Research Paper Chemistry

N-Cyclohexylacrylamide Based Copolymers: Synthesis and Antimicrobial Studies of Poly(N-Cyclohexylacrylamide -CO- N-VINYL Pyrrolidone)

R.krithika	Research and Development Centre, Bharathiar University. Coimbatore, India.
P. Pazhanisamy	Department of Chemistry, SirTheagaraya College, Chennai-600 021, India.
P.Jeyanthi	Department of Chemistry, Bharathi women's College, Chennai-600 108 , India.

ABSTRACT

A series of copolymers N-cyclohexylacrylamide(NCA) and N-Vinyl Pyrrolidone(NVP) were prepared by free radical polymerization in Dioxane medium at 700C using AIBN as initiator. The copolymers were characterized by 1H-NMR spectroscopy and the copolymer compositions were determined by1H-NMR analysis. It shows antimicrobial activity. The activity of copolymers against Gram positive and Gram negative also determined by Well-difussion method.

KEYWORDS free radical polymerization, copolymer composition, antimicrobial activity

Introduction

Contamination by microorganism is of great concern in several areas such as medical devices, health care products, water purification systems, hospital and dental equipments etc. One possible way to avoid the microbial contamination is to develop antimicrobial agents. 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[1-7].

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, increasingtheir efficiency and selectivity, and prolonging the lifetime of the antimicrobial agents. In the present study, we described the synthesis of copolymers N-cyclohexylacrylamide(NCA) and N-vinyl pyrrolidone(NVP) were prepared by free radical polymerization in Dioxane medium at 70^0C 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.

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 5^0C.

Preparation of N-cyclohexylacrylamide (NCA)

The monomer N-cyclohexylacrylamide was prepared by the reaction of cyclohexanol with acrylonitrile. N-cyclo hexylacrylamide was recrystallized in warm dry benzene. The white crystals have amp.115° C and the yield was 87%. The monomer was confirmed by both ^{1}H-NMR and ^{13}C-NMR.

^{1}H-NMR spectroscopy

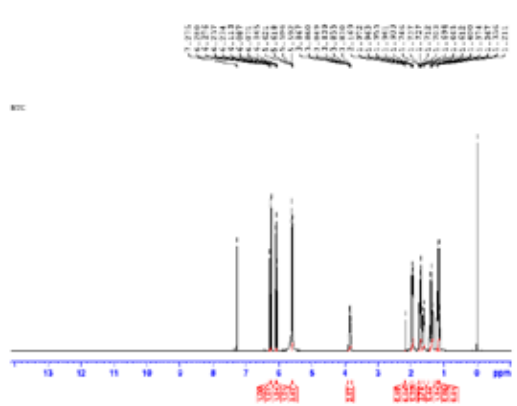
The ^{1}H-NMR spectra of monomers and copolymers were recorded on the GSX-400 spectrometer(JEOL,Tokyo,Japan) operating at 400 MHz respectively in $CDCl_3$. The following peaks appear in NCA spectrum;

at 1.2- 1. 9 ppm for cyclohexyl CH_2 ,at 3.84

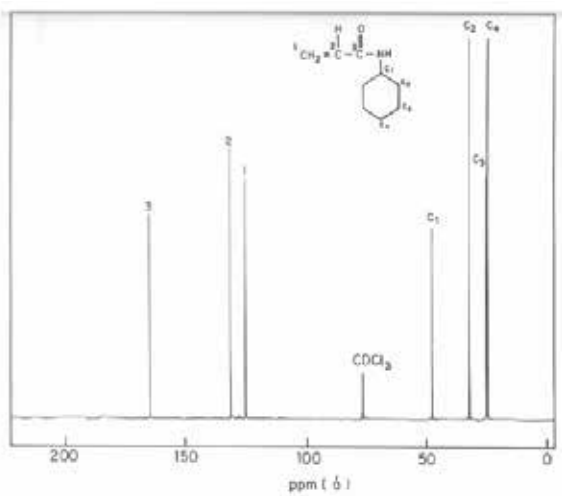
ppm for cyclohexyl methane , at 5.59-6.28 ppm for vinyl protons and at 7.27 ppm for N-H proton.

