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SYNTHESIS, CHARACTERIZATION AND ANTIMICROBIAL STUDIES OF POLY N-TERT-AMYLACRYLAMIDE (NTA) AND (PA) COPOLYMERS

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ABSTRACT: *Copolymers of N-tert-amyl acrylamide (NTA) and copolymerized with Phenyl acrylate (PA). Copolymers of N-tert-amyl acrylamide (NTA) and Phenyl acrylate(PA) were prepared by free radical polymerization in DMF at 60^{0}C using AIBN as an initiator. (Scheme 1). The copolymer composition were determined by ^{1}H-NMR analysis. The reactivity ratios of monomers were determined by Fineman-Ross (FR) (r1 = 1.33 and r_2 = 0.60), Kelen-Tudos (KT) (r1 = 1.33and r_2 = 0.59). The $r_1.r_2$ = 0.798 indicated the formation of random copolymers. T_g found to increasing feed content of PA. The antimicrobial studies showed that the copolymers are active against both Bacteria and Fungi.*

Keywords: *N-tert-amyl acrylamide, Phenyl acrylate, reactivity ratios, copolymer composition.*

INTRODUCTION

The determination of copolymer composition and reactivity ratios of the monomers is important in evaluating the specific application of copolymer[1]. The monomer reactivity ratios determined by conventional linearization methods are not always accurate and several non-linear methods have been attempted to determine their [2-4] . ^{1}H-NMR spectroscopic analysis has been established as a powerful tool for the estimation of copolymer composition[5,6] . Knowledge of the copolymer composition is an important step in the evaluation of its utility. Copolymer composition and monomer distribution in the copolymer are dependent on the reactivity ratios. The most common mathematical model of copolymerization is based on finding the relationship between the composition of copolymers and the composition of the monomer feed in which the monomer reactivity ratios are the parameters to be determined[7]. The accurate estimation of copolymer composition and determination of monomer reactivity ratios are significant for tailor-made copolymers with required physical and chemical properties and in evaluating the specific and application of the copolymers. The present article reports the synthesis and characterization of copolymers of N-tert-amyl acrylamide with Phenyl acrylate.

Synthesis of antimicrobial polymers is one of the leading frontiers of research in polymer science. With this view our work N-tert-amyl acrylamide was copolymerized with Phenyl acrylate with different feed ratio were prepared and characterized by ^{1}H-NMR spectroscopy. The reactivity ratios of monomers were determined by Fineman-Ross (FR), Kelen-Tudos (KT) methods.

Experimental

Preparation of N-tert-amyl acrylamide (NTA)

The monomer N-tert-amyl acrylamide was prepared by the reaction of tert-amyl alcohol with acrylonitrile. N-tert-amyl acrylamide was recrystallized in warm dry benzene. The white crystals have amp.91° C (Lit.91-92° C) and the yield was -87%. The monomer was confirmed by both ^{1}H- NMR and ^{13}C-NMR.

Copolymerization of NTA and PA

Copolymers of N-tert-amyl acrylamide (NTA) and Phenyl acrylate (PA) were prepared by free radical polymerization in DMF at 60^{0}C using AIBN as an initiator.(Scheme 1).

Characterization of copolymer spectra of Poly (NTA-co- PA)

The ^{1}H-NMR spectra of copolymers, poly (NTA-co-PA) is shown in Figure 1. The following peaks appear in the copolymer spectrum : at 1.1 -2.9 ppm for CH_2 group , at 2.7 ppm for backbone CH_2 , at 6.8-7.4 ppm due to PA aromatic protons and 7.7 for NH proton of NTA.

Determination of copolymer composition of Poly (NTA-co- PA)

The phenyl acrylate area is used to determine the copolymer composition. Resonance signal at 6.8-7.4 ppm corresponds to aromatic proton, and their integrated intensity of this peak is compared to the total intensities of all the peaks in the copolymer spectrum, which is a measure of their relative areas.
The copolymer compositions can be obtained using

$$X_{PA} = \frac{15A\ (aryl)}{5A_{total} + 7A(aryl)} \quad \text{------ (1)}$$

Where X= mole fraction and A= peak area.

Determination of Reactivity ratios

The reactivity ratios value for NTA (r_1) and PA (r_2) from the F-R plot (Figure 2), K-T plot (Figure 3) are given in Table3. The value of r_1 is greater than1 and r_2 is less than1. The system is lying in the range $r_2 < 1$, $r_1 > 1$. As $r_1.r_2$ is towards unity, the sequence of both monomeric units in copolymer chain is at random.

