IJARSCT

ISSN (Online) 2581-9429

International Journal of Advanced Research in Science, Communication and Technology (IJARSCT)

Impact Factor: 5.731

Volume 12, Issue 4, December 2021

Synthesis, Characterization and Antimicrobial Studies of a Copolymer and Its Composite

Yashpal Rathod[1,2], Shyam Dafare[1] and Wasudeo Gurnule[2]

Department of Chemistry, Jashbhai Mulajibhai Patel College, Bhandara, (MS), India[1]

Department of Chemistry, Kamla Nehru Mahavidyalaya, Sakkardara Square, Nagpur, Maharashtra, India[2]

wbgurnule@gmail.com

Abstract: *8-hydroxyquinoline5-sulphonic acid and anthranilic acid with formaldehyde were used to prepare the new composite. Various characterisation techniques, such as elemental analysis, FTIR, UV–Visible, NMR (^{13}C and ^{1}H), and SEM, were used to examine the structure and characteristics of the copolymer and copolymer/carbon composite. Antimicrobial analysis was also used to look into the copolymers and composites. The disc diffusion method have been used to study the antimicrobial analysis. The copolymer and its composites possess antimicrobial activity for certain bacteria such as Staphylococcus aureus, Escherichia coli, and fungi Aspergillusniger and Candida albicans. The surface morphology of the copolymer and its composites was established by SEM.*

Keywords: Copolymer, Composites, Nano carbon, Antimicrobial Analysis, SEM.

I. Introduction

Low molecular weight polymers have been synthesized in the recent years, to use as antimicrobial agents because pathogenic bacteria and fungi pose a major threat to human health, increasing research has been devoted to the discovery of new antimicrobial agents. Polymers offer improved antimicrobial efficacy and reduced residual toxicity when compared to traditional low-molecular-weight biocides. Antimicrobial polymers have been used as coatingsin food processing, filters and biomedical devices [1].A copolymer containing 2,4-dichlorophenylmethacrylateand vinyl acetate has been found to be a potent inhibitor of microorganisms development [2].Hemalatha P. and co-authors prepared copolymer of N-vinylpyrroli done (NVP) with maleic anhydride (MA) by radical copolymerization with 2,2-Azobisisobutyronitrile(AIBN) as initiator at 65^0C in dioxane solution, under nitrogen atmosphere. The resulting polymeric antibacterial material is useful in a number of medicines and food industry [3]. The antimicrobial property of the polymers plays a significant job for large numbers of its applications. Tainting by microorganisms is of incredible worry in a few regions, for example, clinical gadgets, medical care items, water filtration frameworks, medical clinic and dental supplies and so on one potential way to keep away from microbial defilement is to create the polymeric materials having antimicrobial properties [4-10].Gurnule et al. create a novel copolymer resin containing 2,4-dihydroxy benzoic acid, phenyl hydrazine and formaldehyde(2,4-DBPHF) and a composite with activated Charcoal using condensation process. Activated charcoal and synthetic copolymer were used to make polymeric composite [11-12].

Azarudeen et al. orchestrated new terpolymer ligand and metal chelates got from anthranilic corrosive aminopyridine, formaldehyde anthranilic corrosive, phenyl hydrazine and the complex were read for warm steadiness and antibacterial screening. The outcomes uncovers that all the structures are exceptionally thermally steady and more powerful antibacterial specialists than their relating ligands [13].R. Chitra et al incorporated 7-Acryloyloxy-4-methylcoumarin monomer with N-cyclohexyl-Acrylamide in various feed proportion utilizing Azobis isobutyl nitrite as initiator in DMF. The orchestrated copolymer were tried for their antibacterial action and was done by utilizing Escherichia coli, Salmonella typhi and bacillus cereus [14]. Sharma et al synthesized acetylthiophene polymers were assessed the antimicrobial activity of copolymer films against bacteria by calculating minimum inhibitory concentrations[15].The acrylate monomer, 7-acryloyloxy-4-methyl coumarin was integrated by responding 7-hydroxy-4-methyl coumarin with acryloyl chloride at 0–5 C within the sight of NaOH. Integrated copolymers were tried for their antimicrobial properties by utilizing Staphylococcus citreus, Escherichia coli and Bacillus subtilis [16].A novel comparative thermodynamic investigation of a terpolymer and its composite was carried out by Velmurugan et al. [17]. In this research article we synthesized copolymer by using 8-hydroxyquinoline5-sulphonic acid and anthranilic acid with formaldehyde and it's composite with nano carbon. The prepared copolymer and its composites were studied for antimicrobial activity for certain bacteria such as Staphylococcus aureus, Escherichia coli, and fungi Aspergillusniger and Candida albicans.

