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N-Tert-Amylacrylamide Based Copolymers: Synthesis And Characterization Of Poly (NTA -Co-NVP)

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Abstract: ***A series of copolymers N-tert-amylacrylamide (NTA) 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

Polymers have very promising applications in the biomedical field as delivery systems of therapeutic agents, tissue engineering scaffolds, cell culture supports, etc., Polymers is materials that prevent microorganism growth and are needed for many everyday applications from food packaging and water treatment to medicine and healthcare. The control of microbial infections is a very important issue in modern society. In general there are two ways to stop microbes from infecting humans or deteriorating materials—disinfection and antimicrobial surfaces. The first is usually realized by disinfectants, which are a considerable environmental pollution problem and also support the development of resistant microbial strains.. One possible way to avoid the microbial contamination is to develop antimicrobial agents [1-5].

Antimicrobial agents are those materials capable of killing pathogenic microorganisms. Antimicrobials gained interest in both academic research and industry due to their potential to provide quality and safety benefits to many materials. PVP has wider applications in the pharmaceutical field and in the cosmetic field also .Thus synthesis and development of antimicrobial polymers is one of the leading frontiers of research in polymer science [6-8].

In the present study, we described the synthesis of copolymers N-tert-amylacrylamide(NTA) and N-vinyl pyrrolidone were prepared by free radical polymerization in Dioxane medium at 70^{0}C using AIBN as initiator. The copolymer were 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 anhydrousCaCl2. 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.

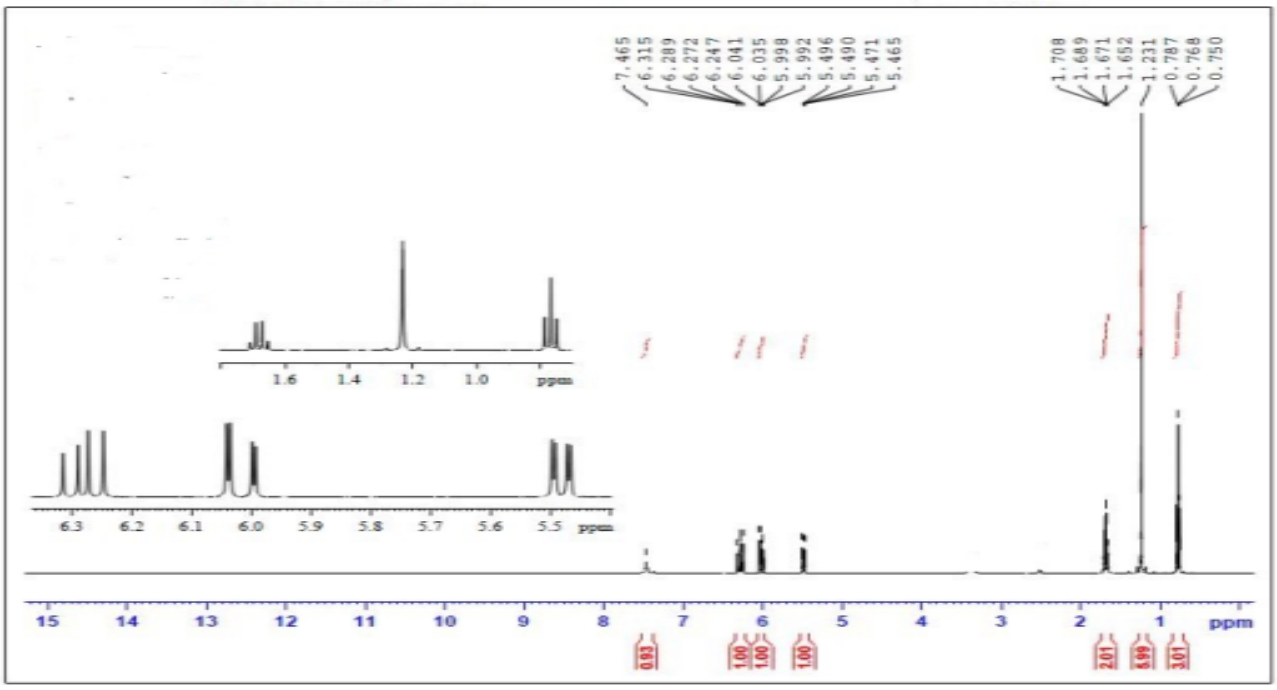
2.1. Preparation of N-tert-amylacrylamide (NTA)