Thermal studies of Poly (NTA-co- PA)

TGA curves shows two stage decomposition regions for Poly (NTA-co-PA). The initial weight loss region appears around -100˚C due to moisture content. Weight loss at stage 1 is associated with dehydration of partially degraded amide groups; Maximum weight loss occurs at stage 2 around 300-400˚C indicates the main chain degradation and degradation of the polymer backbone. The DSC analysis indicates that the T_g values increases with increasing the content of PA in feed are given in table 4.

Antimicrobial Activity of Poly (NTA-co- PA)

Antimicrobial analysis was followed using standard agar well diffusion method to study the antimicrobial activity of compounds antimicrobial activity was evaluated by measuring the diameter of the zone of inhibition against the test microorganisms. DMSO was used as solvent control. Chloramphenicol was used as reference antibacterial agent. Amphotericin - B was used as reference antifungal agent. The tests were carried out in triplicates. The results are shown in Figure 6, the values are given in Table 5 and 6. The antimicrobial studies showed that these polymers are active against both bacteria and Fungi. Copolymers showed more activity than the control.

Scheme 1: Copolymerization of NTA and PA

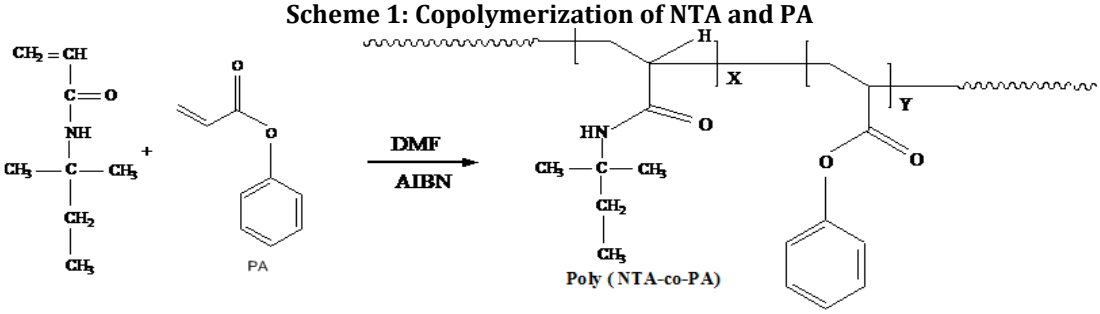


Figure 1:^{1}H-NMR spectrum of poly (NTA-co- PA) (a) 0.5:0.5

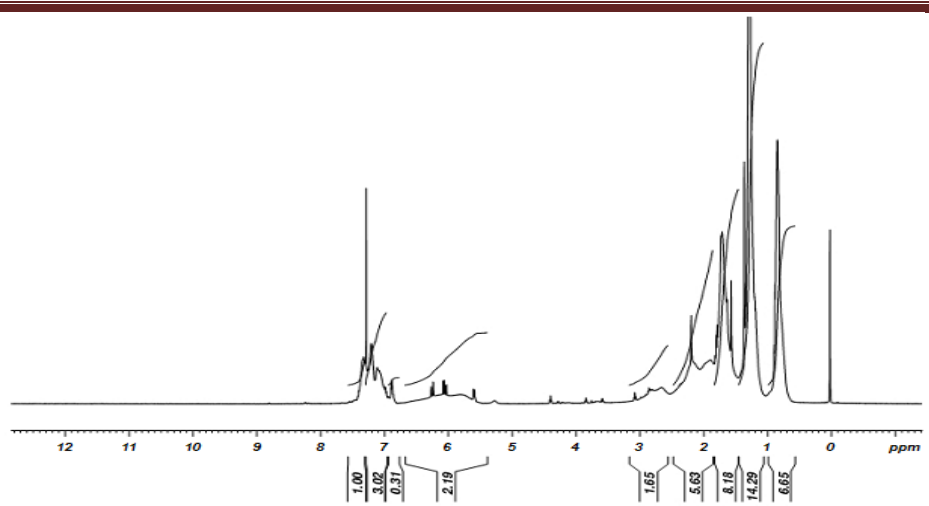


Figure 2 : Fineman-Ross plot for Poly (NTA-co- PA)

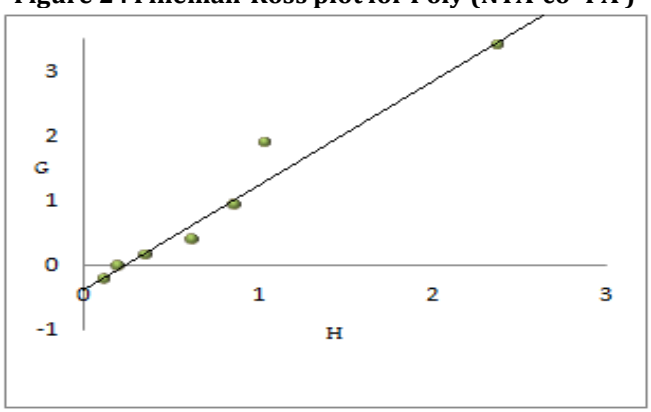


Figure 3: Kelen-Tudos plot for Poly (NTA-co- PA)

