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SYNTHESIS, CHARACTERIZATION AND ANTIMICROBIAL ACTIVITY OF ACETYL THIOPHENE POLYMER

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ABSTRACT

In this study, a biological evaluation of the antimicrobial activity of copolymer of styrene and acetyl thiophene containing uranyl nitrate initiated by BPO was carried out using *Escherichia coli* (Gram negative) and *Staphylococcus aureus* (Gram positive) as model organisms. BPO is free radical initiator so copolymer was prepared by free radical polymerisation. The physical properties such as softening range, solubility, permeability and adsorption of the synthesized polymer films were checked in laboratory. Preparation of polymer films were confermed by IR and NMR spectroscopy. The antibacterial activity of copolymer films against bacteria were investigated by calculation of minimum inhibitory concentrations. These investigations reveal that the highest concentration of acetyl thiophene and metal show the minimum growth of *S. aureus* and *E. coli*.

Kew words: Styrene, Thiophene, Polymer, Antimicrobial study.

INTRODUCTION

Antimicrobial polymers, also known as polymeric biocides, is a class of polymers with antimicrobial activity, or the ability to inhibit the growth of microorganisms such as bacteria, fungi or protozoans. These polymers have been engineered to mimic antimicrobial peptides which are used by the immune systems of living things to kill bacteria. Typically, antimicrobial polymers are produced by attaching or inserting an active antimicrobial agent onto a polymer backbone via an alkyl or acetyl linker. Antimicrobial polymers may enhance the efficiency and selectivity of currently used antimicrobial agents, while decreasing associated environmental hazards because antimicrobial polymers are generally nonvolatile and chemically stable. This makes this material a prime candidate for use in areas of medicine as a means to fight infection, in the food industry to prevent bacterial contamination, and in water sanitation to inhibit the growth of microorganisms in drinking water[1].

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Sulphur had been an intresting element particularly for the varieties of its compounds and it is involved in varities of bondings[2]. It has a prominent bioorganic chemistry and a large number of enzymes and other proteins exhibited fantastic properties involving the sulphur atoms in them[3,4]. It is an important element, which forms chains[5] and more and more interestingly, bridges in polymers and in varieties of composite materials, giving rise to strength and resistance to aging. The remarkable pharmacological efficiency of the compounds with the thiophene nucleus in their structure such as duloxetine, phethenylate, pyrantel, tiomonium iodide, chlorothen, carticaine, thennalddine, tipepidine and tenosol known for their antidepresent anticonvulsant, anthelmintic, antispasmoidic, antihistaminic, anesthetic, antiprritic, antitussive and analgesic action.

Literature is available on antimicrobial polymers synthesized by the condensation of epichlorohydrin (1-chloro-2,3-epoxy propane) with Schiff base metal complexes in alkaline medium. Schiff base was initially prepared by the reaction of 2,6-dihydroxy 1-napthaldehyde and o-phenylenediamine in 1 : 2 molar ratio and then with metal acetate[6]. A literature survey also reveals that metal containing antibacterial polymers were prepared by the polymerization of methylmethacrylate and methacrylic acid[7] with copper or zinc.

A new polymeric Schiff base containing formaldehyde and 2-thiobarbituric acid moieties was synthesized by the condensation of a monomeric Schiff base derived from 2-hydroxyacetophenone and hydrazine. Polymer–metal complexes were also synthesized by the reaction of the polymeric Schiff base with Mn (II), Co (II), Ni (II), Cu (II), and Zn (II) acetate. Among all of the complexes, the antimicrobial activity of the Cu (II) polymer-metal complex showed the highest zone of inhibition because of its higher stability constant and may be used in biomedical[8]. Organotins are known to have broad antitumor activity[9-14] through different mechanisms including the inhibition of cellular DNA replication[15-17].

EXPERIMENTAL

Material and method

Monomer styrene and uranyl nitrate was purchased from Alfa Aesar (Germany). Styrene was washed by 10/NaOH solution before use and kept in a dry and cool place specially, kept under refrigeration and brought to room temperature before use. BPO was supplied by CDH, L. R., India, was used after recrystallization from methanol. Acetyl-thiophene was also obtained from Alfa Aeasar (Germany). Organic solvents like benzene, toluene, choloroform and acetone were used after double distillation.

The physical properties such as softening range, solubility, permeability and adsorbity of polymer samples are checked in laboratory. The IR spectra of newly

synthesized polymer samples were recorded by Perkin-Elmer spectrophotometer with in the range of 450-4,000 cm^{-1} using resolution of 4 cm^{-1} from SAIF, CDRI, Lucknow. The NMR spectra of the polymer samples were recorded from SAIF, CDRI, Lucknow.

