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RESEARCH ARTICLE

RSF-III NOVEL POLYMER: SYNTHESIS, CHARCTERIZATION AND BIOLOGICAL ACTIVITY STUDIES

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ABSTRACT

Resorcinol (R), Semicarbazine (S) and Formaldehyde (F) were taken as starting materials for synthesis of new terpolymer which has been abbreviated as 'RSF-III'. Acid catalysed condensation polymerization method was adopted for the synthesis. This terpolymer was characterized using modern techniques like FTIR, 1H-NMR, XRD and SEM. On the basis of physicochemical and spectral evidences the most possible chemical structure has been elucidated for the novel polymer. The number average molecular weight of this polymer has been determined with the help of conductometric titration method in non aqueous media. The applicability of this novel polymer under investigation towards its biological (antibacterial) activities has been studied against standards. The experimental biological activities have been compared to draw final conclusion for microbs like E. *coli*, C. *albicans*, S. *aureus* and P. *aeruginosa*. The polymer under present investigation has been found to possess very good biological activities for above quoted microorganisms.

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INTRODUCTION

Growing interest in designing and synthesis of novel polymers and organic copolymers/tercopolymers is due to their special properties and potential applications in sorption, waste water treatments, organic synthesis, hydrometallurgy, catalysis, antimicrobial, antifungal, luminescence and recovery of trace metal elements (Finn and Zubieta, 2001; Kaliyappan and Kannan, 2000; Akelah and Sherrington, 1981; Kantipuly *et al.*, 1990). The Significant research is being done in recent years on organic terpolymers/copolymers because of their antifungal, antibacterial and other biomedical applications. Some of the polymers become conducting upon doping with oxidizing and reducing agents. In biological applications, conducting copolymers are used as biosensors. Conducting copolymer based biosensors may be used to obtain clinical information for control of diseases. Areaofpolymeric resins has wide inter-relevance between various fields of science, engineering discipline and wide industrial applications. Therefore researchers in various disciplines are being attracted towards these fields. The interdisciplinary approach in the polymer resin research has emerged due to their major applications in waste water treatment, hydrometallurgy, catalysis, recovery of trace metals, electrical conductance and biological activities.

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Hence it developed special interest mostly to waste water treatment and antibacterial properties as well (Guivetchi, 1963; Saxena and Munshi, 1991; Bhave and Iyer, 1987; Geil, 1981; Rahangdale *et al.*, 2007; Gurnule *et al.*, 2003; Gurnule *et al.*, 2003; Gurnule *et al.*, 2003). The present research paper reports synthesis of a new novel polymer using resorcinol and semicarbazine as starting materials along with formaldehyde as a bridge forming agent; followed by its characterization employing various physicochemical and spectral techniques like FTIR, ^{1}H NMR, XRD and SEM. On the basis of physicochemical and spectral evidences the most possible structure has been elucidated for the new novel polymer that isRSF-III (Maskey *et al.*, 2015; Sudhirkumar Maskey *et al.*, 2015; Rahangdale *et al.*, 2003). Finally, biological/ antibacterial/antimicrobial activities of the polymer under investigation have been studied and presented in this article.

Chemicals

All chemicals used were of analytical grade. Principle starting materials viz. Resorcinol, Semicarbazine and Formaldehyde (37%) were procured from Merck, India. Double distilled water was used for all the experiments. Non aqueous solvents like DMF and DMSO were double distilled prior to their use during investigations.

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Synthesis of RSF-III Terpolymer

The novel terpolymer (RSF -III) was synthesized by condensing Resorcinol, Semicarbazine with Formaldehyde in molar ratio (3:1:4) in the presence 2M Hydrochloric acid (catalyst). The reaction mixture was taken in 500ml round bottom flask fitted with water condenser and heated in an electrically operated oil bath at 120 ± 2^{0}C for 5hrs with occasional shaking. The temperature of the oil bath was controlled with the help of dimmer stat. The solid mass obtained was removed immediately as reaction process was over. The separated terpolymer product (RSF-III) was purified employing known techniques. The solid terpolymer product was repeatedly washed with hot distilled water followed by methanol to remove unreacted monomers. The resinous product was air dried and powdered. The powder was washed several times with petroleum ether in order to remove resorcinol - formaldehyde copolymer which may be present with the terpolymer. The product so obtained was further purified by reprecipitation technique. The terpolymer was dissolved in 2.5N NaOH, filtered and reprecipitated by drop wise addition of 1:1 (v/v) conc. HCl / distilled water with constant stirring. The precipitated resin product was filtered off, washed with hot water until it was free from chloride ions which was then dried and powdered. The purified polymer sample was dried in vacuum at room temperature. The finely ground resin was passed through a 300 mesh size sieve. The purity of the polymer was tested and confirmed by TLC. The yield of terpolymer resin was found to be 88%. The reaction scheme involved and proposed structure of (RSF-III) is shown in Figure1.

