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N-tert-Butylacrylamide Based Copolymers: Synthesis and Characterization of Poly (NTB -co-NVP)

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Abstract: ***A series of copolymers N-tert-butylacrylamide(NTB) and N-vinyl pyrrolidone (NVP) were prepared by free radical polymerization in Dioxane medium at 70^{0}C using AIBN as initiator. The copolymers were characterized by ^{1}H-NMR spectroscopy and the copolymer compositions were determined by^{1}H-NMR analysis. It shows antimicrobial activity. The activity of copolymers against bacteria and fungi also determined by Well-diffusion method.***

Keywords: free radical polymerization, copolymer composition, antimicrobial activity.

1. Introduction

Microbial infection remains one of the most serious complications in several areas, particularly in medical devices, drugs, health care and hygienic applications, water purification systems, hospital and dental surgery equipment, textiles, food packaging, and food storage. Antimicrobials gain interest from both academic research and industry due to their potential to provide quality and safety benefits to many materials. Antimicrobial agents are those materials capable of killing pathogenic microorganisms. Poly-vinyl pyrrolidone has wider applications in the pharmaceutical field and in the cosmetic field also .The use of antimicrobial polymers offers promise for enhancing the efficacy of some existing antimicrobial agents and minimizing the environmental problems accompanying conventional antimicrobial agents by reducing the residual toxicity of the agents, increasing their efficiency and selectivity, and prolonging the lifetime of the antimicrobial agents.

In the present study, we described the synthesis of copolymers N-tert- butylacrylamide(NTB) and N-vinyl pyrrolidone(NVP) were prepared by free radical polymerization in Dioxane medium at 70^{0}C using AIBN as initiator. The copolymer was characterized by ^{1}H-NMR spectroscopy and the copolymer compositions were determined by^{1}H-NMR analysis. These copolymers subjected to antimicrobial activities against selected Bacteria and Fungi.

2. Experimental

Acrylonitrile was first washed with 5% NaOH solution in water to remove the inhibitor and then with 3% Orthophosphoric acid solution in water to remove basic impurities. Then the Acrylonitrile was washed with double distilled water and dried over anhydrous $CaCl_2$. The acrylonitrile was then distilled in an atmosphere of Nitrogen and reduced pressure. It was then collected in a clean dry amber colored bottle and kept in the refrigerator at 5^{0}C.

2.1. Preparation of N-tert-butyl acrylamide (NTB)

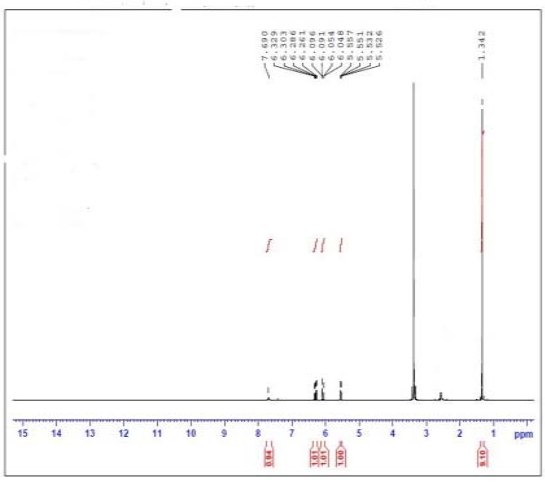
The monomer N-tert-butylacrylamide was prepared by the reaction Tertiary-butyl alcohol with acrylonitrile. N-tert-butylacrylamide was recrystallized in warm dry benzene. The monomer was confirmed by both ^{1}H-NMR.

2.2 .^{1}H-NMR spectroscopy

The prepared monomer N-tert- butylacrylamide was characterized by both ^{1}H-NMR . The characteristic group peak assignments are given as follows

The 1H-NMR spectra of monomers and copolymers were recorded on the GSX-400 spectrometer (JEOL,Tokyo, Japan) operating at 400 MHz respectively in CDCl3.