Preparation of Sty +Ac-Thio copolymer containing uranyl nitrate

10 mg uranyl nitrate was dissolved in 3 mL choloform and stirred for 40-50 minutes at 60-70^{o}C temperature. After stirring the solution styrene, acetyl thiophene and BPO were added and the mixture were heated on water bath and polymerization proceeded at 80-85^{0}C and for 4 h using the different concentration of acetyl thiophene as (PF1-PF2), styrene as (PF3-PF4), and uranyl nitrate as (PF5-PF7).

The reaction was stopped by the addition of 2 mL of 5% (w/v) quinol solution to the reaction mixture. The mixture was poured into large excess of methanol with stiring to precipitate the polymer. The polymer was dissolved in benzene and stirred for about 30 minutes at 60-70^{o}C on water bath. The polymer solution was poured on flat glass plate to prepared polymer films.

Biocidal assay

The antibacterial and antifungal activities of the metal containing polymer films have been evaluated by testing these films against bacterial (*Staphylococcus aureus* and *Escherichia coli*) at different concentrations by using filter paper disc diffusion method[18].

Preparation of solution of test compounds

The solutions of metal containing polymer films are prepared (250 ppm, 500 ppm and 1000 ppm). The calculated weight of the sample was dissolved into suitable solvents i.e. benzene.

Inoculation of test plates

At least four or five well-isolated colonies having the same morphological types were selected from an agar plate culture with a wire loop. The top of each colony was touched and transferred the growth to a tube containing 4 to 5 mL of a suitable broth medium. The broth culture was incubated at 37^{0}C for 8 hours. The sterile cotton swab on woolen application was dipped into inoculums. The excess of inoculums was removed from swab by rotating (several times) inside the wall of the test tube above the final level. The dried surface of a Miller Hinto agar plate was inoculated by streaking the swab over the entire sterile agar surface. The streaking was prepared two or three times so as to ensure an even distribution of the inoculums[19-22]

Procedure of bactericidal study

Twenty five days old culture of *Escherchia Coli* and *Staphylococcus aureus* were used as the test organism. Sterilized filter dises of 5 mm diameter impregnated with different concentration of test compound (250, 500, 1000 ppm) were placed on the surface medium. The activity was recorded after 24 hours of incubation $37^0 + 0.1^0$C and zone of inhibition was measured. Assey discs soaked in the solvents were used as control. The Percentage of inhibition was calculated by using the equation as -

$$\% \text{ Inhibtion} = [C\text{-}T / T] \times 100 \quad \ldots(1)$$

Where C = Diameter of organism colony in control plate and

T = Diameter of organism colony in test plate.

RESULTS AND DICUSSION

The copolymer of styrene and acetyl thiophene containing uranyl nitrate was prepared by varying the concentration of styrene and acetyl thiophene (Table 1) and uranyl nitrate (Table 2). The copolymer films are brown in colour, brittle and very light in weight. Softening range of copolymer films is between 110-150^{0}C. Films are completely soluble in benzene, toluene and chloroform, insoluble in inorganic solvents and partially soluble in DMF, dioxane and ethyl acetate. All the polymer films are found to be impermeable in water. The adsorption power of copolymer films was checked in DMSO, ethanol, nitric acid and water. The adsorpation power is highest in water. In IR spectra of copoylmer film, styrene monomer show absorption band at 3025 cm^{-1} due to aromatic hydrocarbon, at 1491 cm^{-1} due to C=C stretching of styrene, at 350 cm^{-1} due to carbon metal linkage, at 757 cm^{-1} due to C-S-C stretching of thiophene ring and at 1804 cm^{-1} due to acetyl group. ^{1}H NMR spectra show signal at 7.0 δ due to aromatic proton, at 6.5 δ due to thiophene ring proton, at 1.8 δ due to $–CH_3$ proton of acetyl thiophene and peak at 1.2 δ due to $-CH_2$ proton. All these bands observed in the NMR spectra confirmed the formation of copolymer of styrene and acetyl thiophene containing uranyl nitrate initiated by BPO.

Eschirichia coli and *Stayphlococeus aureus* were used as test organisms for bacterial studies. The bacterial activity of copolymer of styrene, and acetyl thiophene containing uranyl nitrate has shown that it was most active because sulphur atom and acetyl group is present in acetyl thiophene. So the polymer film PF2 shows higher inhibition zone than PF3 and PF4. The most extensive inhibition zone were observed in case of *S. aureus* than *E. coli*. The bactericidal activity of polymer films increased with concentration i,e- at 250 ppm, it is less active but at 1000 ppm, it is most active. Higher is the concentration of metal in copolymer, more is the biological activity of copolymer. The observed results are presented in Table 3.

