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Acrylic Copolymers Based on Phenol: Synthesis, Characterization and Antimicrobial Activity

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Copolymers of 2,4-dichlorophenyl acrylate (2,4-DPA) and 4-chlorophenyl acrylate (PCPA) were synthesized with different monomer feed ratios using N, N-dimethylformamide as a solvent and 2,2'-azobisisobutyronitrile (AIBN) as an initiator at 70°C. Copolymers of different molecular weight having different proportion of the monomers were prepared. The copolymers were characterized by Infrared Red spectroscopy and copolymer composition was determined with UV-spectroscopy. The linearization methods of Fineman-Ross and Kelen-Tudos were employed to obtain the monomer reactivity ratios. The molecular weight and polydispersity index were determined by gel permeation chromatography (GPC). Thermogravimetric analysis of polymers was carried out in nitrogen atmosphere. The homo and copolymers were tested for their antimicrobial properties against selected microorganisms and it was observed that depending on the chlorine content of the polymers the antimicrobial activities differ.

Keywords: 2,4-dichloro phenyl acrylate; 4-chloro phenyl acrylate; copolymer; antimicrobial activity

1 Introduction

Acrylic polymers are a class of reactive polymers that find extensive applications due to the presence of electron attracting groups in the aromatic ring (1). Acrylate homopolymers along with their copolymers are used in various fields such as films, fibers, filament, lithography, lacquers, adhesives, printing inks and binders (2, 3). Studies on phenyl acrylate and phenyl methacrylate polymers have gained considerable importance and synthesis of these polymers is now technologically feasible (4, 5). Nanjundan and co-workers (6, 7) synthesized the copolymers of 4-benzoyloxycarbonylphenyl acrylate (BCPA) and 4-benzoylphenyl methacrylate (BPM) with glycidyl methacrylate (GMA). They have also prepared adhesive based on (BCPA-co-GMA) and (BPM-co-GMA) for the application on leather and reported that these copolymers exhibit good adhesive characteristics.

The activated acrylate polymers find applications in pharmacological drugs as polymer supports. Chlorine containing phenyl methacrylate and its polymer have been used as biocides in various applications (8, 9). Erol and co-workers (10, 11) have prepared new methacrylate monomers, their

derivatives and observed good biological activity for these polymers. Potin and co-workers (12) synthesized acrylate esters, useful in antifungal paints by reacting pentachloro phenol/o-chloro phenol/p-chloro-m-cresol with acryloyl chloride. Soykan and co-workers (13) synthesized copolymers of 2-((5-methoxyisoxazol-3-yl) amino]-2-oxo-ethyl methacrylate with glycidyl methacrylate using 2,2'-azobisisobutyronitrile initiator in 1,4-dioxane solution. They tested antimicrobial activity of these copolymers against different strains of bacteria and fungi and reported that all the copolymer showed moderate activity.

This article discusses the synthesis and characterization of 2,4-dichlorophenyl acrylate (2,4-DPA) and 4-chlorophenyl acrylate (PCPA) monomers, as well as the synthesis of homo and copolymers using different monomer feed ratios. This work was taken up with a view to examining the role of chlorine in the antimicrobial activity of these polymers. The copolymer composition was determined by UV-spectroscopy. The monomer reactivity ratios were determined by linearization method of Fineman-Ross (14) and Kelen-Tudos (15). Molecular weight was determined by gel permeation chromatography, and thermal analysis of polymers are also included in this paper. Homo and copolymers have been characterized for their antimicrobial activity against microorganisms such as bacteria (*Escherichia coli*, *Bacillus subtilis*, *Staphylococcus citreus*), fungi (*Aspergillus niger*, *Sporotrichum pulverulentum*, *Trichoderma lignorum*) and

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yeast (*Candida utilis*, *Saccharomyces cerevisiae*, *Pichia stipitis*).

2 Experimental

2.1 Materials

2,4-dichloro phenol and 4-chloro phenol (S.D. Fine chemicals), 2,2'-azobisisobutyronitrile (AIBN, Aldrich), acrylic acid (Merck) and benzoyl chloride (Chiti Chemical) were used as received. Fractionally distilled solvents were used in the reaction.

2.2 Synthesis of Acryloyl Chloride

Acryloyl chloride was prepared according to the process reported in the literature (16).

