 basic analyser instrument. The electronic ingestion spectra (UV-noticeable) of the copolymer in DMSO were recorded on twofold bar spectrophotometer in the scope of 200-800 nm. Infrared spectra of copolymer were completed in najol reflect on Perkin-Elmer-Spectrum RX-I, FTIR spectrophotometer in KBr pellets in the scope of 4000-500 cm^{-1}. ^1H NMR range was recorded on Bruker Advance-II 400 MHz NMR spectrophotometer utilizing DMSO-d_6 as a dissolvable solvent.

The surface examination was performed utilizing checking electron magnifying lens at various amplifications. SEM has been filtered by a JEOL JSM-6380, a scientific checking electronic magnifying lens. The TGA of the copolymer has been completed utilizing Perkin Elmer jewel TGA/DTA analyser. All the logical and unearthly examinations for the recently orchestrated copolymer were done at modern logical instruments office SAIF, Cochin University, Cochin, India.

Thermal investigations

Thermal examination technique is related with an adjustment of weight as for temperature. Warming is performed under stringently controlled conditions and can uncover changes in structure and other significant properties of the material being considered. In non-isothermal or dynamic TGA, the example is exposed to conditions expansion in temperature at straight rate. The thermogravimetric examination was acted in air with warming rate at 10^0C min^{-1} utilizing 5-6 mg of tests in platinum pot from temperature of 40^0C to 800^0C and thermogram is recorded for SASF. With the assistance of thermogravimetric data, the thermal activation energy (Ea) and order of reaction (n) determined. Additionally, the other thermodynamic boundaries such as entropy change (ΔS), apparent entropy change (S*) free energy change (ΔF) and frequency factor calculated.

Theoretical considerations

Thermogram was deciphered and examined to get data about the rate weight reduction at various temperatures which gives data about example synthesis, item shaped in the wake of warming. Dynamic boundaries not set in stone utilizing Sharp-Wentworth and Freeman-Carroll techniques as follows,

Sharp-Wentworth technique using the condition inferred by Sharp and Wentworth [20],

LogdC/dT 1−C=log(A/β)- Ea 2.303R.1 *TT* ……. (1)

where, dC/dT= rate of fraction of weight with change in temperature

β = linear heating rate dT/dt.

The graph of log dC/dT 1−C versus 1/T has been plotted. The graph is a straight line with activation energy (Ea) as incline and an as catch. The straight relationship affirms that the expected request (n =1) is correct.

Freeman-Carroll method

The straight line equation derived by Freeman and Carroll [21], which is in the form of

$\Delta \log f_0 (dW/dt) \Delta \log Wr = n - Ea\ 2.303R\Delta (1/T) \Delta \log Wr$ …… (2)

Where, dW/dt = rate of change of weight with time.

Wr = Wc-W

Wc = Weight loss at completion of reaction.

W = Fraction of weight loss at time t.

Ea= Energy of activation.

n = Order of reaction.

The plot between the terms $\Delta \log f_0 (dW/dt) \Delta \log Wr$ versus $\Delta(1/T) \Delta \log Wr$ gives a straight line. The slant, Ea/2.303R, gives energy of initiation (Ea) and catch on Y-pivot as request of response (n). The adjustment of entropy (ΔS), frequency factor (Z), apparent entropy change(S*) can likewise be determined by additional estimations.

Results and Discussion

The copolymer SASF was found to solvable in tetrahydrofuran and dimethyl sulfoxide, whereas found unsolvable in other common organic solvents usually employed. The produce of the copolymer was determined to be 82%. Elemental composition was tested using Elementary Vario EL III instrument for the carbon, hydrogen, nitrogen, and sulfur. Based on the analytical data, the empirical formula of the repeating unit was dispersed as $C_{16}H_{19}N_5S_2O_7$ with an empirical molecular weight of 457, which showing good treaty with the theoretically calculated values [23-24].