^{13}C-NMR($CDCl_3$),δ(ppm) :

δ 164.80(CH_2 = C(H)-CO-NH..);
δ 132.93(CH_2 =C(H)-CO-NH…);
δ 122.82(CH_2=C(H)-CO-NH..) ;
δ 49.82(cyclohexyl- C_1)
δ 32.84(cyclohexyl- C_2);
δ 26.19 cyclohexyl- C_3)
δ 26..17 cyclohexyl- C_4)



^{1}H-NMR spectra of N-cyclohexylacrylamide

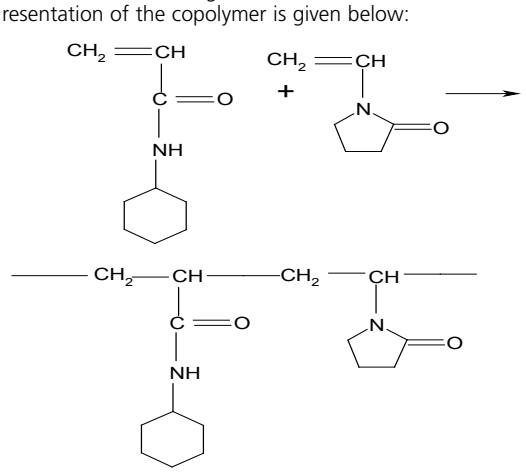


[13] C-NMR spectra of N-cyclohexylacrylamide

Copolymerization:
A total feed of 5g of monomers N-cyclo hexyl acrylamide ,N-Vinylpyrrolidone 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.

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 105 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

Results and Discussion
A series of copolymers N-cyclohexyl acrylamide (NCA) and NVP werepreprared 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 NCA and NVP

Antifungal activity
These polymer samples were tested against the Gram positive and Gram negative at various concentrations as mentioned in Table1 and Figure 1. From the table-1 it noticed that the activity of polymers against bacteria increases with increasing mole % of NVP. These polymers are more active against bacteria.

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

Table 1. Antibacterial analysis of polymers

Mole fraction of NVP in feed	Zone of Inhibition	
	Gram Positive	Gram Negative
0.3	20	13
0.5	23	17
0.7	25	18

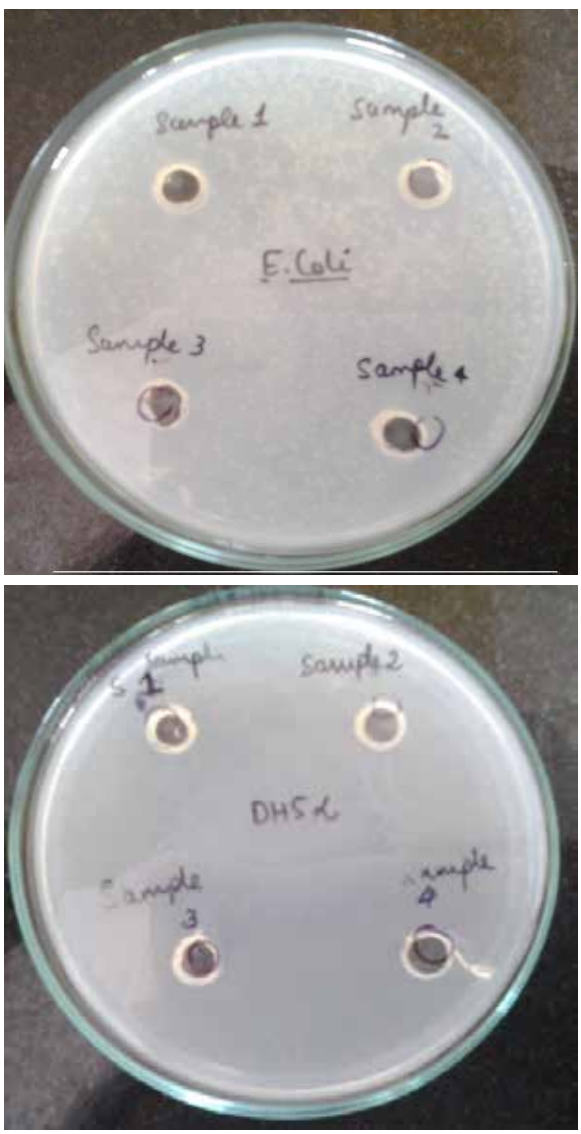


Figure 1. Antibacterial analysis of polymers

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. Polymer26, 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)[5] | Leroux J., Roux E., Le Garrec D., Hong K.,Drummond D. C, Journal of Controlled Release, | 72,71– 84 (2001). | [6] Kurisawa M., Yokayama M., Okano T, Journal of Controlled Release, 69, 127–137 (2000). | [7] KofukutaE.,Advances in Polymer Science, 110,157–176 (1993). |