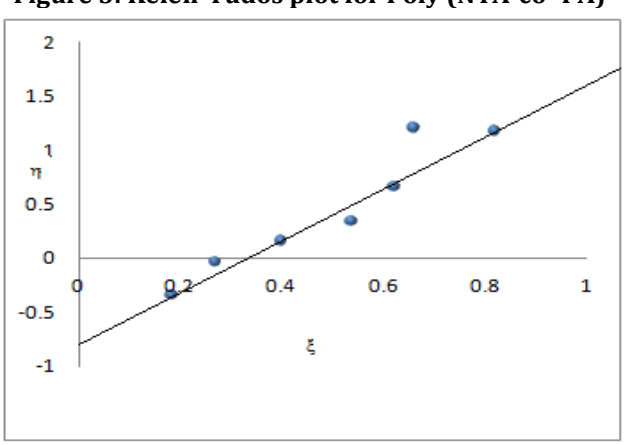


Table 1: Mole fractions of the copolymers of N-tert-amylylacrylamide (NTA) and (PA)

Mole fraction of NTA in feed, M_1	Mole fraction of PA in feed, M_2	Mole fraction of NTA in copolymer, m_1	Mole fraction of PA in copolymer, m_2	$F=M_1/M_2$	$f=m_1/m_2$
0.2	0.8	0.3450	0.6550	0.2500	0.5267
0.3	0.7	0.4850	0.5150	0.4286	0.9417
0.4	0.6	0.5588	0.4412	0.6667	1.2665

0.5	0.5	0.6200	0.3800	1.0000	1.6316
0.6	0.4	0.7224	0.2776	1.5000	2.6023
0.7	0.3	0.8398	0.1602	2.3333	5.2422
0.8	0.2	0.8708	0.1292	4.0000	6.7399

Table 2: Fineman-Ross and Kelen – Tudos parameters for the Copolymers of NTA and PA

G = F(f-1)/f	H=F²/f	η=G/(α+H)	ξ=H/(α+H)
-0.2247	0.1187	-0.3463	0.1829
-0.0266	0.1954	-0.0366	0.2694
0.1404	0.3513	0.1593	0.3986
0.3871	0.6129	0.3387	0.5363
0.9236	0.8646	0.6622	0.6200
1.8880	1.0383	1.2038	0.6621
3.4064	2.3746	1.1727	0.8175

α=0.53

Table 3: Reactivity ratios of NTA (r_1) and PA (r_2)

Methods	r_1	r_2	$r_1.r_2$
Fineman-Ross	1.60	0.41	0.65
Kelen-Tudos	1.55	0.41	0.64

Table 4: TGA data for Poly (NTA-co- PA)

Copolymers	Mole fraction of NTA, in feed	Mole fraction of PA, in feed	Mole fraction of PA, in copolymer	IDT (°C)	T_{50} (°C)	T_f (°C)	T_g (°C)
NTA-PA	0.70	0.30	0.1602	120	361	666	120
NTA-PA	0.50	0.50	0.3800	127	378	677	150
NTA-PA	0.30	0.70	0.5150	162	383	711	160
Poly-NTA		-	-	-	-		86.2

IDT: Initial Decomposition Temperature
T_{50} : decomposition temperature at 50% weight loss
T_f : final decomposition temperature
T_g : glass transition temperature

Table 5 : Anti bacterial activity of Poly (NTA-co- PA)

Sl. No	Copolymers	*Staphylococcus aureus(mm)*	*Escherichia coli (mm)*	*Pseudomonas aeruginosa (mm)*
1.	0.3 NTA:0.7 PA	14	No zone	17
2.	0.5 NTA:0.5PA	18	No zone	22
3.	0.7 NTA:0.3 PA	19	No zone	23
4.	Chloramphenicol	26	28	10

Table 6 : Anti fungal activity of Poly (NTA-co- PA)

Sl. No	Copolymers	*Aspergillus niger (mm)*	*Candida albicans (mm)*	*Candida tropicalis (mm)*
1.	0.3 NTA:0.7 PA	11	08	07
2.	0.5 NTA:0.5 PA	24	13	20
3.	0.7 NTA:0.3 PA	21	09	20
4.	Amphotericin - B	16	17	18