Table 1: Composition of SASF Copolymer

Copolymer	% of C Calculated Observed	% of H Calculated Observed	%N Calculated Observed	%S Calculated Observed	Repeated Unit in copolymer	Formula Weight
SASF	(44.10) 43.68	(4.02) 4.54	(14.13) 14.41	(3.12) 2.67	$C_{16}H_{19}N_5S_2O_7$	457

Molecular mass by non-aqueous conductometric titration

Non-aqueous conductometric system was developed for the determination of number-average molecular weight of the SASF copolymer in DMF solvent. For this 0.1 molar potassium hydroxide in absolute alcohol was used as the titrant. The variation of specific conductance with the milliequivalents of base was systematically scrutinized through graphical representation. The first and final discontinuities in the curve were identified and utilized for the estimation of the degree of polymerization (DP). Thereafter, the number-average molecular weight of the copolymer was determined using the equation.

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

Mn = DP x Empirical formula weight of the repeating unit

Viscosity determination was accomplished in DMF solvent at six diverse concentrations extending from 0.5 to 3% at 27 °C. The SASF copolymer displayed normal performance. Huggins and Kraemer equations were used to find intrinsic viscosity values. The calculated values of constants K_1 and K_2 (Table 2) exposed good agreement, with each other [25]. It was seen that with increase in the molecular weight intrinsic viscosity increases in case of SASF copolymer.

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

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

Where η_{sp}= Specific viscosity calculated as $\eta_{sp} = \eta - \eta_0 / \eta_0 = \eta_{rel} - 1$

η_{red}= Reduced Viscosity

η_{rel}= Relative viscosity i.e. ratio of the viscosities of solution (η) to the viscosity of solvent (η_0)

η = The reduced viscosity (red) varies with the concentration of the polymer sample; therefore, an extrapolated plot of reduced viscosity versus polymer concentration at zero concentration is required to determine the intrinsic viscosity, a key characteristic parameter of the polymer. Hence Intrinsic Viscosity is given as

$[\eta] = \lim_{c \to 0} \eta_{sp}/C$ Where C= Concentration in g/100ml

K_1= Huggins's Constant and K_2 = Kraemer's Constant

Table 2: Molecular weight and Viscosity of SASF copolymer

Copolymer	Empirical formula weight	DP	Mn	Intrinsic viscosity dl/g	Huggins Constant K_1	Kraemer Constant K_2	K_1+K_2
SASF	457	7.89	2146	0.79	0.1138	0.4792	0.593

FTIR Spectral Investigation

Thermo Nicolet iS50 FTIR spectrophotometer was used for characterization of polymer sample in Sophisticated Test and Instrumentation Centre (STIC), Kochi, Kerala, India.The broad band at 3450 cm^{-1} be ascribed to stretching frequency of amino group ($–NH_2$) within polymer series [26]. Characteristic stretching vibration at 2967 cm^{-1} may be due to Aryl C-H stretching vibration [27]. A peak at 1586.76 cm^{-1} indicates carbonyl group (-C=O) Stretching vibration group [28]. The characteristic peak at 1494.23 cm^{-1} corresponds to CH_2 bending and wagging vibrations. A sharp and intense band at 1030.84 cm^{-1} specifies the presence of S=O symmetric stretching vibration frequencies, whereas the bands at 1166.58 cm^{-1} are ascribed to its asymmetric stretching [29]. Spectral evidence of SASF copolymer is retained in table 3 and The FTIR spectrum is described in Fig. 2.

Table 2: Fourier Transform IR spectrum of SASF copolymer

Detected frequencies (cm^{-1})	Allocating groups	Estimated frequencies (cm^{-1})
3450 (b, s)	N-H stretching for amino group	3500-3200
2967 (m)	Aryl-C-H stretching frequency	2900-2500
1586.76 (m)	C=O stretching of semicarbazide group	1600-1500
1494.23	-CH_2 bending and wagging	1500-1300
1030.84	S=O symmetric stretching	1080-1030
1166.58	S=O Asymmetric stretching	1230-1160