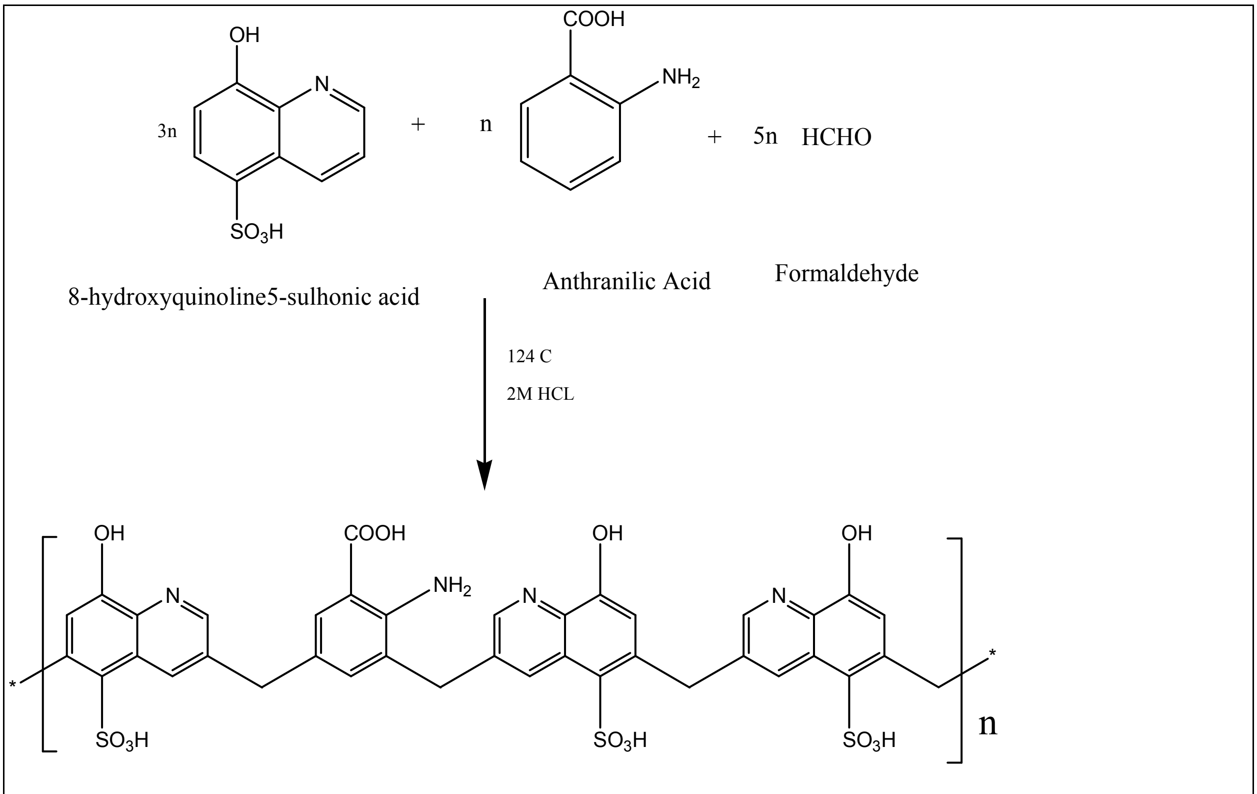
II. Experimental

2.1 Materials

All chemicals used were of AR grade. The solvents were used after distillation.

