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Synthesis, Multi-Spectral Characterization, and Physicoanalytical Evaluation of the SATSCF-I Copolymer: Insights into Surface Morphology and Antimicrobial Potentia[1]

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Abstract: *The SATSCF-I copolymer was engineered via a catalytic polycondensation of sulphanilic acid (SA), thiosemicarbazide (TSC) and formaldehyde (F). The reaction was meticulously maintained at 124±2°Cfor a 5-6 hours duration in a 1:1:2 stoichiometric ratio, utilizing 2M HCl as a reaction catalyst. The structural integrity of the resin was validated through an integrated approach of UV-Visible, FT-IR, and H1 NMR spectroscopy, complemented by elemental analysis. Surface morphology and crystallinity were interrogated using Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD), revealing the copolymer's topographical features. The number average molecular weight was precisely determined through conductometric titration in a non-aqueous KOH medium. Furthermore, the terpolymer demonstrated robust antimicrobial efficacy when screened against a panel of clinically relevant pathogens, including E. coli, S. aureus, P. mirabilis and K. pneumoniae.*

Keywords: *SATSCF-I copolymer, Polycondensation, Spectral characterization, SEM-XRD analysis, Conductometric titration,Antimicrobial efficacy, Sulphanilic acid, Thiosemicarbazide.*

I. INTRODUCTION

The synthesis and characterization of copolymers have garnered significant attention due to their diverse applications in fields such as materials science and biomedicine. Researchers have focused on various monomer combinations to enhance the physical properties and functional performance of these copolymers. N. Mujafarkani and his tco-workers synthesized and characterized a novel terpolymer derived from N-Phenyl-p-phenylenediamine and 2-aminopyrimidine with formaldehyde, employing a polycondensation method in dimethylformamide as the reaction medium. This terpolymer was then used as a ligand to form a noteworthy terpolymer-metal complex with Ni(II) ions. Furthermore, the antimicrobial activity of the complex was evaluated against *Staphylococcus aureus*, *Escherichia coli*, *Candida albicans*, and *Aspergillus niger*.[1]. Similarly, J. Khobragade., M. Ahamed, and W. B. Gurnule et al. studies on the copolymer derived from phthallic acid and melamine also highlighted the importance of these materials in various technological applications [2]. D. S. Shedmake and J. V. Khobragade synthesized three coordination polymers of Cu(II), Ni(II), and Zn(II) salts using a phthalic acid-thiosemicarbazide-formaldehyde copolymer (PTF-I) as the ligand. The structures of these metal complexes were confirmed through elemental analysis and various spectroscopic techniques. Their antibacterial activities were evaluated against four bacterial strains: *Escherichia coli* (ETEC), *Staphylococcus aureus*, *Bacillus pumilus*, and *Vibrio cholerae*. Additionally, thermogravimetric analysis (TGA) was performed to study their thermal stability[3]. In a similar vein, the research conducted by M. B. Thakre and W. B. Gurnule on the copolymer derived from new 4

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hydroxybenzoic acid and adipamide emphasized the significance of these materials in a range of technological applications [4].

The comprehensive studies on copolymer resins have led to significant advancements in understanding their multifunctional properties, making them suitable for applications ranging from environmental remediation to energy conversion, as demonstrated by the work on terpolymer resins by Gurnule and Rathod [5-6].

II. MATERIALS AND METHODS

Materials

All chemicals used in this study were A.R. grade and sourced from Central Scientific Company, Nagpur, India, including sulphanilic acid, thiosemicarbazide, formaldehydeand hydrochloric acid. Double-distilled water was used for all experiments. Solvents like N, N-dimethylformamide (DMF), dimethyl sulfoxide (DMSO), tetrahydrofuran, acetone, chloroform, and diethyl ether were purchased and used as received.