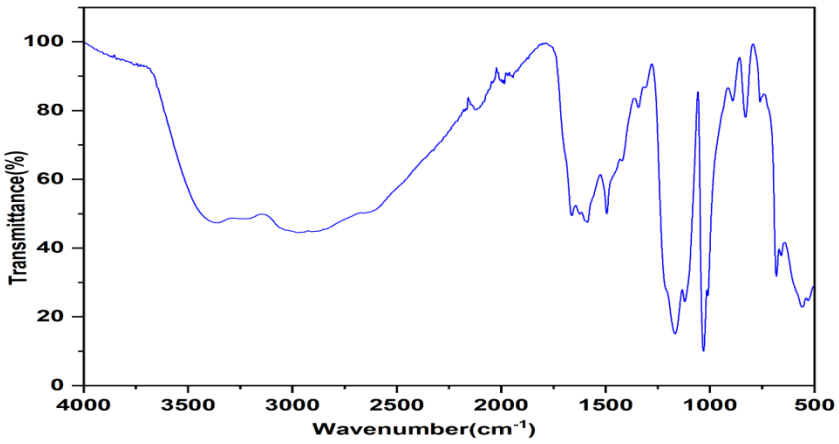


Fig. 2: FTIR spectrum of SASF copolymer

XRD Analysis

The XRD analysis of the SASF copolymer discloses both semi-crystalline nature of the material. Broad bumps in the diffraction pattern specify amorphous regions, whereas sharp, intense peaks confirm the presence of crystalline domains. Fig. 3 XRD graph shows both features high intensity with narrow band and small broadness [30]. However, the presence of a broad characteristic peak is symptomatic of substantial amorphous content, suggesting that the copolymer holds a semi-crystalline nature with both ordered and disordered structural regions.

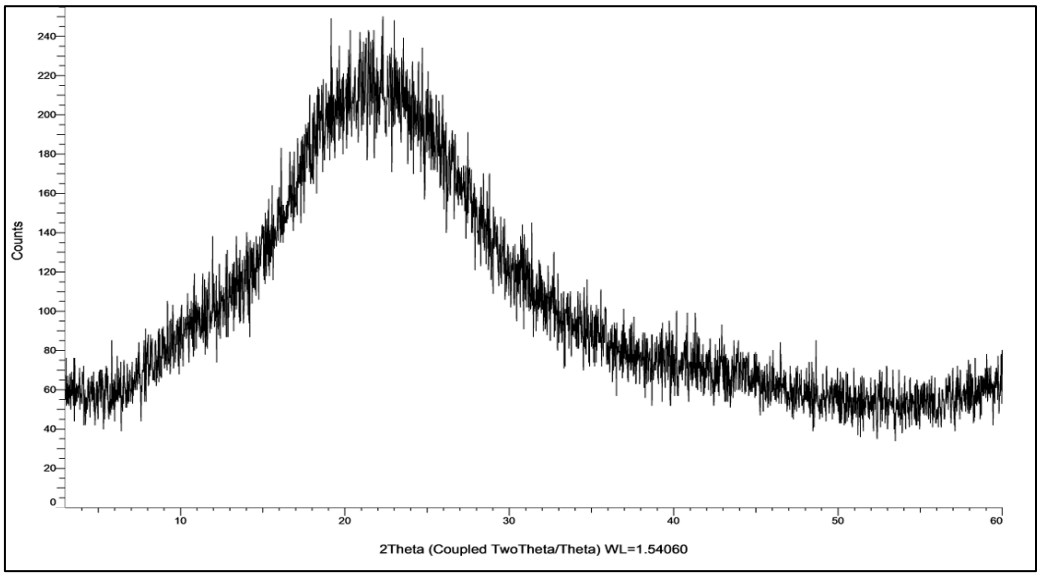
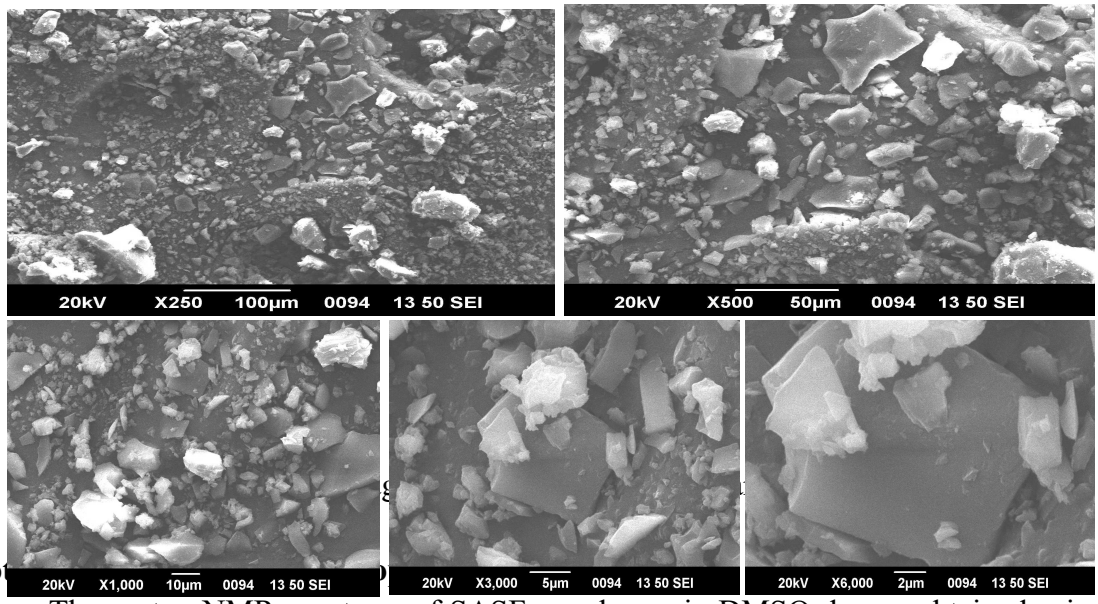