Spectral Studies

H^{1}NMR, FTIR, XRD and SEM pictures of RSF-III Terpolymer/novel polymer have been respectively presented in Figure 2,3,4 &5.

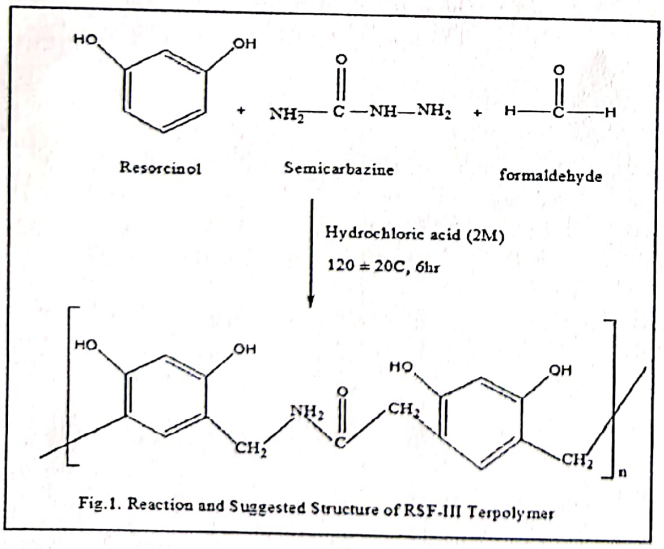

Fig.1. Reaction and Suggested Structure of RSF-III Terpolymer

Spectral data of RSF-III

M.F. -$C_{16}H_{16}O_5N$; ^{1}H NMR (δ,ppm,TMS)– 7.2-6.2(m,4H), 8.2(S,4H), 4.4 (d,2H), 2.5(d,4H), 4.55(m,2H) IR(cm^{-1})-3611(C-H, str, aromatic), 1479.6(-CH_2-Bridge, str), 1454(C=C, str) , 1090 (C-N, str) , 3411.09(-NH-, str) , 2316 (R-N=C, str (Duby *et al.*, 2000; Azarudeen *et al.*, 2009; Riswan Ahamed *et al.*, 2010; Silverstein, 1998)