Method of Synthesis of SATSCF-I Terpolymer:

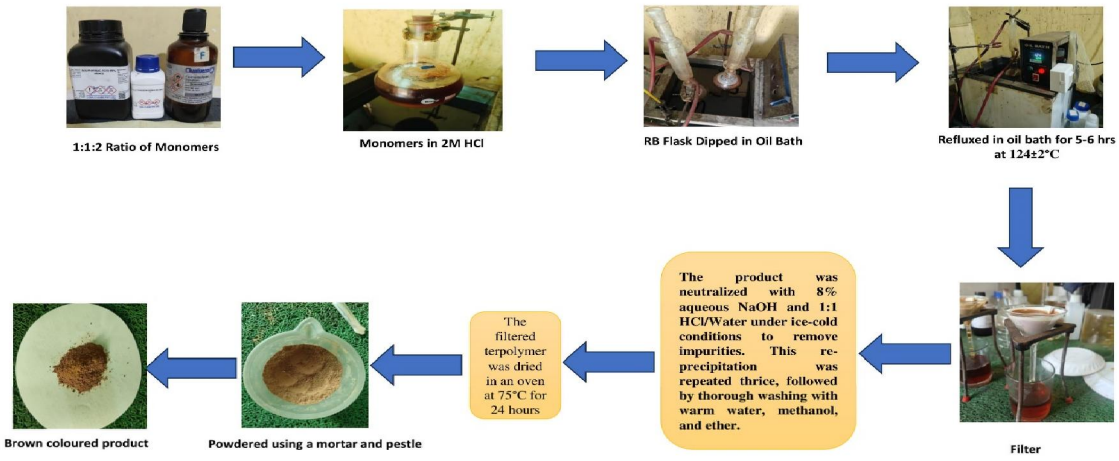

Fig 1: Schematic representation of synthesis of SATSCF-I Copolymer

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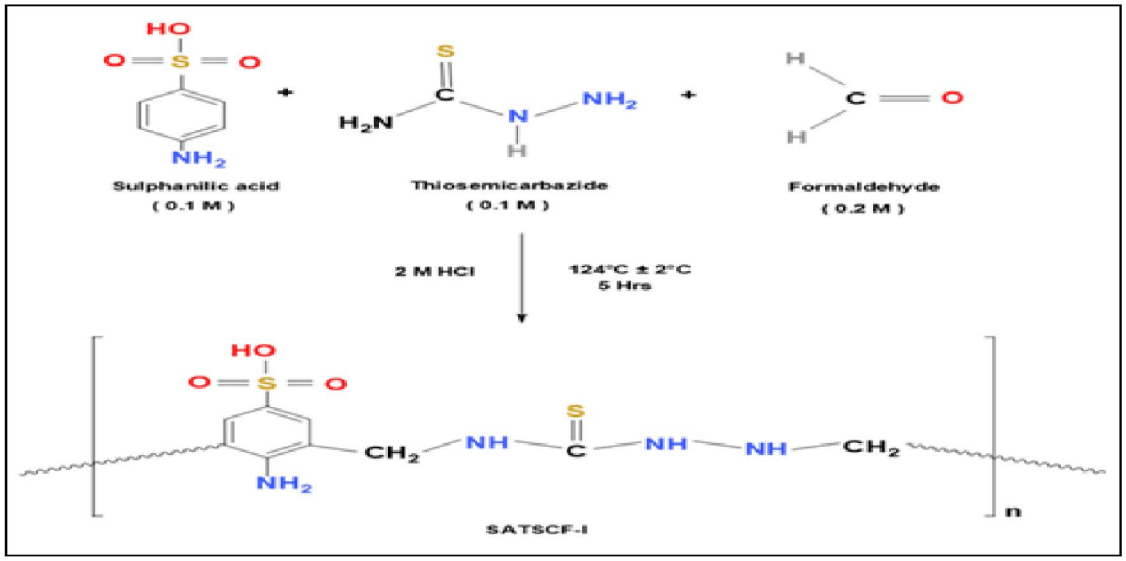

Fig. 2: Synthesis Reaction of SATSCF-I Terpolymer

III. RESULTS AND DISCUSSION

(Result obtained from STIC Sophisticated Test and instrumental Centre, Kochi)

Physico-chemical and Analytical Data

Elemental analysis

The physical and analytical data for the SATSCF-I terpolymer are presented in Table1.