2.3 Synthesis of 2,4-Dichlorophenyl Acrylate (2,4-DPA)

Absolute alcohol (200 ml) and NaOH (0.1 mole) were added to a one liter three-necked flask equipped with stirrer, thermometer, and guard tube. The contents were stirred until all the NaOH dissolved and 2,4-dichloro phenol (0.1 mole) was added. The reaction mixture was heated to 60°C for 30 min with stirring and then cooled to room temperature and finally to 0–5°C. Freshly prepared acryloyl chloride (0.11 mole) was added drop wise within 60 min to the cooled reaction mixture. The temperature was maintained around 0–5°C during the addition. After completion of the addition, the reaction mixture was stirred for 90 min, and it was poured into a crushed ice water mixture when a light brown color liquid product settled. It was extracted with ether, and the ether layer was separated out. The liquid monomer obtained after evaporation of ether was dried over anhydride calcium chloride in a vacuum desiccator. The yield was 83%.

2.4 Synthesis of 4-Chlorophenyl Acrylate (PCPA)

This compound was synthesized by the method followed for 2,4-DPA. However, there have been minor variations. To start with, 400 ml absolute alcohol and NaOH (0.2 mole) were taken. 4-Chlorophenol (0.2 mole) was added to the NaOH solution in alcohol and finally acryloyl chloride (0.21 mole) was added drop wise to the mixture. The final product is also liquid. The yield was 73%.

2.5 Characterization of Monomers 2,4-DPA and PCPA

The monomers were characterized by FT-IR and ¹H-NMR spectroscopy. FT-IR spectra of monomers were recorded using a NICOLET 400D FT-IR spectrophotometer. ¹H-NMR spectra of monomers were recorded on a HITACHI-R-1500 FT-NMR spectrometer (60 MHz) using CDCl₃ as solvent and tetramethylsilane as an internal standard.

The IR spectrum of monomer 2,4-DPA is shown in Figure 1(A) and that of PCPA is shown in Figure 2(A). The ¹H-NMR spectra are shown in Figures 1(B) and 2(B) for 2,4-DPA and PCPA, respectively.

2.6 Copolymerization

Copolymers of 2,4-DPA with PCPA having different composition were synthesized by free radical polymerization in DMF solvent using AIBN as a free radical initiator. The feed composition of monomer and comonomer is given in Table 1. Appropriate quantities of monomer, comonomer, DMF (10 ml) and AIBN (1% w/w based on total monomers 1 and 2) were taken in a flask (equipped with mechanical stirrer and reflux condenser) flushed with oxygen free nitrogen gas for 15 min. The reaction mixture was heated at 70°C for 5 h with stirring. It was then cooled to room temperature and the resulting polymer solution was slowly poured in a large volume of methanol with stirring, when the polymer precipitated out. It was filtered and washed

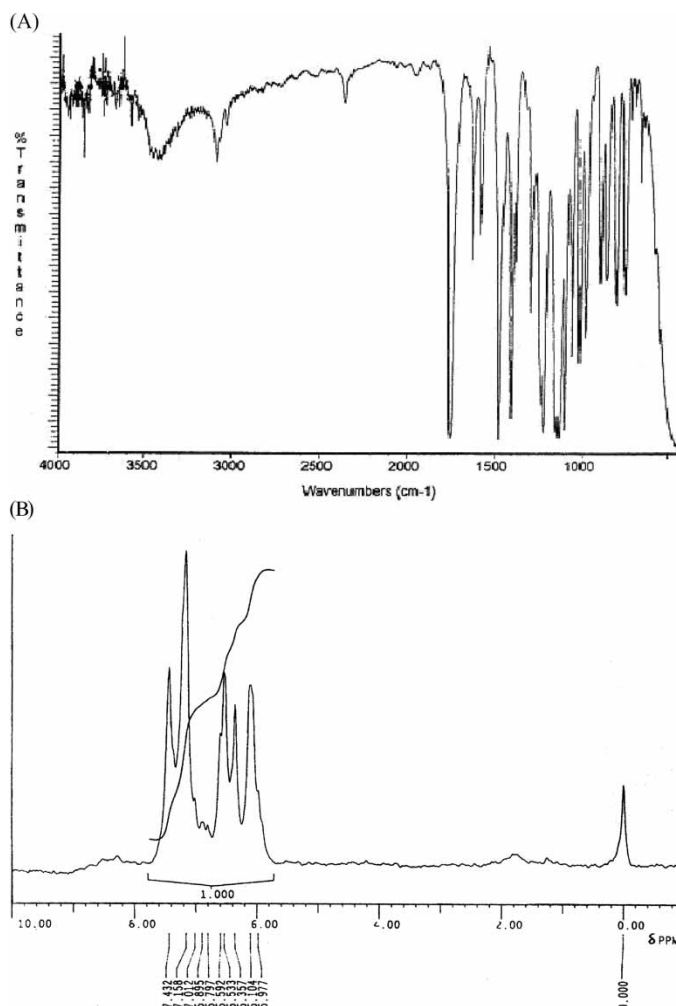


Fig. 1. (A) IR spectrum of monomer 2,4-DPA, (B) ¹H-NMR spectrum of monomer 2,4-DPA.