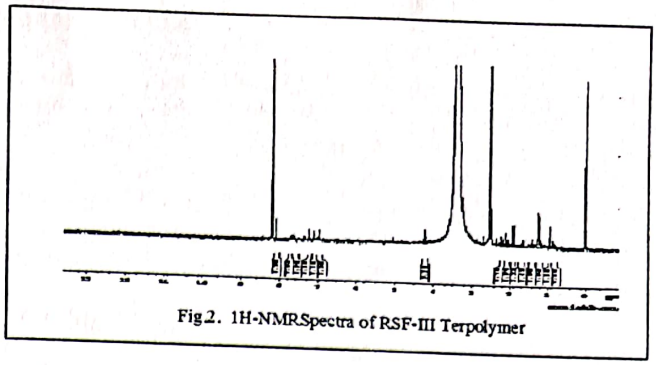

Fig.2. 1H-NMRSpectra of RSF-III Terpolymer

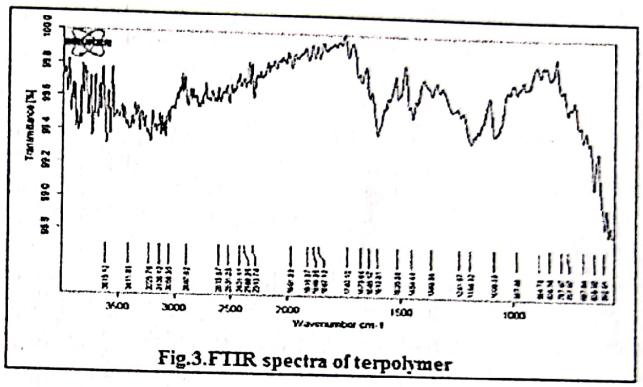

Fig.3.FTIR spectra of terpolymer

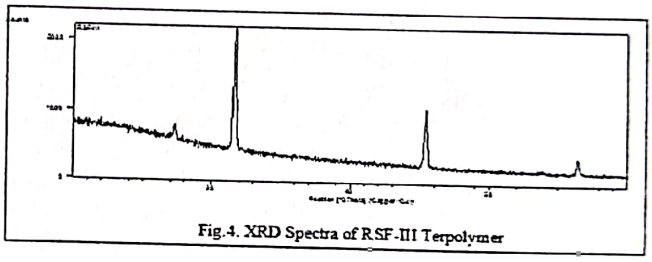

Fig.4. XRD Spectra of RSF-III Terpolymer

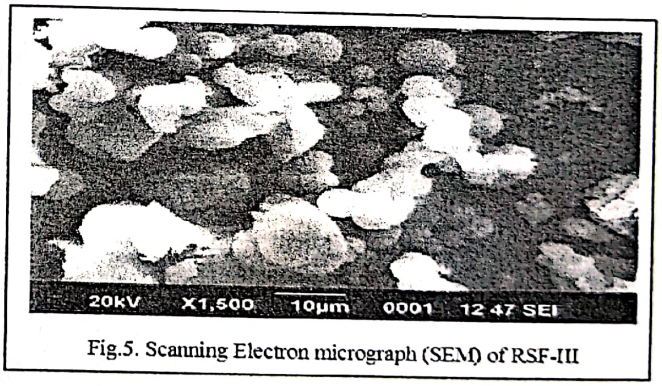

Fig.5. Scanning Electron micrograph (SEM) of RSF-III

XRD Studies

The XRD spectrum of RSF-III illustrated the presence of mixed/dual crystline nature. 1st peak nearly at 2θ=65^0 shows highly amorphous character while the peak at 2θ = 26^0 to 32^0 shows semi crystalline nature of the material (Pretsch *et al.*, 2000; Nawaz *et al.*, 2007).

SEM Studies

The scanning electron microscopy (SEM) of RSF-III indicates that clear porous structure has been properly developed in the polymer matrix. There are very small and many cavities over the surface of the RSF-III. Due to such cavity like structure of the surface, the material under study possessed high surface

area and high adsorptive properties. Adsorption depends upon the pore size of the material. Thus, RSF-III beers excellent adsorptive properties which may facilitate to enhance biological activities (Azarudeen *et al.*, 2009; Riswan Ahamed *et al.*, 2010).

In-vitro antibacterial activities

The in-vitro antibacterial activities of the RSF-III were investigated against several strains of bacteria. Nutrient agar media was utilized for the bacterial growth. Cup method was used to screen the synthesized compounds. Four microbial strains i.e. E. *coli*, C.*albicans*, S. *aureus* and P. *aeruginosa* were used in antimicrobial assay. Bacterial plates were incubated at 37^0C for 24 hrs. Streptomycin was used as standard. The self synthesised and characterized polymer was tested for its antimicrobial potency as compared to reference drug within a MIC range of 25-50µg/ml. The screening results are depicted in the Table 2.

Table 2. Antibacterial screening of the synthesized RSF-III Novel Polymer

Compound	Diameter of Zone of inhibition (mm)			
	C.albicans	*E. coli*	*S. aureus*	*P. aeruginosa*
RSF-III	24	23	16	33
Streptomycin	26	25	24	28

RESULTS AND DISCUSSION

Terpolymer RSF-III has been successfully synthesised. The purity of the synthesized compound was established by TLC and determination of the melting point. The structure of the synthesised terpolymer was elucidated by IR, ^{1}H NMR, XRD and SEM analysis. The synthesised terpolymer was screened for its antibacterial activities by cup method against various strains of gram positive and gram negative bacteria. The newly synthesised polymer under investigation has been found to possess significant antibacterial activities against E. *coli*, C.*albicans*, S. *aureus* and P. aeruginosa.

Conclusion

- Synthesis of novel polymer/terpolymer, RSF-III is successful.
- Characterization and structure elucidation by physicochemical and spectral methods is also successful.
- RSF-III possesses significant microbial/biological activities against E. *coli*, C.*albicans*, S. *aureus* and P. *aeruginosa*

Scope for future work

The work can be extended to test other applications, like ion exchange properties, of this novel polymer that is RSF-III.

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