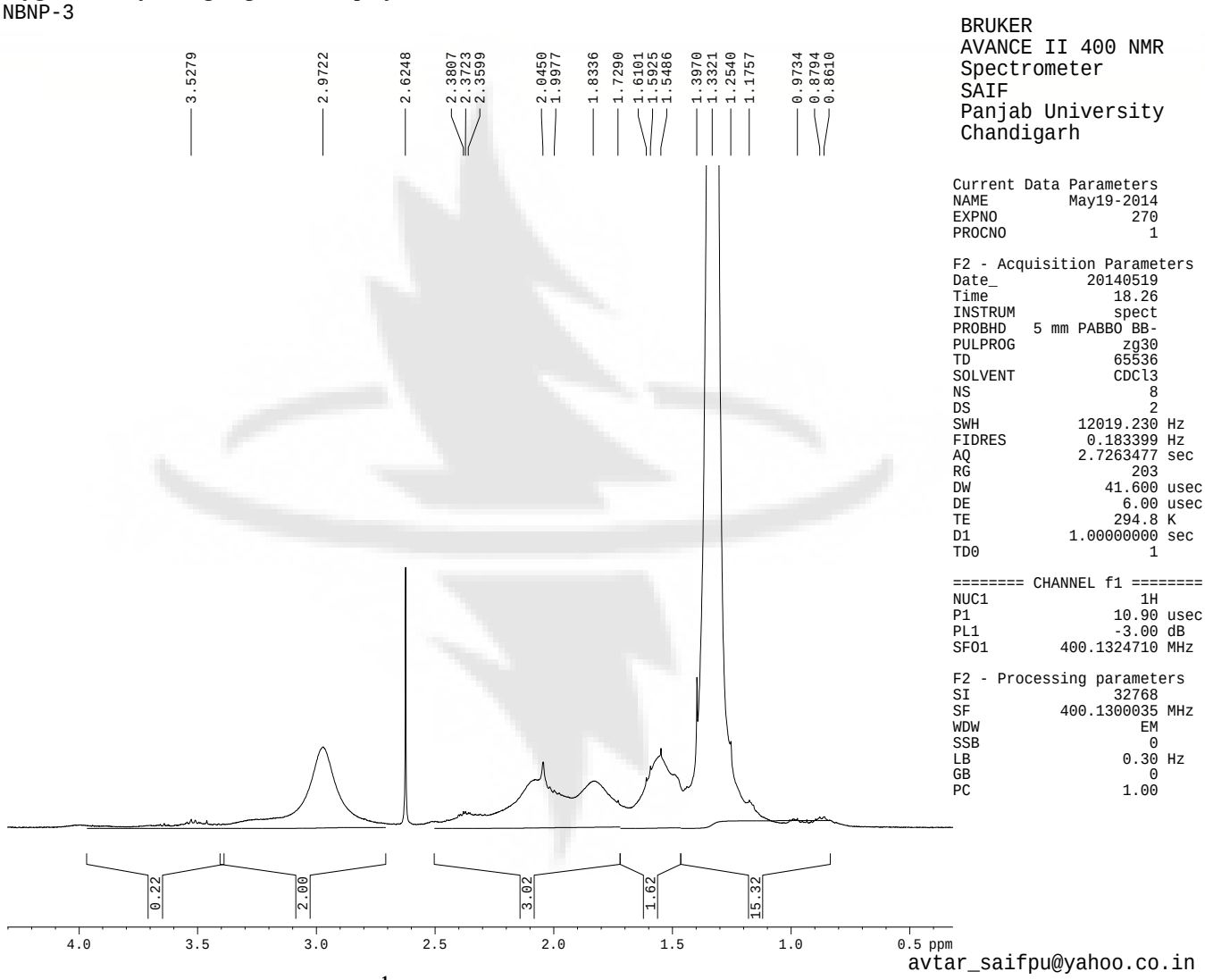
The following peaks appear in NTB spectrum; at 1.42 ppm for tert-butyl protons, at 5.59-6.28 ppm for vinyl protons and at 7.27 ppm for N-H proton



^{1}H-NMR spectrum of N-tert-butylacrylamide

2.3. Copolymerization

A total feed of 5g of monomers N-tert-butylacrylamide, N-vinyl pyrrolidone and 50 mg of AIBN initiator were dissolved in Dioxane and the mixture was flushed with oxygen free dry nitrogen gas. The copolymerization reaction was carried out at 70 ^{0} C .The solution poured in ice cold water to precipitate the copolymer and the copolymer washed with methanol to remove unreacted monomers. It was then dried in vacuum oven for 24 hours. The copolymer polymer was characterized by ^{1} H- NMR spectroscopy.



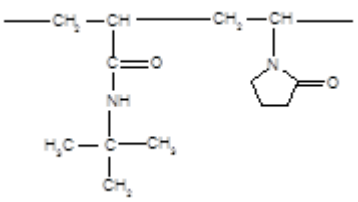
^{1}H-NMR spectrum of copolymer NTB-NVP

2.4. Antibacterial Activity and antifungal activity (well diffusion method)

Antibacterial analysis was followed using standard agar well diffusion method to study the antibacterial activity of compounds. Each bacterial and fungal isolate was suspended in Brain Heart Infusion (BHI) broth and diluted to approximately 10^{5} colony forming unit (CFU) per mL. 5mm diameter wells were cut from the agar using a sterile cork-borer and 30 µL (5µg compound in 500 µL DMSO) of the sample solution were poured into the wells. The plates were incubated for 18 h at 37°C for bacteria and at room temperature for fungi. Antimicrobial activity was evaluated by measuring the zone of inhibition in mm against the test microorganisms. DMSO was used as solvent control. The tests were carried out in triplicates

3. Results and Discussion

A series of copolymers N-tert-butylacrylamide (NTB) and NVP were prepared by free radical polymerization in Dioxane medium at 70^{0}C using AIBN as initiator The schematic representation of the copolymer is given below:



Scheme 1: Copolymerization of NTB and NVP

3.1. Antifungal activity

These polymer samples were tested against the Gram positive and Gram negative at various concentrations as

mentioned in table. From the table it is noticed that the activity of polymers against bacteria and fungi increases with increasing mole % of NTB. These polymers are more active against bacteria and fungi

S. No.	*Organism (Bacteria)*	*Zone of Inhibition in mm*		
		Poly(NTB-NVP)1:3	*Poly(NTB-NVP)3:1*	*DMSO*
1	*Escherichia Coli*	*11*	*15*	*No Zone*
2	*αDH5*	*Resistant*	*13*	*No Zone*
	FUNGI			
1	*T.Rubrum*	*Resistant*	*11*	*No Zone*

Figure 1: Antibacterial analysis of polymers

4. Conclusion

The copolymer was found to play a crucial role in conferring antibacterial activity towards the inhibition of bacterial infections. Scope of this study:

Production of antimicrobial copolymer has enormous applications in bio-related fields. The design and development of such polymers have opened a new era for constructing well designed novel class of material for catalytic, optical, electronic and bio-medical applications.

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