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Synthesis, Characterization and Antimicrobial Activity of Poly(2-acrylamido-2-methyl-1-propanesulfonic acid-co-crotonic acid)

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2-Acrylamido-2-methyl-1-propanesulfonic acid (AMPS), and crotonic acid (CrA) copolymerized with different feed ratios using *N*,*N*-dimethylformamide as a solvent and benzoyl peroxide (Bz_2O_2) as an initiator at 70°C. Structure and composition of copolymers for a wide range of monomer feed were determined by elemental analysis (content of N for AMPS-units). Monomer reactivity ratios for AMPS (M_1)-CrA (M_2) pair were determined by the application of conventional linearization methods such as Fineman-Ross (F-R), Kelen-Tüdös (KT) and Extended Kelen-Tüdös (EKT) and a nonlinear error invariable model method using a computer program RREVM. The characterizations were done by Fourier transform infrared spectroscopy (FTIR), ^{1}H-and ^{13}C-NMR spectroscopy, differential scanning calorimetry (DSC) thermal gravimetry analysis (TGA), scanning electron microscopy (SEM), and X-ray diffraction. The antimicrobial effects of polymers were also tested on various bacteria, and yeast.

Keywords: poly(2-acrylamido-2-methyl-1-propanesulfonic acid-co-crotonic acid); thermogravimetric analysis; SEM analysis; monomer reactivity ratios; X-ray diffractions

1 Introduction

It has been pointed out that polymers or copolymers containing carboxylic acid groups are highly desirable because such groups represent functionality that is useful for yielding a wide variety of products. Copolymers have the newly available carboxylic acid functional groups, which can be used in any further incorporation of drugs or other bioactive agents (1–5). Covalent linkages between polymer chains can be established by the reaction of functional groups with complementary reactivity, such as an amine, -carboxylic acid or an isocyanate -OH/NH_2 reaction (6). 2-acrylamido-2-methyl-1-propanesulfonic acid (AMPS) is a relatively strong acid (7) that has had a wide variety of applications (as the acid or its salts or as a comonomer) including photographic materials (8), contact lenses (9), hydrogels (10), and water absorbents (11), polyelectrolyte membranes and foam stabilizers (12). In the last years, sulfonated polymer membranes (13), including AMPS-containing polymers (14–16), became of interest in processes requiring preferential exchange of cations for electrodialysis and in fuel cell applications. Copolymers of AMPS with diethylene dimethacrylate (9), acrylamide (17), N-isopropylacrylamide (18), 2-hydroxyethyl methacrylate (11), and its graft copolymer with styrene (19) have been reported. Hydrogels based on AMPS, synthesized using various methods, have been used in biochemistry (20). The potential biological activity of compounds containing sulfur and nitrogen may be responsible for this increased interest. Crotonic acid is a bioactive factor in carrot seeds (21). Poly(crotonic acid) (PCrA), as a component in copolymers with 2-acrylamido-2-methyl-1-propanesulfonic acid (AMPS), has not been previously reported.

In the present work, the results of radical copolymerization of AMPS with CrA, determination of monomer reactivity ratios and the effects of copolymer composition on thermal behavior and crystallinity are presented and discussed. Homo- and copolymers have been tested in their antimicrobial activity against microorganism.

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2 Experimental

2.1 Materials

Crotonic acid (CrA) (Aldrich) was used after recrystallization from distilled water, m.p. 72–74°C. 2-acrylamido-2-methyl-1-propanesulfonic acid (AMPS) (Merck, 99%) was used without further purification. Benzoyl peroxide (Bz_2O_2) was recrystallized from chloroform-methanol mixture. *N,N*-dimethylformamide, chloroform, methanol, and ethanol (Merck), anhydrous magnesium sulphate (Aldrich) were analytical grade commercial products and used as received.