Fig. 3: XRD Spectrum of SASF copolymer

Morphology by Scanning Electron Microscope

Morphology of synthesized copolymer was studied by using Scanning Electron Microscope which is shown in the Fig. 4. At the various magnifications e.g. 250, ×500, ×1000, ×3000, and

×6000The SEM images of the SASF copolymer show crystalline–amorphous coexistence among them. Microstructural analysis exposes spherulitic and fringed features, with a heterogeneous and scattered surface morphology [31]. SEM images show dispersed surface regions with fine pits, representing phase transitions between ordered and disordered domains. The presence of fringes supports a semi-crystalline structure, marking interfacial boundaries between crystalline and amorphous phases [32]. The copolymer is just like amorphous with localized compact regions and deep surface pits.



Prot

The proton NMR spectrum of SASF copolymer in DMSO-d_6 was obtained using a Bruker Avance III 400 MHz instrument at STIC Kochi, Kerala, India. Fig. 5 shows the spectral data interpretation with reference to reported literature. A discrete sharp peak observed at δ 2.3 ppm is predictable to the methylene (–CH_2–) protons [33]. The signals appearing in the region of δ 7.0-7.8 ppm correspond to the aromatic protons of the benzene ring [34]. A singlet at δ 8.9 ppm confirms the presence of the –SO_3H functional group [35]. Additionally, a single signal at δ 5.4 ppm is allocated to the Ar–CH_2–NH group, supporting the proposed structure of the copolymer [36-39].

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

Observed chemical shift value in SAMF	Proton type assigned	Recognized range of chemical shift (∂) ppm
2.3	Methylene protons aromatic moiety	2.0-3.0
5.4	Methylenic proton of Ar-CH_2-N moiety	5.00-7.00
7.0-7.8	Aromatic ring proton	6.0-8.5
8.9	Proton of acidic linkage SO_3H group	8.00-10.00