2.2 Synthesis of 8-hydroxyquinoline-5-sulphonic acid-anthranilic acid-formaldehyde copolymer

New terpolymer 8-HQ5-SAAF-III was orchestrated by gathering 8-hydroxyquinoline-5-sulphonic acid, Anthranilic acid and formaldehyde within the sight of 2M HCl as a catalyst in 3:1:5molar extent of monomers with intermittent shaking in an oil bath. Then the reaction mixtures were refluxed about 1240C ± 2 ^{0}C temp up to 5hr. The yellow color solid compound was acquired and washed a few times with hot water. After specified time, the product obtained was extricated with diethyl ether to unreacted starting materials and corrosive monomers which may be available alongside 8-HQ5-SAAF-III copolymer. It was additionally cleansed by dissolving in 8% NaOH and afterward separated. The copolymers was then reprecipitate by drop savvy expansion of 1:1 (v/v) conc. HCl and water with consistent and quick blending to maintain a strategic distance from the lumping development. The filtered test was dried, powdered and kept in vacuum desiccator over silica gel. The yield of the copolymer gum was observed to be 83% and the reaction sequence for the synthesis is shown in figure-1.



8-hydroxyquinoline5-sulphonicacid-anthanilicacidformaldehyde copolymer

2.3 Synthesis of copolymer Composites with nano carbon

The new copolymer/activated charcoal composite had been organized with the aid of polymer (0.5 g) and activated charcoal (1.0 g) in 1:2 ratios. The copolymer dissolved in 25 mL of dimethyl sulfoxide (DMSO) was taken in a round bottomed flask and activated charcoal become delivered into it and the combination was subjected to ultrasonication for 3 h with regular stirring for twenty-four h at room temperature. After the specific time, the black color composite turned into received and separated, washed with ethanol and acetone to take away the impurities, the composite was filtered and dried at 70°C for 24 hours.

III. Results and Discussion

3.1 Elemental Analysis

Elemental quantitative analysis is used to determine the content of these elements in the molecule of an organic compound. This facts is critical to assist determine the shape and purity of a newly synthesized copolymer. In the present work 8-HQ5-SAAF-III copolymer have been examined for C, H, N, and S at Indian Institute of Technology (IIT) Mumbai. The empirical formula was derived using the elemental analysis data. The estimated values and observed outcomes are found to be in good agreement. The empirical weights of a single repeating unit were determined using the empirical formula and are included in Table 1.

Table 1: Micro analysis and empirical formula of copolymer

Copolymer	% of C observed (Cal.)	% of H observed (Cal.)	% of N observed (Cal.)	% of S observed (Cal.)	Empirical formula of repeated unit	Empirical formula Weight
8-HQ5-SAAF-III	52.04 (52.34)	3.06 (3.30)	6.48 (6.8)	11.06 (11.32)	$C_{37}H_{28}N4O_{14}S_3$	848.07

3.2 FTIR Spectra of Copolymer (8-HQ5-SAAF-III)

The most powerful tool for determining the types of chemical bonds and functional groups is the Fourier transform infrared spectroscopy (FTIR). Figure 2 shows the FTIR spectrum of 8-HQ5-SAAF-IIIcopolymer and its composites. The hydroxyl group of COOH present in the aromatic ring is attributed to a broad band in the ligand spectrum in the 3423 cm^{-1} region [18]. This band appears to have fused with the hydroxyl group of 8-hydroxyquinoline's hydroxyl group. A strong band at 1313 cm^{-1} is due to CN stretching of the Ar-NH_2 group [19], while a peak at 3080 cm^{-1} is due to aromatic ring stretching modes. Between 1200 cm^{-1} and 800 cm^{-1}, sharp, medium=weak absorption bands of the 1,2,3,5 tetra substitution of the aromatic benzene ring occurred.A weak band appearing in the region 3044 cm^{-1} is attributed to -CH_2 linkage present in the copolymer.