Table 1. Analysis of elements and empirical formula of SATSCF-I Copolymer

Compounds	% of C Observed Found (Cal.)	% of H Observed Found (Cal.)	% of N Observed Found (Cal.)	% of S Observed Found (Cal.)	Empirical Formula of Repeated Unit	Color	Empirical Formula Weight
SATSCF-I	36.12 (37.50)	4.02 (4.16)	18.48 (19.43)	22.16 (22.24)	$C_9H_{12}N_4O_3S_2$	Dark Brown	288.21g/mol.

Molecular Weight Determination

The number average molecular weights of the newly synthesized SATSCF-I copolymer were determined using a non-aqueous conductometric titration method in a dimethyl sulfoxide (DMSO) medium. For this analysis, 50 mg of the copolymer sample was titrated with 0.5 N potassium hydroxide in absolute ethanol. A plot of specific conductance versus the milliequivalents of ethanolic KOH required to neutralize 100 g of the copolymer was constructed. The titration curves are shown in Figure 3. From these plots, the average degree of polymerization was calculated using a specific formula, which was then used to determine the average molecular weight of the copolymer.

$$(\overline{DP}) = \frac{\text{Total milliequivalents of base required for complete neutralization}}{\text{Milliequi,of base required for smallest interval}}$$

Mn = DP x Repeat unit weight of repeating unit

The results of non-aqueous conductometric titration curves were used to calculate the degree of polymerization and number average molecular weight of SATSCF-I copolymer, as shown in Table no.2.

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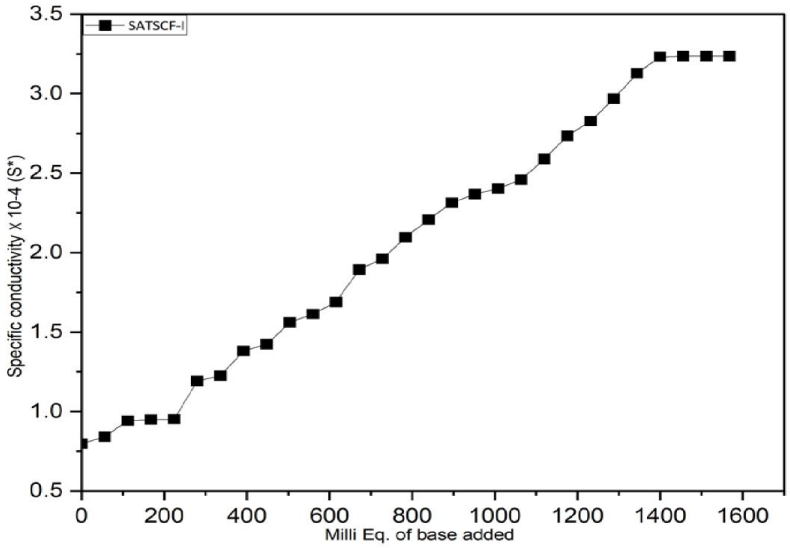

Fig.3. Conductometric titration curve of SATSCF-I Copolymer

Table 2: Number Average Molecular Weight Determination by Non-aqueous Conductometric Titration of SATSCF-I copolymer

Copolymer	First stage of neutralization (Meq./100 g Sample)	Final stage of neutralization (Meq./100 g Sample)	Degree of polymerization (Dp)	Empirical weight (gm)	Number average molecular weight (Mn)
SATYSCF-I	120	1456	12.13	288.21	3,496.95

IV. SPECTRAL ANALYSIS

UV-Visible

The UV-Vis spectrum of SATSCF-I reflects the compound's rich electronic conjugation and donor-acceptor character. The peaks indicate π→π* and n→π* transitions due to aromatic and thiosemicarbazide functionalities. The polymeric chain contributes to spectral broadening and extended absorbance, which is characteristic of macromolecular systems with multiple conjugated units.