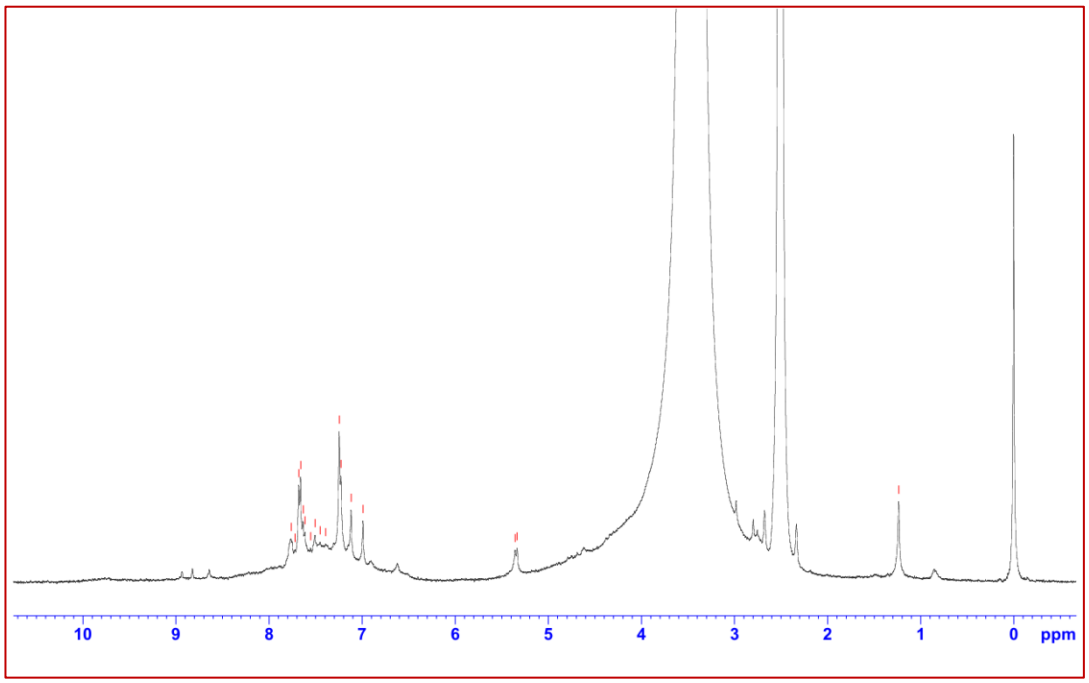


Fig. 5: ^{1}H NMR spectra of SASF Copolymer

Thermoanalytical study of synthesized copolymer

Thermal properties of copolymer give us idea about its stability, decomposition pattern and its utility under variable temperature. Thermal parameter like glass transition temperature and decomposition temperature helps us to define material's processing window, structural durability, and suitability for applications in coatings, adhesives, electronics, and heat-resistant materials [40-46]. The SASF copolymer validated good thermal stability, as per TGA curve obtained using material loss verses temperature. It shows three step degradation releasing water molecules initially within temperature 65 °C to 85 °C (3.82% found; 3.79% calculated). The thermo gram of the SASF copolymer display first major degradation step takes place within 400-550°C which look like to loss of side chain such as $–SO_3H$ and $–NH_2$ (44.82% found; 44.43% calculated;), The second degradation stage jerks among 650 °C to 750 (85.82% found; 85.05% calculated;) which is due to elimination of aromatic nucleus and methylene bridges. Finally, degradation ends up with damage of semicarbazide group at the temperature 730 °C. All these degradation steps are described in the Fig. 6.

Thermal kinetic parameters, including activation energy (Ea), entropy change (ΔS), and free energy change (ΔF), were derived using the Sharp–Wentworth model for the SASF copolymer. The linear dependence observed in the plot of log [(dc/dt)/(1 − C)] versus inverse temperature (1/T) facilitates the estimation of Ea from the slope (Figure 7). Sharp-Wentworth equation is given as:

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

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

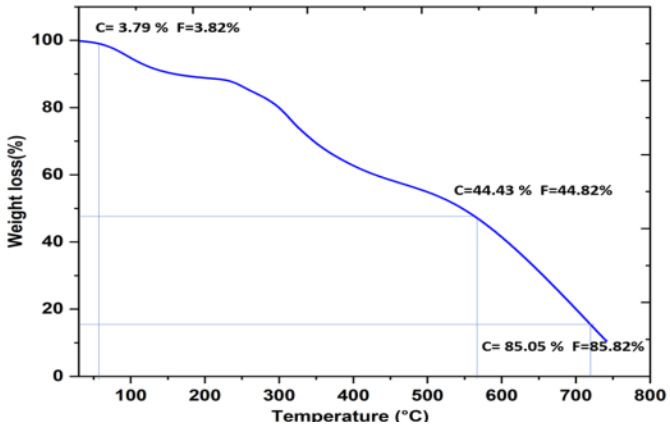


Fig. 6: Thermal degradation of SASF

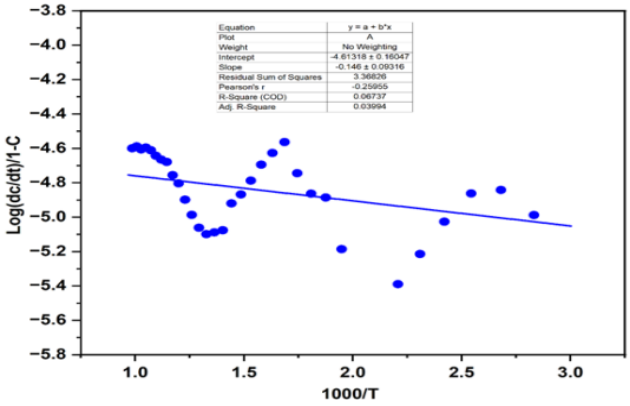


Fig. 7: Sharp-Wentworth plot Freeman-Carroll Method

The most important kinematic technique which is extensively used in the thermal studies of polymers specially for appraising their breakdown pattern by thermo-gravimetric method [39-40]. This method is scientifically known as the Freeman–Carroll method. The above method determines kinetic parameters in association with disintegration of polymer. Also indicate its thermal stability along with dissolution behavior. The Freeman–Carroll method utilizes TGA data, which records the polymer's weight loss with respect to temperature or time. It employs a differential approach to the TGA curve, correlating the rate of weight loss with the fraction of material decomposed. 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