In the spectra of composites, a broad band appearing compared to its copolymer, a broad band appears in the range of 3300 cm^{-1} to 3600 cm^{-1}, which confirms the formation of composites with nano carbon.

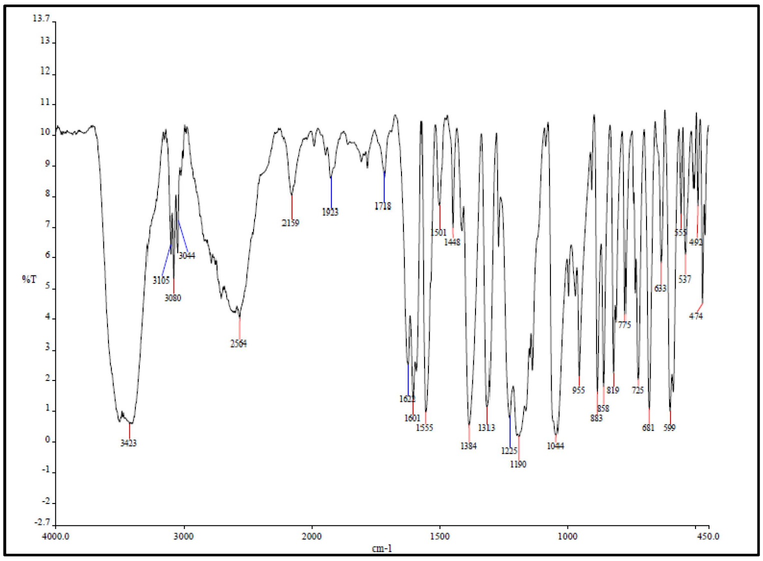


Figure 2: FTIR Spectra of 8-HQ5-SAAF-III

IJARSCT

ISSN (Online) 2581-9429

International Journal of Advanced Research in Science, Communication and Technology (IJARSCT)

Impact Factor: 5.731

Volume 12, Issue 4, December 2021

3.3 H NMR Spectra of Copolymer (8-HQ5-SAAF-III)

Figure 3 depicts the 1H NMR spectrum of the 8-HQ5-SAAF-III copolymer, The OH of Ar-COOH is responsible for the signal at 8.78ppm, and the downfield shift is caused by intramolecular hydrogen bonding between the OH of Ar-COOH and the proton of NH2 of the Ar-NH2 group. All protons in the aromatic ring are allocated to signals in the 7.1–8.0 ppm range. The methylene proton of the Ar-CH_2bridge is responsible for the signals in the 2.5 to 4.89 ppm range [20]. The -OH of the quinoline ring is responsible for the signal at 9.09 ppm. A sharp signal at 9.82 may be due to –SO_3H group.