Table 3: UV-Vis spectral data of SATSCF-I terpolymer

Wavelength (nm)	Absorbance	Type of Transition	Likely Origin
276	Strong (~1.0)	π → π*	Aromatic ring, conjugated system
378	Moderate (~0.6)	n → π*	Lone pair electrons on –NH□, –NH–, and C=S
>400	~0.4–0.2 (tailing)	Overlapping transitions	Polymericconjugation

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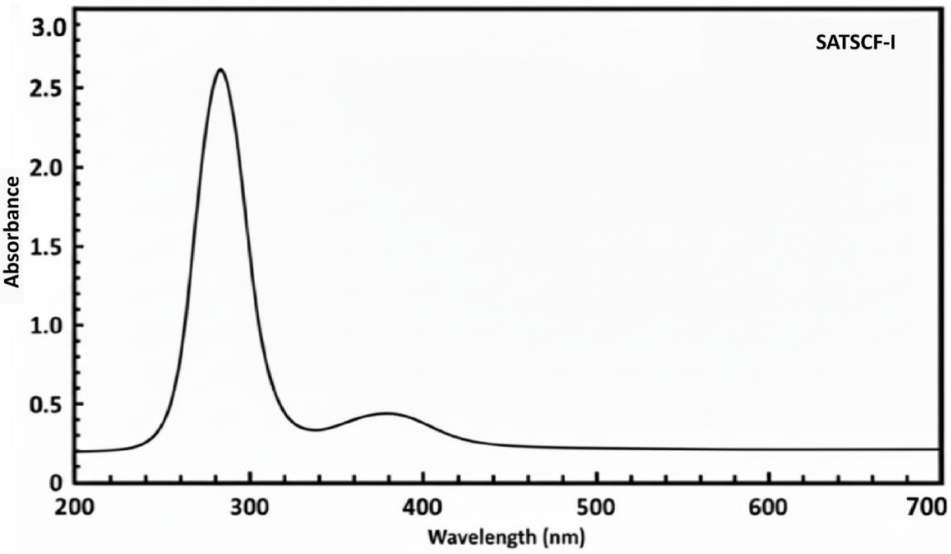

Fig.4: UV-Visible spectra of SATSCF-I terpolymer

Fourier transform infrared spectra

The infrared spectral data of SATSCF-I show notable shifts in key functional group frequencies are as follows:

Table. 4: Infrared spectral data of SATSCF-I terpolymer

Wavenumber (cm^{-1})	Functional Group / Bond	Expected band frequency (cm^{-1})
3353.07	N–H stretch	3300-3500
1713.94, 1627.94	C=S stretch	1700-1725 1622-1640
1428.85	Aromatic C=C stretching	1440-1450
1165.54, 1122.62	S=O symmetric and asymmetric stretch	1150-1200
1033.13, 894.99	N-N Stretch	1020-1060 880-910
836.74	Aromatic C–H out-of-plane bending	900-680