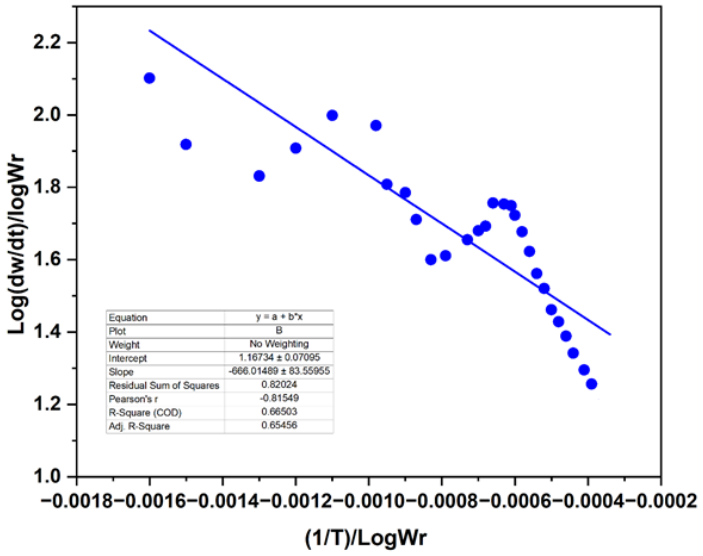


Fig. 8: F-C plot of SASF copolymer

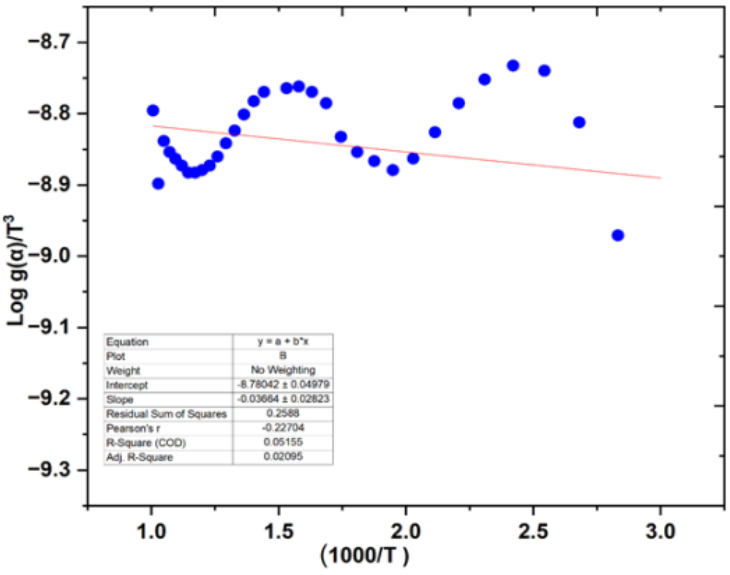


Fig. 9: Freeman-Carroll Plot

Table 5 Kinetic parameter of SASF copolymer

Copolymer	Activation Energy (KJ/m)		Entropy change	Free energy change	Frequency factor	Apparent entropy	Order of reaction
	S-W	F-C					
SASF	2.796	5.538	0.3074 J	12.402 kJ	458	-67.5678	0.82

Antimicrobial Activities of Synthesized copolymer

Measure source of infection are microorganisms like bacteria, fungi, and parasites. As recorded, worldwide death ratio by pathogenic microbes is more than any other single cause. An antimicrobial is an agent that kills microorganisms or inhibits their growth. Different types of antimicrobial drugs have been developed to acquire discontinuity of microorganism [47-53]. Both academic and industrial research is significantly influenced by antimicrobial polymers [54-64]. The antimicrobial efficiency of the SASF copolymer was found to be considerable, with enriched motion perceived against *E. coli* qualified to the standard. The most common microorganisms, a gram-negative bacillus, is generally associated in urinary tract infections. Analogous inhibitory possessions were noted against *S. typhi*, a food- and water-borne pathogen accountable for systemic febrile disorder. *S. aureus*, gram-positive species associated with multiple severe infections comprising septic and respiratory circumstances. Additionally, operative clampdown of *P. aeruginosa* highlights its potential in improving infections linked with wound sites and to the respiratory and urinary systems [Table 6].

Table 6 Antimicrobial Activity of SASF copolymer

Copolymer SASF	**Zone of Inhibition (mm)**			
Types	S_1	S_2	S_3	S_4
S. aureus	20mm	21mm	19mm	20mm
E. coli	18mm	20mm	21mm	17mm
S. typhi	21mm	19mm	17mm	19mm
P.vulgaris	23mm	18mm	22mm	23mm
A, aerogenes	23mm	25mm	26mm	24mm

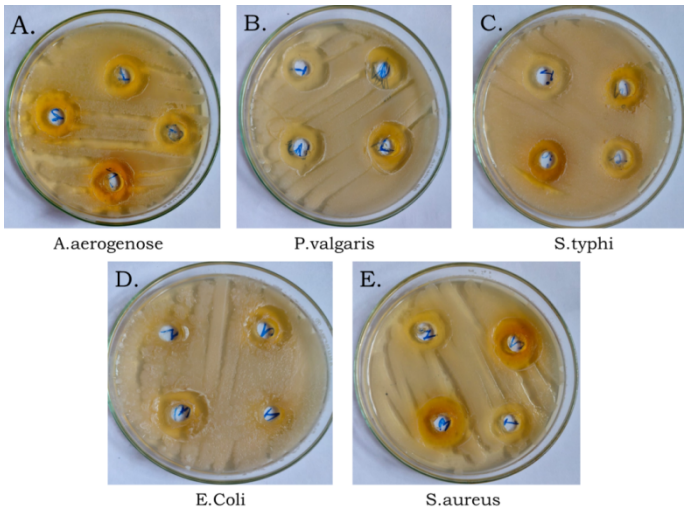


Fig. 10.

Conclusion

The synthesis of SASF copolymer was utilized under acidic conditions via polycondensation in which active synthesis of sulphanilic acid, semicarbazide, and formaldehyde. Its fundamental features were authenticated through a combination of spectroscopic and analytical techniques, comprising FTIR, ^{1}H NMR, XRD, SEM, and elemental analysis. Thermal performance measured by TGA determines appreciable stability,

along with kinetic parameters derived from established models exhibit strong correlation. The substantial displays noticeable antimicrobial activity beside a range of bacterial strains, which might be recognized to the existence of semi carbazide functional groups. Additionally, this invention proposed importance of the synthesized copolymer for antimicrobial applications in diverse field.

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