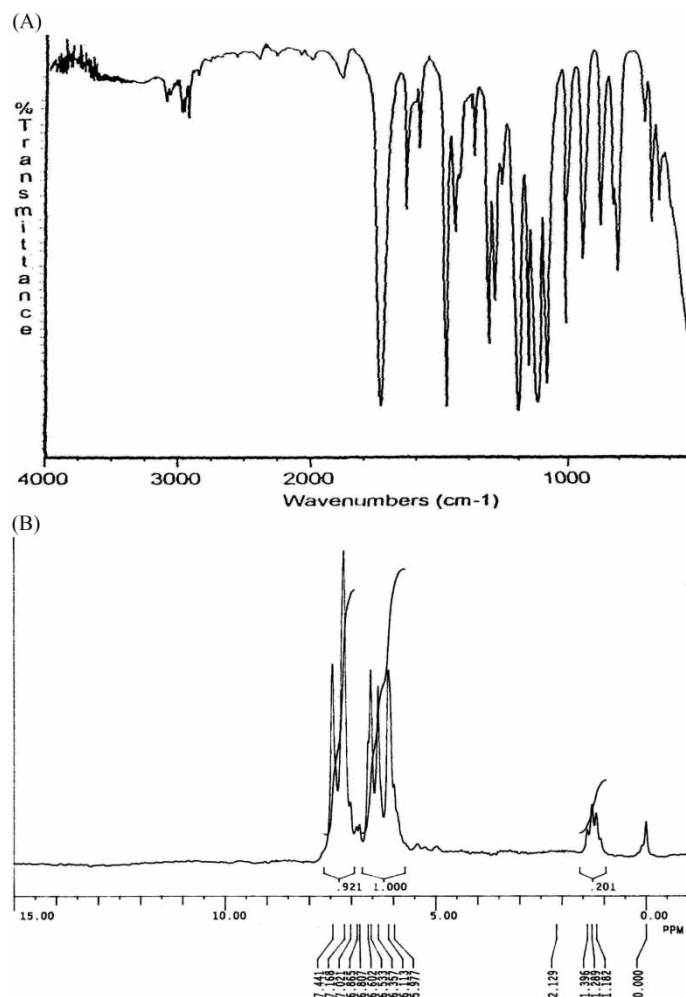


Fig. 2. (A) IR spectrum of monomer PCPA (B) $^1\text{H-NMR}$ spectrum of monomer PCPA.

with methanol. Solid polymers were purified by repeated precipitation by methanol from solution in DMF and finally dried. Scheme 1 shows the reactions leading to the formation of homopolymers as well as copolymers of 2,4-DPA with PCPA.

2.7 Characterization

FT-IR spectra of polymers were recorded on a NICOLET-400_D FT-IR spectrophotometer using a solid KBr pellet method. A Shimadzu-160-A recording UV-visible spectrophotometer was used to determine copolymer composition and reactivity ratios. GPC instrument equipped with a Jasco-PU 1580 pump, multi-solvent delivery system, manual injector and series connected two PL gel column packed with styrenedivinylbenzene bead and R.I. detector (RI-71 shodex made) was employed to determine molecular weights of the polymers. Dimethylformamide (DMF) at 1.0 ml/min flow rate was used as a mobile phase throughout the analysis and polystyrene standards were employed

for calibration. Intrinsic viscosity (η) of the copolymers was measured in toluene solvent at $25 \pm 0.1^\circ\text{C}$ using an Ubbelohde suspension level viscometer. TGA traces were obtained on a TA instrument (USA)- 2960 thermogravimetric analyzer at a heating rate of $10^\circ\text{C min}^{-1}$