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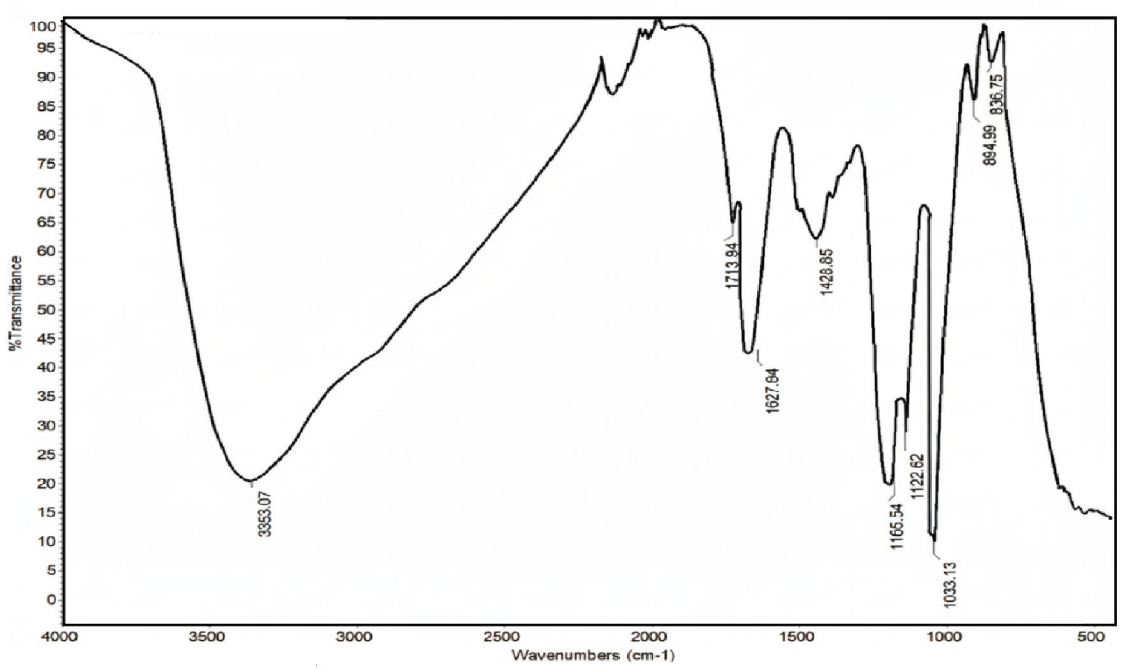

Fig. 5: FTIR spectra of SATSCF-I Copolymer

H^1 NMR

The observed chemical shift data for SATSCF-I indicate shifts in proton environments are as follows:

Table 5. H^1 NMR Spectral data of SATSCF-I Copolymer

δ (ppm)	Assignment
10.73	Broad singlet for acidic SO3H proton
9.8	-NH- Secondary amine protons of the thiosemicarbazide linkage
7.4	Aromatic proton
5.6	-NH□– proton adjacent to aromatic rings (amino group)
3.7	–CH□– Methylene bridge protons liking aromatic rings and thiosemicarbazide
2.0	Methylene proton of the DMSO solvent

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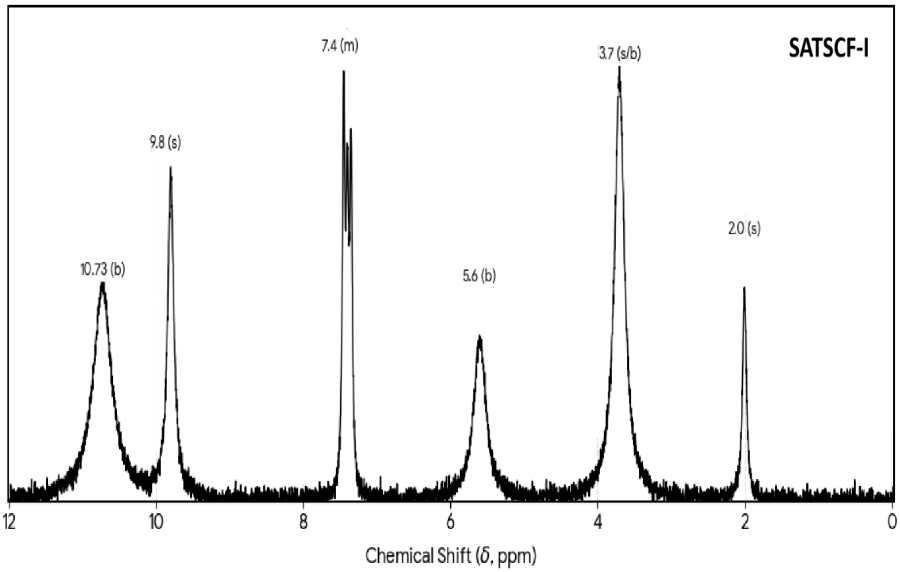