2.2 Copolymerization

Free-radical copolymerization of CrA with AMPS at constant total monomer concentration (1.5 mol L^{-1}) in *N,N*-dimethylformamide, was carried out in sealed glass bottles with polyethylene stoppers at 70°C, using 0.05 mol L^{-1} benzoyl peroxide as an initiator. The solution was purged with nitrogen for about 10 min, and the reaction mixtures were purged again for several minutes prior to heating. Polymerization was terminated by precipitating the reaction mixtures in a large volume of acetone at room temperature. The precipitated copolymers were purified by redissolving in a small amount of *N,N*-dimethylformamide, then reprecipitating. The purification procedure was carried out at least twice to ensure complete removal of unreacted monomers. Finally, the copolymers were dried in vacuum at 40°C to constant weight. Copolymer composition was determined by elemental analysis for nitrogen content.

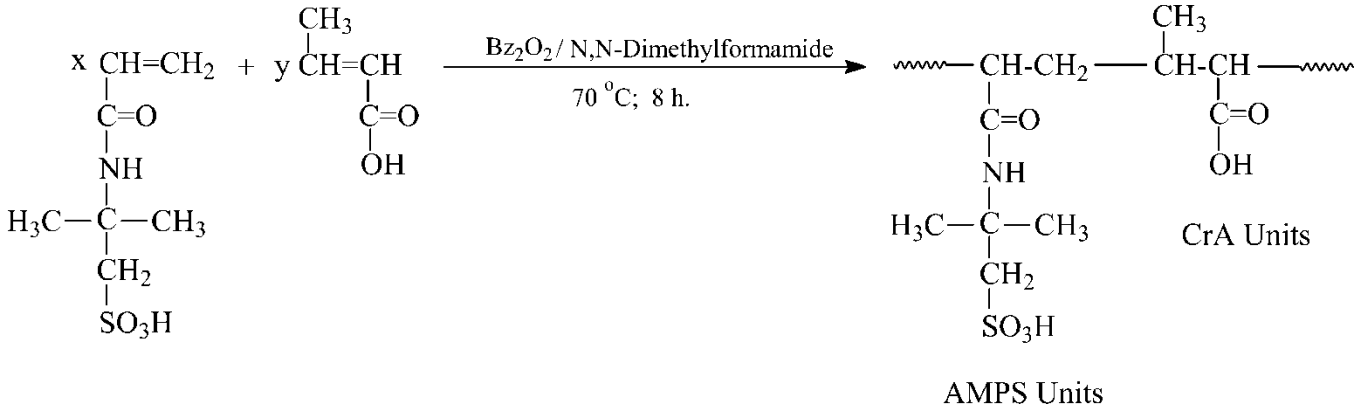
2.3 Characterization Techniques

Infra-red spectra were measured on a Jasco 460 Plus FT-IR spectrometer. ^{1}H- and ^{13}C-NMR spectra of the polymers were recorded in $CDCl_3$ with tetramethylsilane as the internal standard using a Gemini Varian 200 MHz NMR spectrometer. Thermal data were obtained by using a Setaram Labsys TG-DSC/DTA thermobalance in N_2 atmosphere. The glass transition (T_g) temperatures were determined by a Setaram 131 DSC. Samples of about 5–8 mg held in sealed aluminum crucibles and the heating rate of 20°C/min under a dynamic nitrogen flow (5 l · h^{-1}) were used for the measurements. Elemental analyses were carried out by a LECO CHNSO-932 auto microanalyzer. The microstructure of the polymers were examined by a scanning electron microscopy (SEM), Hitachi, Model:JSM-5600 imaging mode. The X-ray diffraction patterns were obtained using a Rıgaku Dmax2200 XRD apparatus.

2.4 In Vitro Antimicrobial Activity Studies

Seven bacteria and two yeasts were used at test organisms. Those are *Staphylococcus aureus* ATCC 6538 (Gr+), *Bacillus subtilis* ATCC 6633 (Gr+), *Bacillus megaterium* DSM 32 (Gr+), the Gram negative; *Echerichia coli* ATTC 25922, *Klebsiella pneumonia* FMC5, *Pseudomonas aeroginosa* DSM 50071, *Enterobacter aeroginosa* CCM 2531 and yeast, *Candida tropicalis* ATCC 13802, *Candida globrata* ATCC 66032. The minimum inhibitory concentrations (MIC) of the compounds against the test organism were determined by using the broth dilution method. Minimal inhibitory concentration (MIC) is the lowest concentration of a compound that completely inhibits microbial growth (22, 23). Bacterial strains were obtained in a Mueller Hinton Broth for incubation at 37 ± 0.1°C for 24 h. The yeasts were cultured in Sabourad dextrose broth after incubation for 24 h at 25 ± 0.1°C.