2.8 Antimicrobial Activity

The homo and copolymers prepared were tested for their antimicrobial activity against bacterial strain (*Bacillus subtilis*, *Escherichia coli* and *Staphylococcus citreus*), fungal strain (*Aspergillus niger*, *Sporotichum pulverulentum* and *Trichoderma lignorum*) and yeast strain (*Candida utilis*, *Saccharomyces cerevisiae* and *Pichia stipitis*). The bacterial strains were grown in Nutrient broth (N-broth), fungal strains were grown in Sabourand's dextrose broth and yeast extract peptone dextrose (YEPD) was added to the N-broth to grow yeast strains (with or without indicated polymers). The content of the flasks were incubated in a shaker at room temperature. At specific time intervals (20-48 h), the optical density was measured at 660 nm for bacteria and yeast cultures. The inhibition percentage (I) is obtained from the relation:

$$I = \frac{100(X - Y)}{X} \quad (1)$$

Where X is the optical density of bacterial suspension in the control set and Y is the optical density of the bacterial suspension in the test set. It was maximum after 48 h. In the case of fungal strain, optical density method is not used. The suspension containing the fungal cultures were harvested after 48 h, and the weight of the dry cell mass was determined gravimetrically. The percentage inhibition (I) is calculated from Equation (1), where X is the weight of the dry fungal cell mass in control set and Y is the weight of the dry fungal cell mass in the test set. Details of the experimental procedure have already been reported elsewhere (17, 18).

3 Results and Discussion

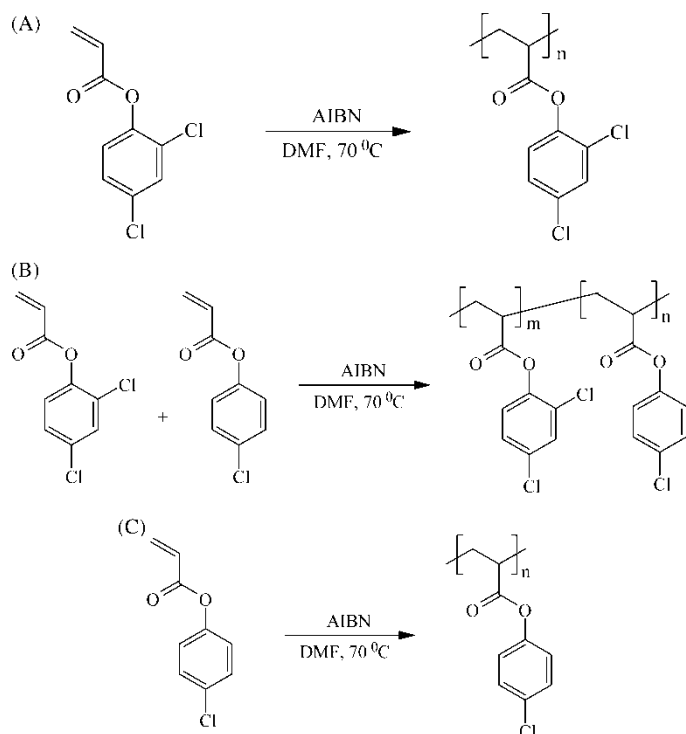
Five copolymers of 2,4-DPA with PCPA were synthesized by taking different mole fraction of the monomers in the feed ranging from 0.2 to 0.8 (Table 1). The copolymers were soluble in dimethylformamide, toluene, chloroform, acetone, tetrahydrofuran, and dimethylsulfoxide, but insoluble in methanol, ethanol, and hexane. The copolymers have been characterized by IR spectral data and the results are discussed below.

The IR spectrum (Figure 2 (A)), of 4-DPA shows $\nu_{\text{C=O}}$ at 1750 cm^{-1} and the $\nu_{\text{C=C}}$ at 1640 cm^{-1} . The C-H out-of-plane bending mode in the vinyl moiety is assigned for the absorption at 890 cm^{-1} and the rocking mode due to this moiety is assigned for the peak at 730 cm^{-1} . As expected, the absorption at 1640, 890 and 730 cm^{-1} associated with the vinyl group are absent after formation of polymers.

Table 1. Composition data, F-R and K-T parameters for copolymers of 2,4-DPA with PCPA

Sample code no.	Monomer feed composition		Conversion (%)	Composition of 2,4-DPA in the copolymer [m ₁]	X	Y	F	G	ξ	η	Reactivity ratios			
	2,4-DPA [M ₁](mole)	PCPA[M ₂](mole)									F-R		K-T	
1	1.0	—	—	—							r₁	r₂	r₁	r₂
2	0.2	0.8	9.15	0.238	0.25	0.313	0.200	-0.549	0.182	-0.500				
3	0.4	0.6	9.78	0.426	0.67	0.743	0.605	-0.232	0.402	-0.155				
4	0.5	0.5	8.35	0.518	1.0	1.075	0.931	0.070	0.509	0.039	0.93	0.75	0.92	0.74
5	0.6	0.4	7.90	0.623	1.5	1.653	1.362	0.593	0.603	0.263				
6	0.8	0.2	10.20	0.798	4.0	3.951	4.050	2.988	0.819	0.604				
7	—	1.0	—	—										

Where, $m_2 = 1 - m_1$; $X = M_1/M_2$; $Y = m_1/m_2$; $F = X^2/Y$; $G = X(Y - 1/Y)$; $\xi = F/\alpha + F$; $\eta = G/\alpha + F$ and $\alpha = [F_M \cdot F_m]^{1/2}$.