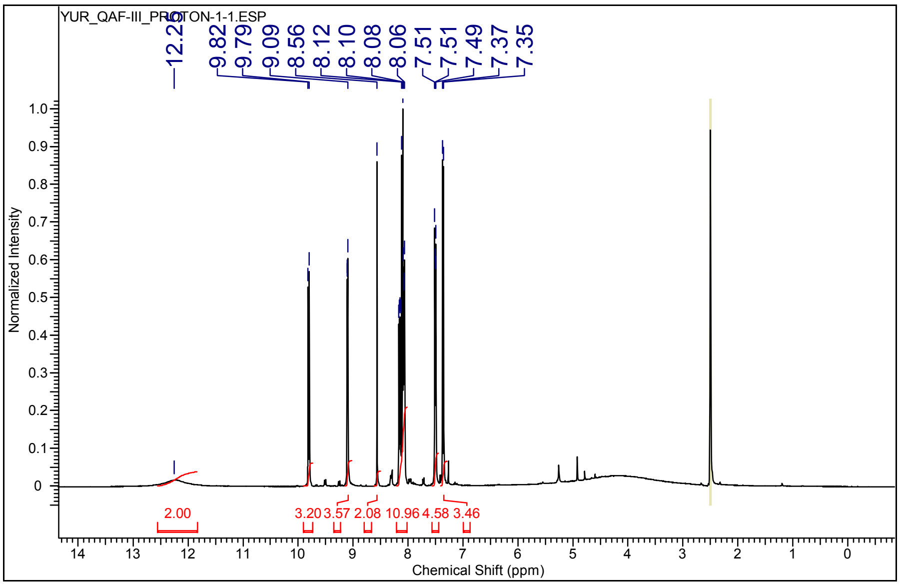


Figure 3: H NMR Spectra of Copolymer (8-HQ5-SAAF-III)

3.4 ^{13}C NMR Spectra of Copolymer (8-HQ5-SAAF-III)

^{13}C NMR spectrum of copolymer 8-HQ5-SAAF-III is shown in Fig. 4. The observed chemical shifts are assigned on the basis of the literature. The spectrum shows the corresponding peaks at 114.21, 123.07, 126.12, 128.58,128.90, and 128.94 ppm with respect to C1 to C6 of the amino aromatic ring. The peak appeared at 39.62 ppm is assigned to the Ar–CH_2 bridge in the ligand. The peaks corresponding to C1 to C9 of the quinoline ring appeared at 149.48,146.03, 144.62, 135.89, 131.71, 127.91, 127.55, 148.9, and 125.70 ppm, respectively, in the copolymer[21].

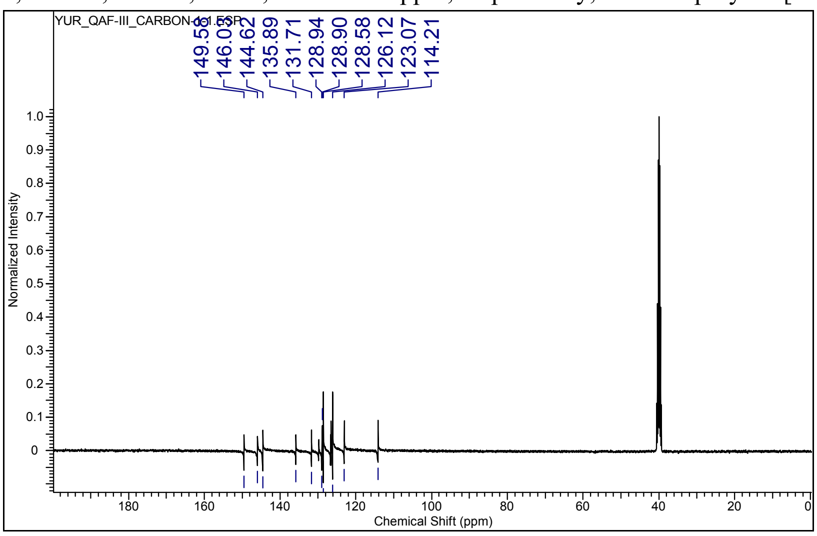


Figure 4. ^{13}C NMR Spectra of Copolymer (8-HQ5-SAAF-III)

ISSN (Online) 2581-9429

International Journal of Advanced Research in Science, Communication and Technology (IJARSCT)