The test carried out in Mueller Hinton Broth (for bacteria) and Sabourad dextrose broth (for yeasts). Inoculum was 10^5 CFU ml^{-1} for bacteria and yeasts. The compounds and standard antibiotics (ampicillin and fluconazole) were dissolved in dimethylsulfoxide (DMSO) at an initial concentration of 1024 μg ml^{-1} and then were serially diluted (512, 256, 128, 64, 32, 16, 8, 4, μg ml^{-1}) in culture medium to 2 μg ml^{-1}.



Sch. 1. Monomeric units of the copolymer.

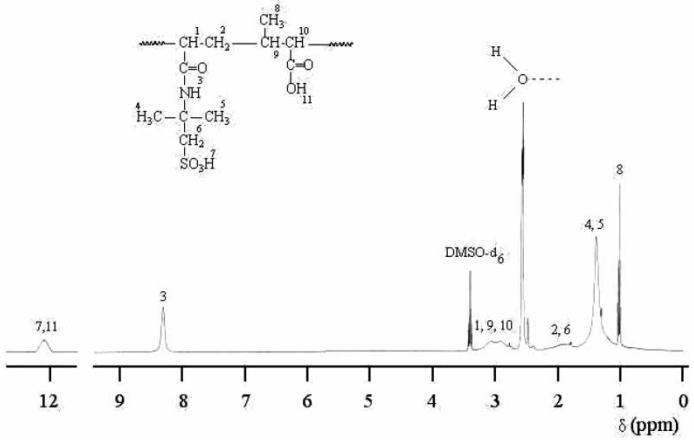


Fig. 1. ^{1}H-NMR spectra of copoly(AMPS-CrA); m_1:m_2: [0.58 : 0.42].

3 Results and Discussion

3.1 Copolymer Characterization

The constituent monomeric units of the copolymer are as on Scheme 1.

The ^{1}H-NMR and ^{13}C-NMR spectrum of poly(AMPS-co-CrA) [0.58 : 0.42) and its attributions are shown in Figures 1 and 2. The NMR spectras of the polymer have the characteristic peaks of the polymeric units. Some characteristic absorption bands (FTIR) and chemical shifts (^{1}H and ^{13}C-NMR), and their assignments of copolymer synthesized are presented in Table 1.

3.2 Copolymer Composition and Monomer Reactivity Ratios

The monomer reactivity ratios for the copolymerization of AMPS with CrA were determined from the monomer feed ratios and the copolymer composition. The classical approach for acquiring copolymer data was to isolate the

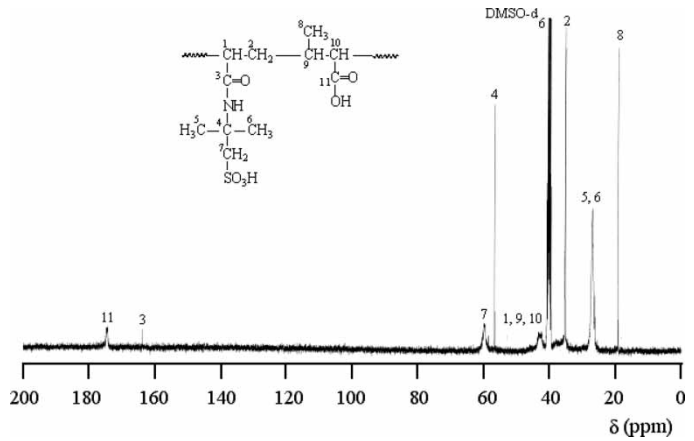


Fig. 2. ^{13}C-NMR spectra of copoly(AMPS-CrA); m_1:m_2: [0.58 : 0.42].

copolymers from each of 9 feed compositions at early conversions and analyze the copolymer compositions by elemental analyses. The results are presented in Table 2. It indicates that the composition of AMPS is the copolymer in always higher than that in the feed.