Fig. 6: H^1 NMR spectra of SATSCF-I Copolymer

V. SURFACE ANALYSIS

SEM (Scanning Electron Microscopy)

The SEM micrographs of SATSCF-I at two different magnifications (X1500 and X3000) reveal a consistent crystalline morphology characterized by regular, cube-like microcrystals. Predominantly rectangular, cubic, and well-faceted, suggesting a crystalline nature, likely from a controlled precipitation or crystallization process. Smooth and well-defined particles, indicating good purity and minimal amorphous content. Fairly uniform, especially at higher magnifications; suggests controlled synthesis parameters.

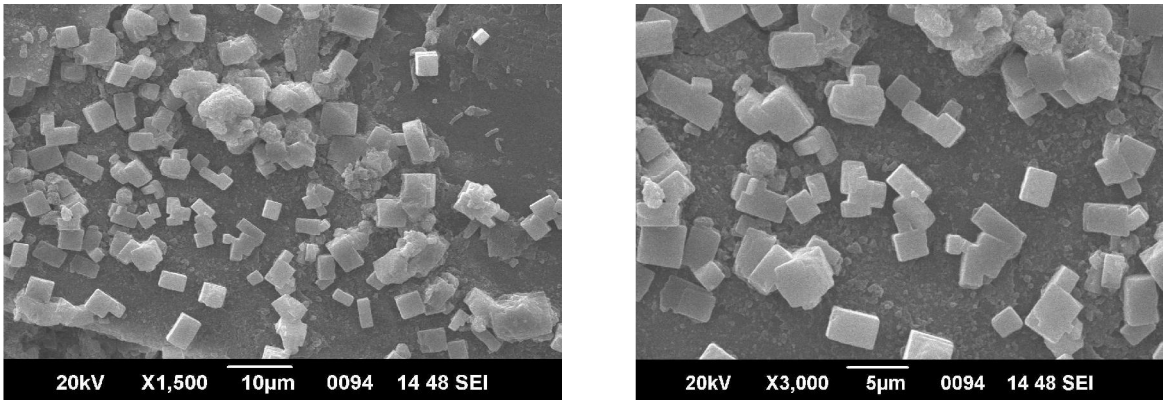

Fig. 7. SEM Micrographs of SATSCF-I Copolymer

XRD (X-ray diffraction)

The X-ray diffraction (XRD) pattern of SATSCF-1 exhibits sharp and intense peaks at 2θ values of 27.519°, 31.153°, 31.802°, 45.573°, 54.001°, 56.565°, 68.337°, and 75.412°, indicating a well-defined crystalline structure. The presence of these distinct diffraction peaks confirms the semi-crystalline nature of the polymeric SATSCF-1 compound. The most intense peak at 31.802° suggests a high degree of crystallinity along a specific crystallographic plane, which may be

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attributed to ordered packing influenced by intermolecular hydrogen bonding and the rigid aromatic backbone. This crystalline behavior supports the successful formation and structural regularity of SATSCF-I as designed.