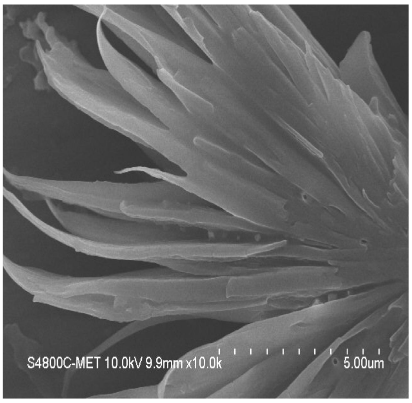
Volume 12, Issue 4, December 2021

Impact Factor: 5.731

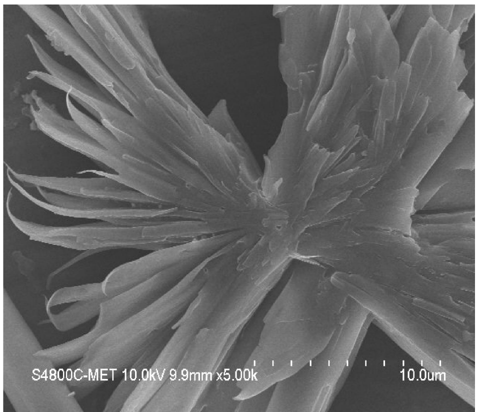
3.5 Scanning Electron Microscopy of Copolymer(8-HQ5-SAAF-III) and its composites.

Scanning electron microscopy was used to determine the surface morphology of the 8-HQ5-SAAF-III copolymer. Spherulites with deep corrugation may be seen in the photograph of the 8-HQ5-SAAF-III copolymer. Spherulites are crystalline formations that arise in a highly viscous and concentrated fluid. The surface area of the crystals is smaller, and there are less closes packed with high porosity. In addition, the spherulites shape of resin shows a crystalline structure with profound corrugation, which can be seen in SEM pictures of resin. According to the information presented above, the resin is more or less amorphous, with a less densely packed surface and deep pits. As a result, the copolymer exhibits amorphous nature and showing higher exchange capacity for metal ions. SEM analysis shows that the 8-HQ5-SAAF-III copolymer has crystalline - amorphous characters[22-23].

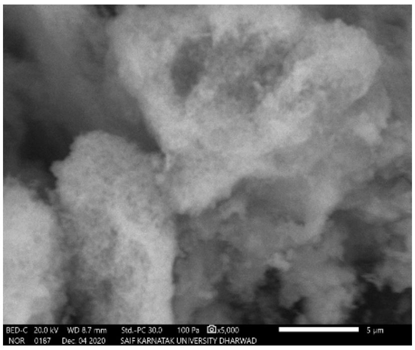
Figure 5 shows the SEM image of the 8-HQ5-SAAF-C composite (c & d). When compared to a copolymer, the surface morphology of the composite displays an overabundance of active sites and distinct pores. This suggests that increasing the surface area of the composite resulted in a greater number of cavities forming. As a result of the image, it can be deduced that the composite has formed extremely firmly with the synthesised polymer and activated charcoal, which has a greater surface area and more active sites, resulting in a higher metal ion removal capacity.



a)SEM Images of Copolymer



b) SEM Images of Copolymer


c)SEM Images of Copolymer

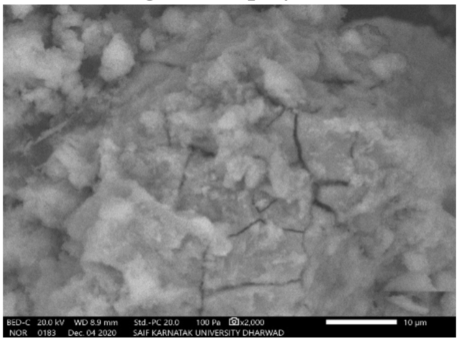

d)SEM Images of Copolymer

Figure 5. SEM Images of Copolymer (8-HQ5-SAAF-III) and its composites.

3.6 Antimicrobial Studies

3.7 Protocol for Antibacterial Activity

A biological assay compares the inhibition of microorganism growthby measuring the concentration of the sample under investigation with aknown concentration of standard antibiotic. The antimicrobialanalysis was performed utilizing the in vitro disc diffusion method.The effect of copolymer and their composites were tested for their effect on various humanpathogenic bacteria was investigated such as Gram-positive (Staphylococcus aureus),Gram-negative (Escherichia coli), and fungi (Aspergillusniger and Candidaalbicans) in this study.