Table 1: Composition of reactant used in preparation of films of copolymer of Sty + Ac-thio containing uranyl nitrate by keeping the change in concentration of styrene and acetyl thiophene

Polymer film code	Styrene (mole. L^{-1})	Acetyl thiophene (mole. L^{-1})	BPO (mg)	Uranyl nitrate (mg)	Choloroform (mL)
PF1	1.7×10^{-2}	8.7×10^{-2}	50	50	03
PF2	1.7×10^{-2}	2.6×10^{-2}	50	50	03
PF3	2.6×10^{-2}	1.7×10^{-2}	50	50	03
PF4	8.7×10^{-2}	1.7×10^{-2}	50	50	03

Table 2: Composition of reactant used in preparation of films of copolymer of Sty + Ac-thio containing uranyl nitrate by keeping the change in concentration of uranyl nitrate

Polymer film code	Styrene (mole. L^{-1})	Acetyl thiophene (mole. L^{-1})	BPO (mg)	Uranyl nitrate (mg)	Choloroform (mL)
PF5	1.7×10^{-2}	1.7×10^{-2}	200	100	03
PF6	1.7×10^{-2}	1.7×10^{-2}	200	10	03
PF7	1.7×10^{-2}	1.7×10^{-2}	200	50	03

Table 3: Biological screening of copolymer of styrene-acetyl thiophene containing uranyl nitrate (Inhibition of radial growth in mm)

Polymer film code	Organism					
	E. Coli			***S. Aureus***		
	250	**500**	**1000**	**250**	**500**	**1000**
PF1	o.5	1	2	0.5	1.5	3
PF2	0.5	1	1.5	0.0	2.0	2.5
PF3	0.5	0.5	0.5	0.5	1.0	2.0
PF4	0.5	0.5	0.5	0.5	0.5	1.0
PF5	0.5	0.5	0.5	1.0	2.0	3.0
PF6	0.5	1.0	1.5	2.0	3.0	4.0
PF7	0.5	1.0	1.5	2.5	3.5	5.0

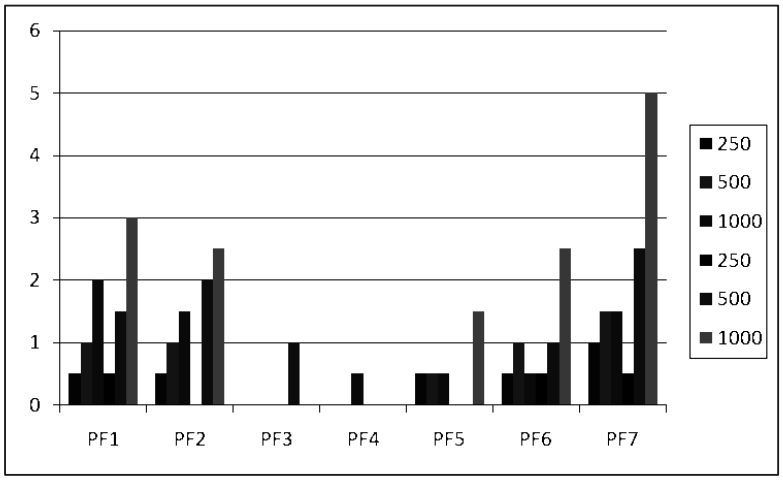

Fig. 1:

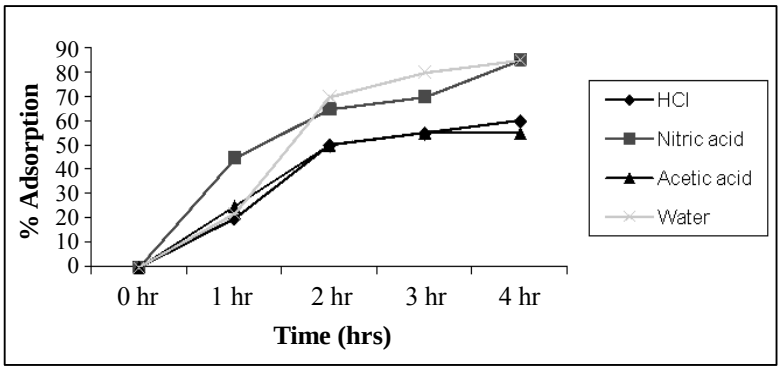

Fig. 2: Adsorption power of terpolymer of styrene, acrylonitrile and thiophene initiated by BPO

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