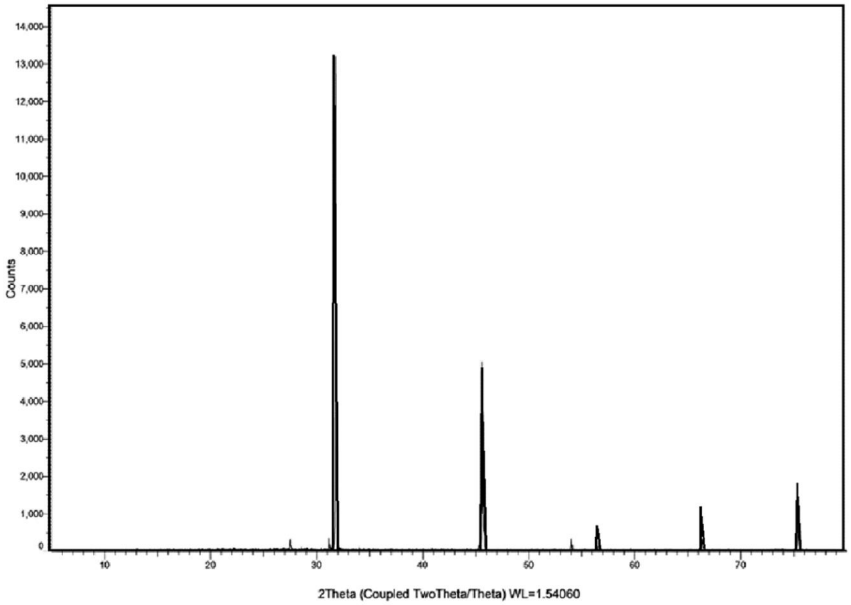

Fig. 8: X-ray diffraction of SATSCF-I Copolymer

VI. ANTIMICROBIAL ACTIVITY

The table presents the antimicrobial activity of the SATSCF-I copolymer against four bacterial strainsEscherichia coli, Staphylococcus aureus, Proteus mirabilis and Klebsiella pneumoniaeusing the agar well diffusion method.

Table no.6: Antibacterial Activity Data of SATSCF-I Copolymer

Concentration of sample	*Escherichia coli*	*Staphylococcus aureus*	*Proteus mirabilis*	*Klebsiella pneumoniae*
100 ul	18 mm	19 mm	17 mm	No zone
50ul	15 mm	15 mm	14 mm	No zone
25ul	No zone	No zone	No zone	No zone
12.5ul	14 mm	No zone	13 mm	No zone

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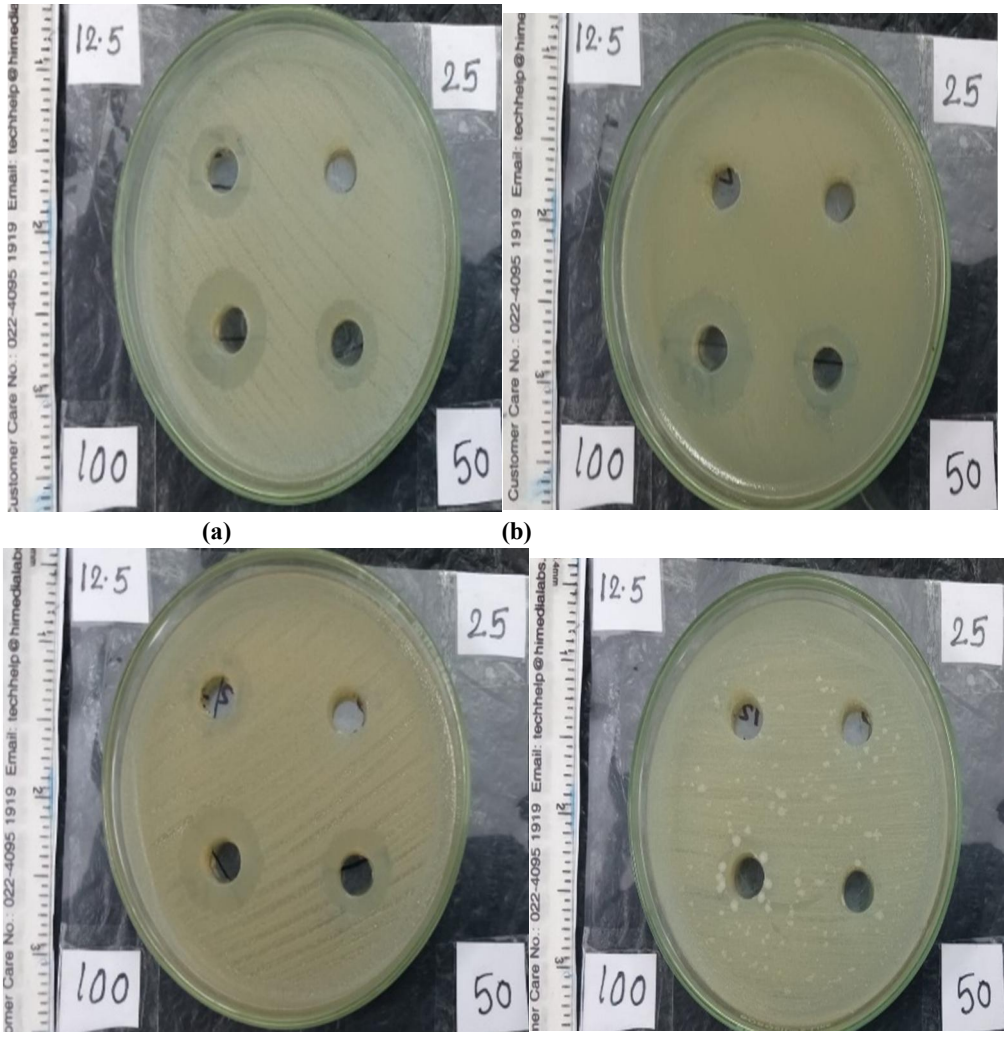