For the study of antibacterial activity the nutrient agar medium was boiled and sterilized by autoclaving at 7 kgpressure (121 °C) for 15 minutes. 20mL mediawas poured into the sterilized petri plates and kept at room

DOI: 10.48175/IJARSCT-2442

temperature for afew minutes, and allowed to solidify in plates. It was then incubated for 12 hand inoculated with microorganisms using sterile swabs.

All of these procedures were carried out with extreme caution under aseptic environment. The testsolution was loaded with the chemicals that had been dissolved in DMSO.Using a micropipette was filled with themedia and incubated for 48 h at 35 °C. The similarprocedure was used for the antifungal analysis;potato dextrose was used as a control.

The test solution diffuses over time, affecting the growth of inoculation microorganisms such as Escherichia coli,Staphylococcus aureus, Aspergillusniger, and Candida albicans. The diameterof the inhibited zone was measured in millimetres to determine the activity developed on the plate. The drug ciprofloxacin was used as thestandard for bacteria and nystatin for fungi.

Results of Antimicrobial Activity of 8-HQ 5-SAAF-III Copolymer and its Composites.

The antibacterial activity of the 8-HQ 5-SAAF-III copolymer and its composites were determined by the disc diffusion method using Ciprofloxacin as a standardantibiotic. The prepared compounds were examined against Escherichia coli, Klebseilla species, Staphylococcus aureus, C. albicans, C.tropicals and A. nigermicroorganisms. The test, results presented in table 2, suggested that the copolymer and its metal composites are active against selective organisms.The 8-HQ 5-SAAF copolymer and its composites showed a moderate activityagainst this bacterial growth. E. coli is an aerobic, gram-negative, rod shapedbacterium. Infection of E. coli and can leads to bloody diarrhea and kidney failure.The prepared copolymer and its composites show moderate activity against the E. coligrowth. Klebsiella is a genus of non-motile, gram-negative bacterium infects in thesites such as nasal passages, urinary tract, bloodstream, eye and bone joints causes apneumonia, soft tissue infections and biliary tract infection. The copolymer and itscomposites showed a better activity against the growth of this bacterium.

Staphylococcus aureus, a gram-positive and spherical bacterium leads to life threatening diseases like pneumonia, osteomyelitis, endocarditis, and toxic shocksyndrome. The 8-HQ 5-SAAF-III copolymer and its composites showed good activity against thegrowth of this species. Bacillus subtilis are rod-shaped, non-pathogenic gram-positive bacteria used on plants as afungicide. Generally, they do not affect humans. But excess of bacteria into the roots causes contamination of food which leads to food poisoning. The 8-HQ 5-SAAF-III copolymer and its composites showed a good activity against the growth of this bacterium[24-25].

In general, the antibacterial effectiveness of polymer-and-metal nanocomposites improves with a high surface/volume ratio, which increases the number of ions released from the nanoparticles into the polymer. The antimicrobial activity of the copolymer may be due to the presence of –NH2 and –OH groups. It is perceived that the factors, such as solubility, conductivity, dipole moment and cell permeability mayalso contribute to the increased antimicrobial activity against the chosen microbes.

Table 2: Antimicrobial activity of 8-HQ 5-SAAF copolymer and its composites

Copolymer		**Inhibition Zone (mm)**				
	E. coli	Klebeseilla sp.	S.aureus	A.niger	C. albicans	C. tropicals
8-HQ 5-SAAF-III	16	23	16	30	25	25
8-HQ 5-SAAF-III-Composites	28	32	30	36	30	28
Standard	26	32	34	38	30	26
Control	-	-		-	-	-