The monomer N-tert-amylacrylamide was prepared by the reaction Amyl alcohol with acrylonitrile. N-tert-amylacrylamide was recrystallized in warm dry benzene. The white crystals have a mp.91° C and the yield was 87%.The monomer was confirmed by both ^{1}H-NMR and ^{13}C-NMR.

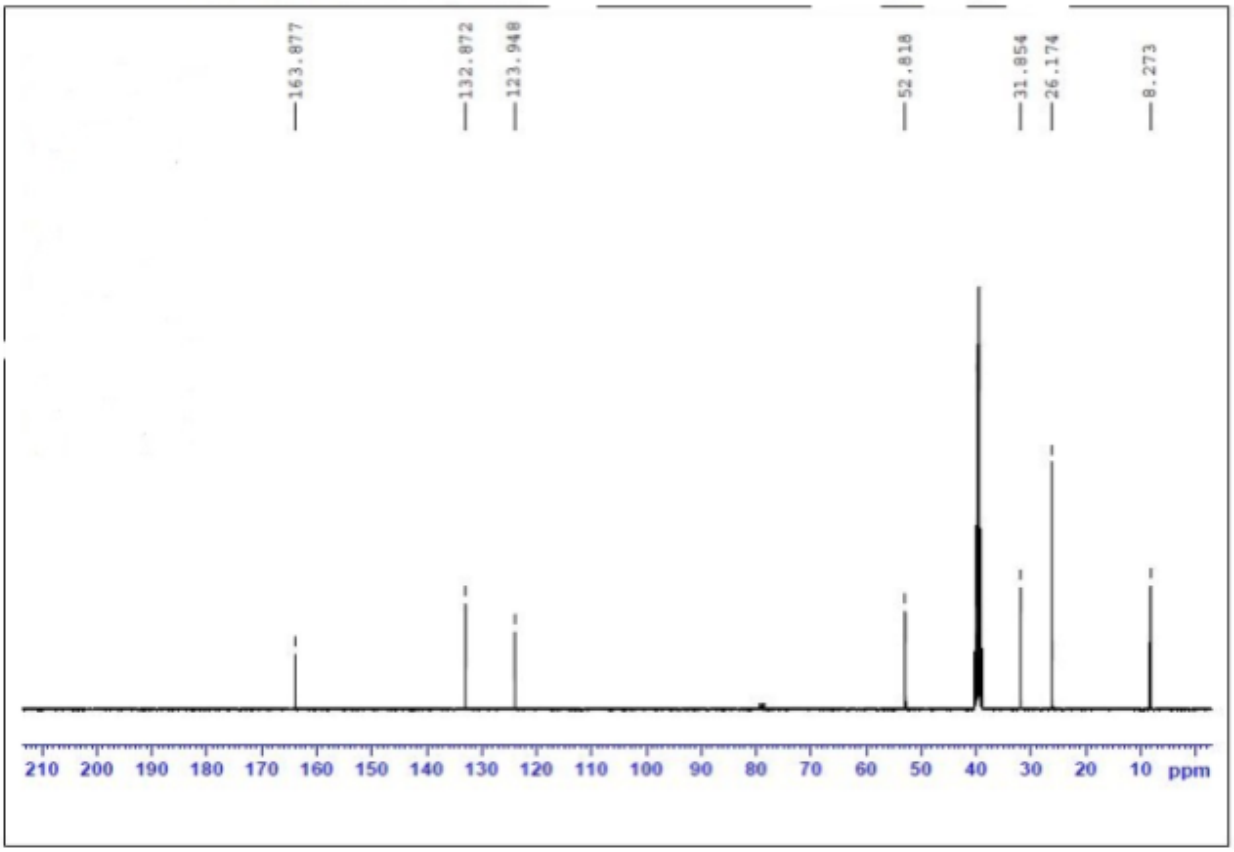
2.2 .^{1}H-NMR spectroscopy

The ^{1}H-NMR spectra of monomers and copolymers were recorded on the GSX-400 spectrometer (JEOL, Tokyo, Japan) operating at 400 MHz respectively in $CDCl_3$.