Sch. 1. (A) 2,4-DPA homopolymer, (B) Poly(2,4-DPA-co-PCPA), (C) PCPA homopolymer.

The homopolymer p(2,4-DPA) shows strong absorption at 1765 cm^{-1} due to $\text{C}=\text{O}$ stretching in ester moiety (19). The $\text{C}-\text{O}-\text{C}$ stretching vibration appears in the $1200\text{--}1223\text{ cm}^{-1}$ region, and the band at 670 cm^{-1} is attributed to $\nu_{\text{C}-\text{Cl}}$ (20).

The $^1\text{H-NMR}$ of 2,4-DPA (Figure 1(B)) shows the following resonance (δ ppm) 5.977, (1H, $-\text{CH}=\text{C}$), 6.357 (1H, $-\text{CH}=\text{C}$), 6.533 (1H, $-\text{CH}=\text{C}$) (i.e., three non-equivalent methylene protons) and 7.158–7.432 (3H, aromatic protons). The aromatic protons are assigned around 7.012–7.344. The formation of polymer is evidenced from the disappearance of signals at 5.977, 6.357, and 6.533 and the appearance of signals at 3.125 (1H, $-\text{CH}$) and 2.129–2.178 (2H, $-\text{CH}_2$).

The IR spectrum (Figure 2(A)) of PCPA shows $\nu_{\text{C}=\text{O}}$ at 1739 cm^{-1} and $\nu_{\text{C}=\text{C}}$ at 1642 cm^{-1} . The CH out-of-plane bending in the vinyl moiety is assigned to a peak at 947 cm^{-1} , while the rocking mode is attributed at 877 cm^{-1} . The $\text{C}-\text{O}-\text{C}$ stretching frequency in PCPA is possibly distributed in the frequencies at 1203 and 1163 cm^{-1} . The $\nu_{\text{C}-\text{Cl}}$ is assigned to the peak at 675 cm^{-1} .

On polymerization, the peaks associated with vinyl moiety i.e., $\nu_{\text{C}=\text{C}}$ at 1642 cm^{-1} , CH out-of-plane bending at 947 cm^{-1} and rocking at 877 cm^{-1} disappear. The other frequencies remain more or less the same as in the monomer.

The copolymer (2,4-DPA-co-PCPA) shows two strong absorption due to $\nu_{\text{C}=\text{O}}$ at 1750 and 1739 cm^{-1} . Depending on the composition of the polymers, the relative intensities of these peaks vary.

$^1\text{H-NMR}$ of PCPA (Figure 2(B)) shows the following resonances: δ 6.318 (1H), δ 5.732 (1H), (non-equivalent methylene proton), δ 7.119–7.275 (4H, aromatic protons). The two non-equivalent $\text{H}-\text{C}=\text{C}$ protons are further deshielded due to the presence of a phenyl ring.

3.1 Copolymer Composition and Reactivity Ratios

The composition of the copolymers of 2,4-DPA and PCPA was obtained from the UV-spectral data (Figure 3). The reactivity ratios of 2,4-DPA and PCPA were determined by the Fineman-Ross (14) method from monomer feed ratios and copolymer compositions and are shown in Table 1. The Kelen-Tudos (15) method was also employed to obtain the reactivity ratio and these are shown in Table 1. The reaction time was selected in such a way that conversion was less than 10% in weight. This was done to satisfy the differential copolymerization equation for calculation of reactivity ratio. (14) The equation involved in the Fineman-Ross method is:

$$F(F-1)/f = (F^2/f)r_1 - r_2$$

Where F is the ratio of mole fraction of monomer 1 (M_1) to the mole fraction of monomer 2 (M_2) in the feed, f is the ratio of mole fraction of monomer 1 (m_1) to the mole fraction of monomer 2 (m_2) in the copolymer. A plot of $F(F-1)/f$ against F^2/f gives a straight line with a slope equal to r_1 and intercept equal to r_2 .