Figure 4 : Comparison of Zone of inhibition of Poly (NTA-co- PA)with bacteria

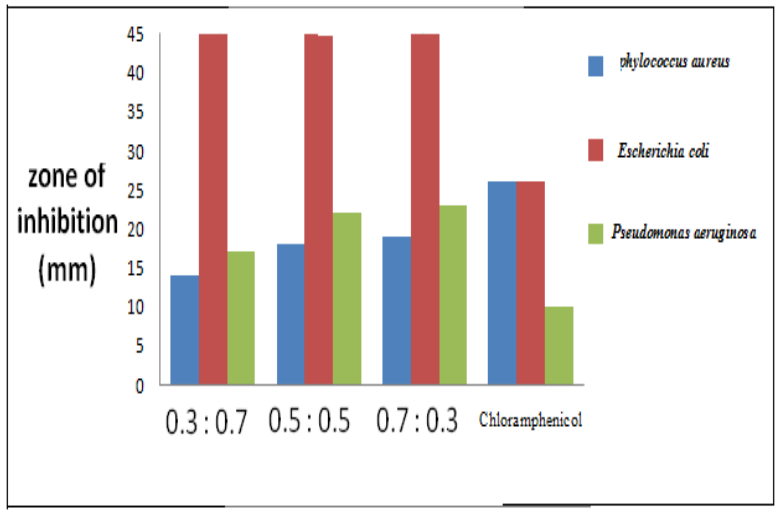


Figure 5 : Comparison of Zone of inhibition of Poly (NTA-co- PA)with fungi

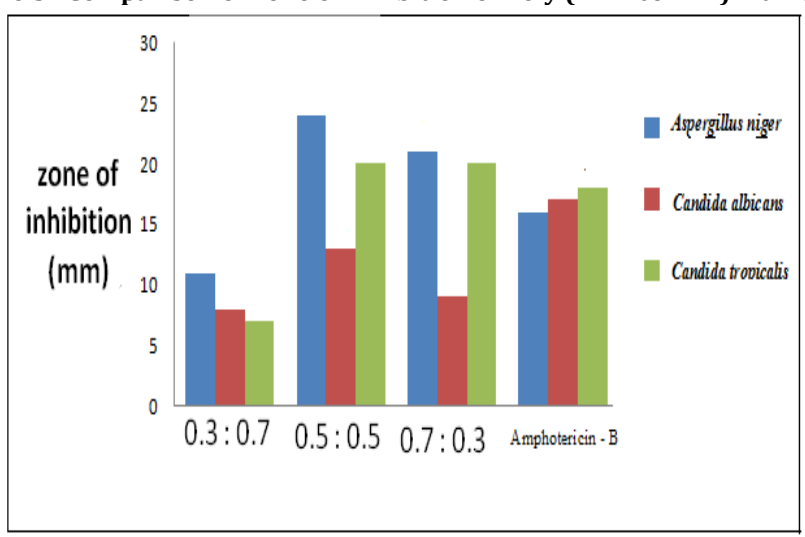
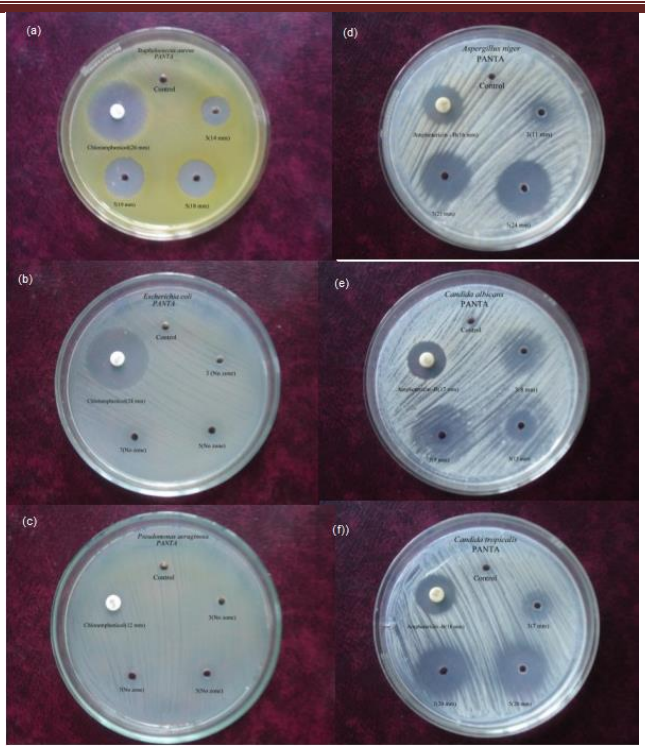


Figure 6: Antimicrobial studies of Poly (NTA-co- PA)



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