The following peaks appear in NTA spectrum; at 0.78ppm for -CH_3, at 1.2ppm for -$(CH_3)_2$, at 1.7ppm for -CH_2, at 5.49 ppm for =CH vinyl proton , at 6.2ppm for vinyl =CH_2 proton and –NH at 7.4ppm.

^{13}C-NMR($CDCl_3$), δ(ppm)
δ163.90(CH_2 = C(H)-CO-NH..);
δ 132.93(CH_2 =C(H)-CO-NH…);
δ 123.87(CH_2=C(H)-CO-NH..) ;
δ 52.82(-CO-NH-C(CH_3)-CH_2);
δ 31.87(-CO-NH-C(CH_3)$_3$-CH_2-CH_3);
δ 26.19(-CO-NH-C(CH_3)$_2$-CH_2-CH_3);
δ 8.26(-C(CH_3)$_2$-CH_2-CH_3);



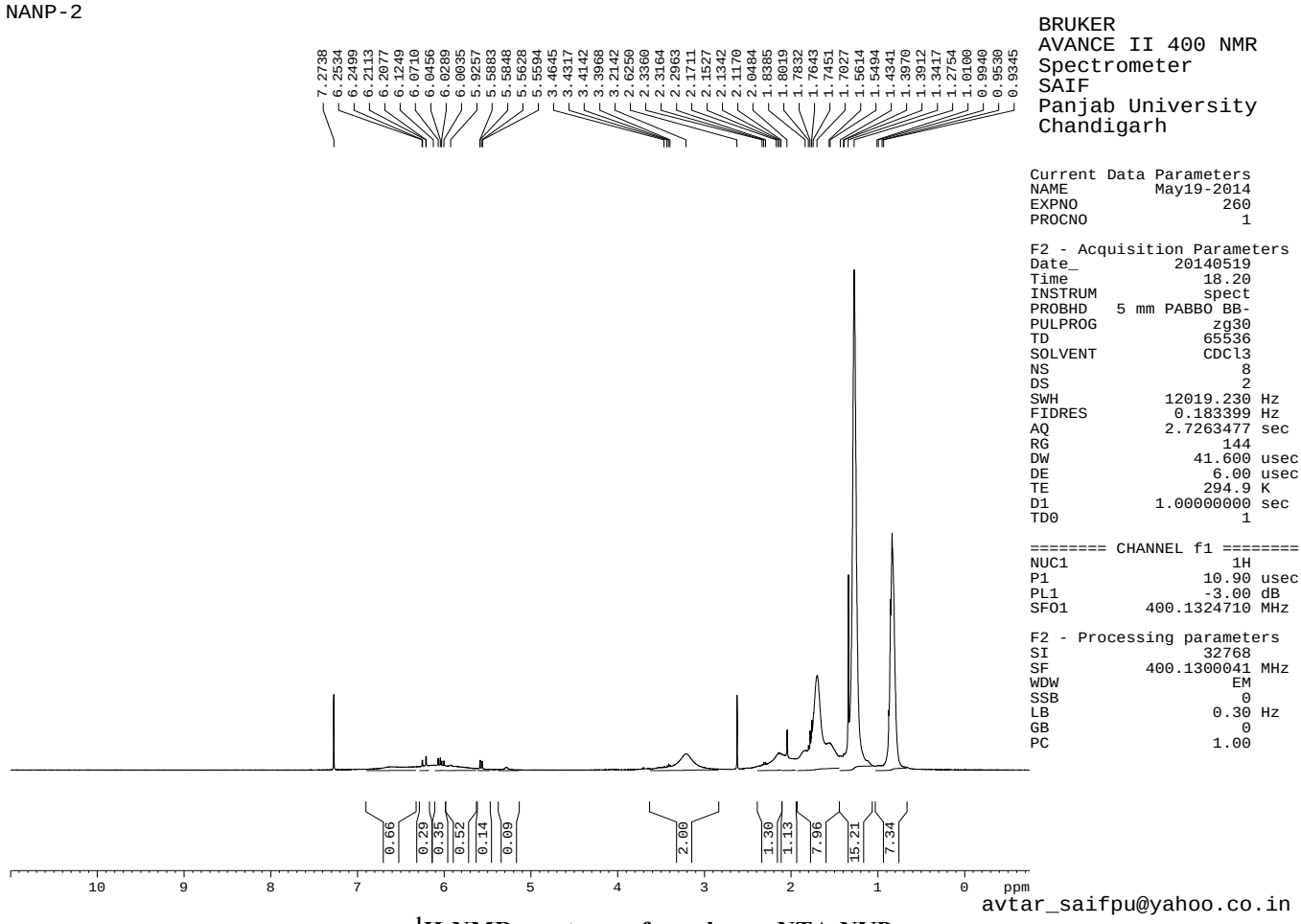
[1]H-NMR spectrum of N-tert-amylacrylamide