In the Kelen-Tudos method, the equation is:

$$\eta = (r_1 + r_2/\alpha)\xi - r_2/\alpha$$

Where $\eta = G/(\alpha + H)$, $\xi = H/(\alpha + H)$, $G = F(F-1)/f$, $H = F^2/f$ and α is the geometric means of the minimum and maximum H values. By plotting η against ξ , a straight line is obtained which when extra plotted to $\xi = 0$ and $\xi = 1$ gives $-r_2/\alpha$ and r_1 , respectively. The values of G , H , η and ξ for copolymers are given in Table 1. The $F-R$

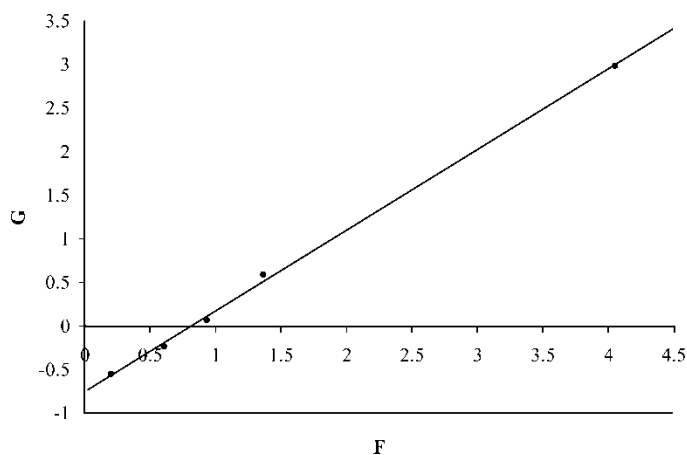


Fig. 3. FT-IR spectra of 2,4-DPA-co-PCPA homo and copolymers.

and K-T plots obtained by linear regression analysis for poly(2,4-DPA-co-PCPA) are given in Figures 4(A) and (B), respectively. The r_1 and r_2 values of copolymer are shown in Table 1. In poly(2,4-DPA-co-PCPA), the value of r_1 is less than r_2 . In this system, PCPA is found to have slightly

higher reactivity than 2,4-DPA. The product of $r_1 r_2$ is less than one, which indicates that the system follows a random distribution of monomeric unit.

When r_1 and r_2 are less than one, the system gives rise to azeotropic polymerization at a particular composition of the monomer, which is calculated using the equation (21):

$$N_1 = \frac{(1 - r_2)}{(2 - r_1 - r_2)} = 0.781$$

where N_1 is the mole fraction of 2,4-DPA in the feed.

When the mole fraction of the monomer 2,4-DPA in the feed is 0.781, the copolymer formed will have the same composition as that in the feed. When the mole fraction of feed is less than 0.781 with respect to 2,4-DPA, the copolymer is relatively richer in this monomeric unit. When the mole fraction of the monomer 2,4-DPA in the feed is above 0.781, the copolymer is relatively richer in PCPA monomeric units.

3.2 Molecular Weights and Viscosity Measurements

The number and weight average molecular weights of homo and copolymers of 2,4-dichlorophenyl acrylate with 4-chlorophenyl acrylate were obtained from gel permeation chromatography. The values of number average and weight average molecular weight range from 9809 to 32380 and 15970 to 48640, respectively whereas, polydispersity index varied in the range of 1.502 to 1.638. Intrinsic viscosity lies in the range 0.090 to 0.210 dl · g⁻¹ (Table 2). These data clearly indicates that as 2,4-DPA content in the copolymer increases, the molecular weight and viscosity increases.

3.3 Thermal Analysis

Thermogravimetric analysis was carried out to study the thermal stability of poly(2,4-DPA), poly(2,4-DPA-co-PCPA) (20 : 80, 50 : 50, 80 : 20 monomer feed ratio) and poly(PCPA). The results of TGA analysis of homo and copolymers are presented in Table 3. The data clearly indicate that all polymers undergo single step decomposition in the temperature range of 191–428°C. These temperatures did not show any definite trend as a function of copolymer