Fig.9. Antimicrobial activity of test sample SATSCF-I against (a)*E. coli, (b)S. aureus, (c)P. mirabilis* and (d)*K. pneumoniae* at different concentration by Agar well diffusion method

The observed antimicrobial activity of the SATSCF-I copolymer against Escherichia coli, Staphylococcus aureus, and Proteus mirabilis suggests its potential as an effective antibacterial agent, particularly against Gram-positive and certain Gram-negative bacteriaThe copolymer exhibited strong inhibitory effects against *E. coli*, *S. aureus*, and *P. mirabilis*, with the highest zones of inhibition observed at 100 µL (18 mm, 19 mm, and 17 mm respectively). Activity decreased with lower concentrations, and no inhibition was observed at 25 µL for any strain. Interestingly, *Klebsiella pneumoniae* showed complete resistance at all concentrations tested, with no zone of inhibition recorded. These results indicate that SATSCF-I possesses concentration-dependent antibacterial activity, particularly effective against *E. coli*, *S. aureus*, and *P. mirabilis*, but is ineffective against *K. pneumoniae*. This is significant as S. aureus is a common pathogen in skin infections, wound infections, and implant-associated infections, suggesting that the copolymer could be utilized in wound dressings, coatings for medical devices, or antibacterial hydrogels. Additionally, the effectiveness against Escherichia coli and Proteus mirabilis, both common causes of urinary tract infections, highlights potential applications in urological devices or antimicrobial surfaces. However, the lack of activity against Klebsiella pneumoniae may indicate structural

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limitations or resistance, suggesting that further modifications to the polymer structure could be necessary to broaden its spectrum of activity.

VII. CONCLUSION

The successful synthesis and comprehensive characterization of the SATSCF-I copolymer underscore its exceptional multifunctional potential, making it a strong contender for advanced applications in both material science and biomedicine. Detailed structural analysis revealed a well-organized crystalline morphology, indicating a high degree of molecular order and uniformity. SATSCF-I exhibited impressive antimicrobial activity, demonstrating significant inhibitory effects against both Gram-negative *Escherichia coli* and Gram-positive *Staphylococcus aureus*. This dual-functionalitycombining structural integrity with biological efficacyhighlights the copolymer's versatility and adaptability for multifunctional uses, such as in antimicrobial coatings and biomedical devices.

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