IV. Conclusion

8-hydroxyquinoline5-sulphonic acid and anthranilic acid with formaldehyde were used to prepare the new composite. Various characterisation techniques, such as elemental analysis, FTIR, UV–Visible, NMR (13C and 1H), and SEM, were used to examine the structure and characteristics of the copolymer and copolymer/carbon composite. Antimicrobial analysis was also used to look into the copolymers and composites. The disc diffusion method have been used to study the antimicrobial analysis. The copolymer and its composites possess antimicrobial activity for certain bacteria such as Staphylococcus aureus, Escherichia coli, and fungi Aspergillusniger and Candida albicans. The surface morphology of the copolymer and its composites was established by SEM. The composites show higher activity against certain bacterial strains such as Staphylococcus aureus, Escherichia coli, and fungal strains Aspergillusniger and Candida albicans.

References

[1]. Ahamad T., Kumar V., Parveen S., Nishat N., ApplOrganomChem 21 (2007)1013.
[2]. Patel M., B., Patel S., A., Ray A., and Patel R., M. J ApplPolymSci 89 (2003) 895.
[3]. Hemalatha P., Veeraiah M., K., Prasannakumar S., Anasuya K., V., Int J Res Eng Tech 3 (3) 2014.
[4]. Muller P., MacDougall C., Lim M., J HospInf, 92 (2) (2016) 7-13.
[5]. Alfei S., PiattiG., Debora C., S.,A.,Maria.,Polym,13 (2021)1140.
[6]. Berber M., R., Hindawi J of Chem, (2020)19.
[7]. Nandekar K., Gurnule W., J of PhyConf Series 1913 (2021) 012063.
[8]. Al-Husaini.,J of Polym and the Env, 29 (2021)1717–1726.
[9]. Jaisankara E., Pavithra M., E., Krishna S., Thirumarimurugana M., Azarudeen R. S., Int J of Bio Macro, 145 (2020) 42-52.
[10]. Ahamed M., A., R., AzarudeenR., S., KaniM., N., HindawiBioinorgChem and Appl (2014) 1-16.
[11]. GurnuleW., B., VajpaiK., BelsareA., D., Mater today proc. 2020.
[12]. Gurnule W. B., P. K. Rahangdale, L. J. Paliwal and Kharat R. B., React. Funct.Polym., 55(3) (2003) 255-265.
[13]. AzarudeenR., S., BurkanudeenA., R., J InorgOrganometllicPolym Mater, 22 (2012) 791.
[14]. ChitraR., KayalVizhyE., JeyanthiP., PazhanisamyP., ChemSci Trans 3 (2014) 722–730.
[15]. Sharma V., A., Bansal K., Jain M., V., Int J ChemSci 11 (2013) 1110–1116.
[16]. SheikhM., I., PatelD., M., PatelN., N., PatelU., S., PatelK., P., PatelR., M., Int J IndChem, 9 (2018) 153.
[17]. VelmuruganG., Ahmed K., R., AzarudeenR., S., Iran Polym J 24 (3) (2015) 229-24.
[18]. Rathod Y., U., Zanje S., B., Gurnule W., B., J PhyConf Series 1913 (2021) 012061
[19]. Gurnule W., B.,Rathod Y., U.,Belsare A., D., Das N., C., Mater Today Proc 29 (4) (2020)1044-1049.
[20]. Gurnule W., B., Rathod Y., U.,CurrApplPolymSci 4 (2020) 1-8.
[21]. W. B. Gurnule, P. K. Rahangdale, L. J. Paliwal, R. B. Kharat, S, Rect. Func. Polym.55 (3), 255-265, 2003.
[22]. K. A. Nandekar and W. B. Gurnule, , Journal of Physics: Conference Series, 1913, 1-8 (2021).
[23]. W. B. Gurnule and Y. U. Rathod, , Current Appl. Polym. Sci., 4, 47-54 (2021).
[24]. Y U Rathod, S B Zanje and W B Gurnule, , Journal of Physics: Conference Series, 1913, 1-8 (2021)
[25]. W. B. Gurnule and N. C. Das, , Materials Today: Proceedings 15, 611–619, (2019).