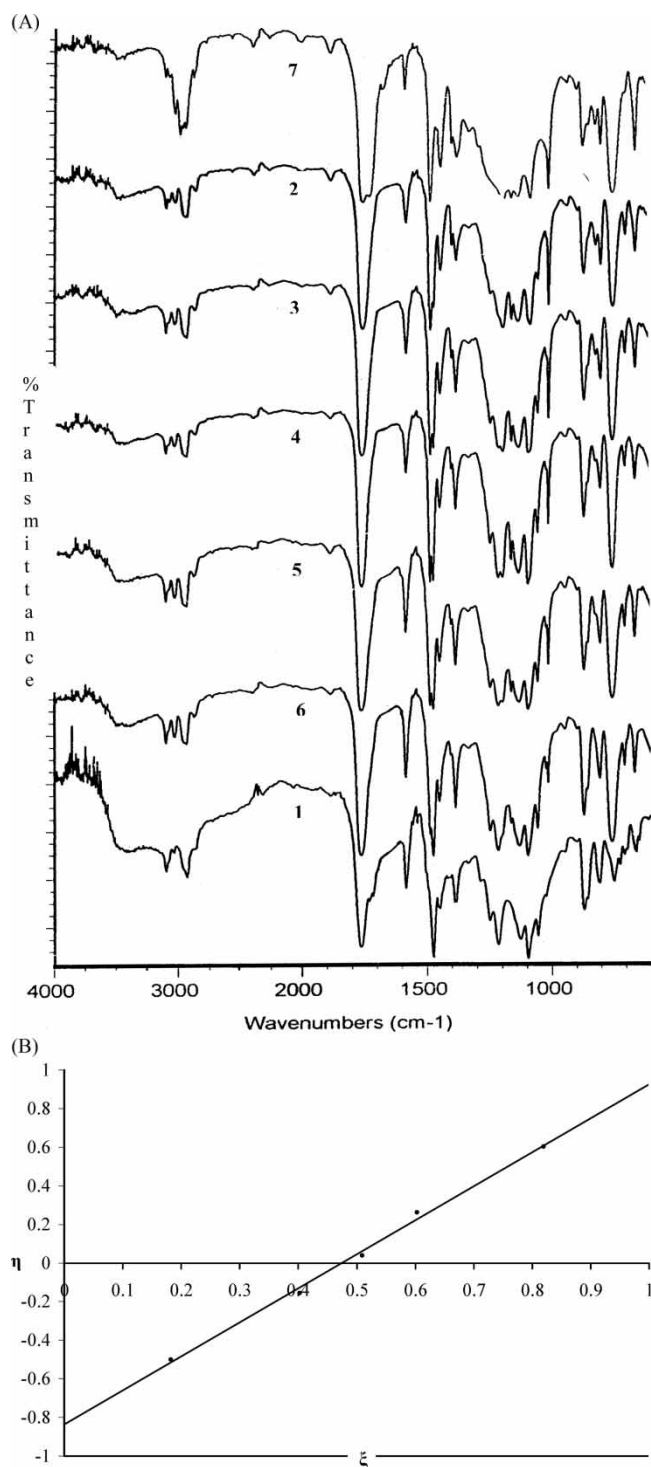


Fig. 4. (A) F-R plot for poly(2,4-DPA-co-PCPA), (B) K.T Plot for Poly(2,4-DPA-co-PCPA).

Table 2. Average molecular weights by GPC data for the copolymers of 2,4-DPA with PCPA

Sample code no.	Mn	Mw	Polydispersity (Mw/Mn)	Intrinsic viscosity $[\eta]$ (dL g ⁻¹)
1	32380	48640	1.502	0.210
2	9809	15970	1.628	0.090
3	10100	16550	1.638	0.105
4	10960	17250	1.573	0.112
5	15600	24050	1.541	0.128
6	23170	36140	1.559	0.147
7	18420	29410	1.597	0.131

Table 3. TGA data for homo- and copolymers of 2,4-DPA with PCPA

Sample code no.	% Weight loss at various temperature (°C)					Decomposition temperature range (°C)	T_{\max}^a (°C)	T_{50}^b (°C)	IPDT ^c (°C)	Activation energy ^d (E_A) (K·J·mole ⁻¹)
	200	300	400	500	600					
1	1	6	79	87	92	191–424	389	383	403	75
2	1	8	17	85	88	283–409	380	378	429	75
4	2	10	78	85	88	318–408	378	376	416	69
6	11	20	82	88	90	194–404	376	371	397	67
7	2	4	77	88	91	321–428	385	381	417	79

^aTemperature for maximum rate of decomposition.

^bTemperature for 50% weight loss.