[13] C- NMR spectrum of N-tert-amyl acrylamide

2.3. Copolymerization

A total feed of 5g of monomers N-tert-amylacrylamide, 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.



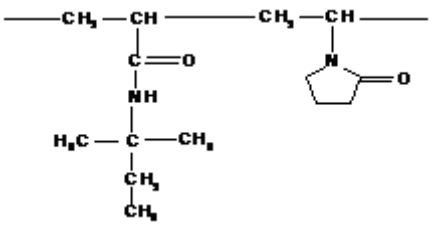
^{1}H-NMR spectrum of copolymer NTA-NVP

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-amylacrylamide (NTA) and NVP were prepared by free radical polymerization in Dioxane medium at 70^0C using AIBN as initiator The schematic representation of the copolymer is given below:



Scheme 1: Copolymerization of NTA 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 noticed that the activity of polymers against bacteria increases with increasing mole % of NTA. These polymers are more active against bacteria.

	Zone of Inhibition in mm	
Mole fraction of NVP	Gram Positive	Gram negative
0.3	20	11
0.5	25	13
0.7	25	18

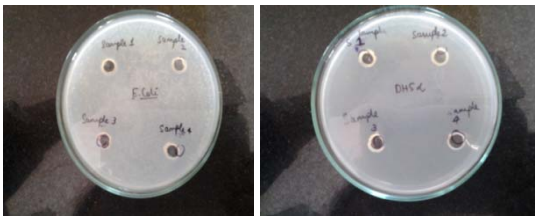


Figure 1: Antibacterial analysis of polymers

4. Conclusion

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

References

[1] Pazhanisamy .P and Anwaruddin.Q , Journal of polymer Science; Part A: Polymer Chemistry, 35 193-195 (1997).
[2] Vogl, O.; Albertsson, A. C.; Jariovic, Z. *Polymer* 26, 1288(1985).
[3] Vijayaraghavan, R.; Mackfarlane, D. R. *Eur. Poly.J.*, 429(10), 2736 (2006).
[4] Erol, I.; Soykan, C. *J. Macromol. Sci., Part A:Pure and Appl. Chem.* 39, 405 (2002).
[5] Nichfor M., Zhu X. X., Polymer, 44, 3053–3060(2003)
[6] Leroux J., Roux E., Le Garrec D., Hong K.,Drummond D. C, Journal of Controlled Release, 72,71– 84 (2001).
[7] Kurisawa M., Yokayama M., Okano T, Journal of Controlled Release, 69, 127–137 (2000).
[8] Kofukuta E., Advances in Polymer Science, 110,157–176 (1993).

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