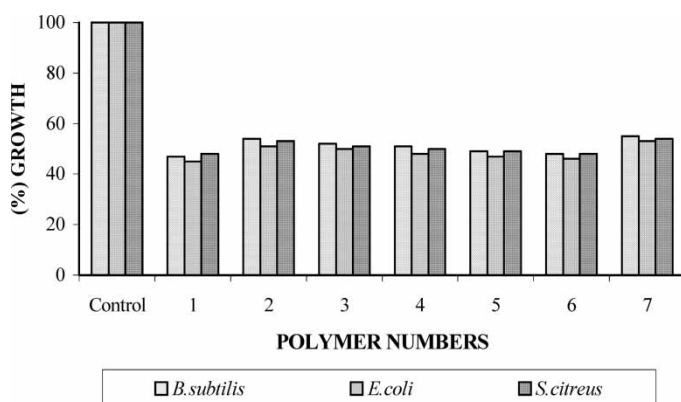
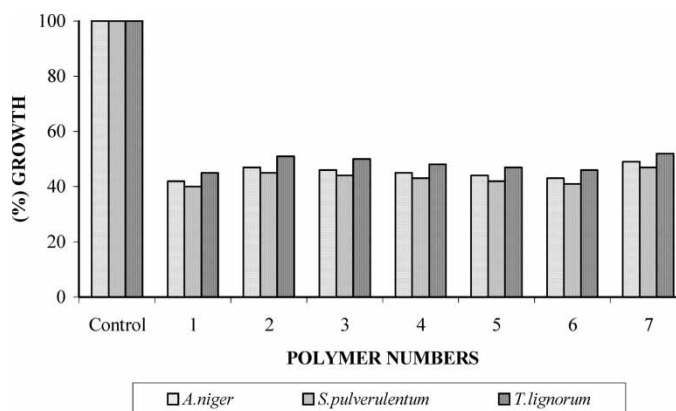
^cIntegral procedural decomposition temperature.

^dBy Broido's method.

composition. The activation energy (E_A) was calculated by Broido's method (22), which ranges from 67–79 K·J·mole⁻¹. The values of characteristic degradation temperature and integral procedural decomposition temperature (IPDT) were calculated by Doyle's method (23). IPDT express the overall thermal stability of the polymer and it varies between 397–429°C.

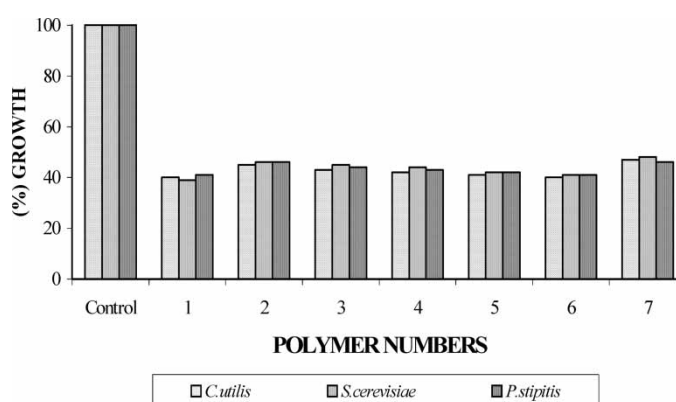
3.4 Antimicrobial Activity

The antimicrobial activity of homo and copolymers of 2,4-DPA and PCPA was investigated. The results obtained are presented in Figures 5, 6 and 7. It has been suggested that the presence of chlorine in a compound is of prime importance for its antimicrobial activity (24, 25). All the copolymers showed almost similar antimicrobial properties against bacteria, fungi and yeast. Poly(2,4-DPA) allowed about 45–48% growth of bacteria, 40–45% growth of fungi and 39–41% growth of yeast, while 53–55% growth of bacteria, 47–52% growth of fungi and 46–48% growth of yeast, respectively was possible in poly(PCPA). The poly(2,4-DPA) is found to be more effective in inhibiting the growth of microorganisms than that of poly(PCPA), this may be traced to the higher chlorine content of poly(2,4-DPA). As the percentage of 2,4-DPA in the copolymers

**Fig. 5.** Effect of 2,4-DPA-co-PCPA homo and copolymers on growth (%) of bacteria.**Fig. 6.** Effect of 2,4-DPA-co-PCPA homo and copolymers on growth (%) of fungi.

increases, the effectiveness of the copolymers to inhibit the growth of microorganisms increases as expected. The % growth in the copolymers can be seen from the chart given in Figures 5, 6 and 7. The inhibition property of the polymers is as follow:

$$1 > 6 > 5 > 4 > 3 > 2 > 7$$

**Fig. 7.** Effect of 2,4-DPA-co-PCPA homo and copolymers on growth (%) of yeast.

This clearly demonstrates that as the chlorine content in the polymer increases, the inhibiting property also increases.

4 Conclusions

The monomers 2,4-DPA and PCPA were synthesized, characterized and copolymerized using different feed ratio by free radical solution polymerization. Conventional methods were employed to characterize the polymers. The reactivity ratio of 2,4-DPA (r_1) is greater than that of PCPA (r_2) and the product of reactivity ratios was less than one. This shows that the monomers were distributed in the copolymer chain in a random fashion. The GPC results show that as 2,4-DPA content in copolymers increases the molecular weight increases. TGA data reveals that all the polymers undergo single step degradation. Chlorine content is important to impart antimicrobial property in these polymers. The fact that amongst the polymers investigated, the homopolymer of 2,4-DPA is most effective antimicrobial agent tends support to this view.

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