The Fineman-Ross (FR) (24), Kelen-Tüdös (KT) (25) and the extended Kelen-Tüdös (EKT) (26, 27) methods were used to determine the monomer reactivity ratios. The Kelen-Tüdös and extended Kelen-Tüdös parameters for copoly(AMPS-CrA) are provided in Tables 3 and 4.

The monomer reactivity ratios determined by conventional linear methods are only approximate and are usually employed as good starting values for non-linear parameter estimation schemes.

To determine more reliable values of monomer reactivity ratios, a non-linear error-in-variables model (EVM) method is used utilizing the computer program, RREVM (28). Various statistical treatments of the feed and copolymer compositions can be used to determine monomer reactivity ratios. The non-linear methodology used selected values of r_1 and r_2, where the sum of the squares of the differences between the observed and the computed polymer compositions was minimized. With this criterion for the nonlinear least-squares method of analysis, the values for the monomer reactivity ratios were unique for a given set of data. The program produces monomer reactivity ratios for the monomers in the system with a 95% joint confidence limit determination. The joint confidence limit is a quantitative estimation of the validity of the results of the experiments and the calculations performed. This method of data analysis consists of obtaining initial estimates of the monomer reactivity ratios for the system and experimental data of comonomer charge amounts and comonomer amounts that have been incorporated into the copolymer, both in molar fractions. Tidwell and Mortimer (29) produced a nonlinear least-squares method that allowed rigorous applications of statistical analysis for reactivity ratios r_1 and r_2. This method is a modification or extension of the curve-fitting model and allows the calculations to be quantitatively analyzed. Extensive calculations are needed, but a computer program by A. Penlidis (28) permits rapid data analysis of the nonlinear calculations. The 95% joint confidence region for the determined r_1 and r_2 values using RREVM is shown in Figure 3. The r_1 and r_2 values from methods such as F-R, K-T, EKT and RREVM are presented in Table 5.

The value of r_1 is near to 1 and that of r_2 is <1, which indicates the presence of a higher amount of AMPS units in the copolymer than that in the feed. However, the value of the product $r_1 r_2 < 1$, which indicates that the system deviates from one of the ideal copolymerizations.

3.3 Thermal Properties

From DSC measurements T_g was taken as the midpoint of the transition region. The gradual increase in the T_g of the copolymer was observed with an increase in the mol percent of AMPS in the copolymer (Table 6) indicating that the presence of

Table 1. Characteristic FTIR absorption bands, ^{1}H and ^{13}C-NMR chemical shifts and their assignments for poly(AMPS-co-CrA)

	FTIR (υ in cm^{-1})	^{1}H-NMR (δ in ppm)	^{13}C-NMR (δ in ppm)
AMPS-Unit	• 3350–3180 (w, broad) NH amide	• 1.34 (CH_3 in side chain -$C(CH_3)_2$ group)	• 56.70 in -C-$(CH_3)_2$
	• 1640 (s, broad) C═O amide I	• 1.7–2.3 (CH_2 backbone)	• 26.25 (2 CH_3 in side chain)
	• 1560 (w) secondary amide II	• 1.7–2.3 (CH_2-SO_3H)	• 59.68 (CH_2-SO_3H)
	• 1420 (w) and 1285 (m) antisym. and sym. S═O	• 8.11–8.32 (broad, NH amide)	• 164.57 (C═O for amide)
	• 1240 (w-m) amide III	• 12.20 (SO_3H)	
	• 1085 (s, broad) and 1040 (m) SO_3H acid group		
	• 1380 (w) and 1345 (m) two bands CH_3 group		
	• 455–420 (w) C-N-C in amide group		
CrA-Unit	• 3330–3500 (w, broad) COOH	• 1.02 (CH_3 in side chain)	• 19.22 (CH_3 in side chain)
	• 1730 (s) sym C═O		
	• 620 (w-m) CH bending	• 12.20 (OH for COOH)	• 174.38 (C═O for COOH group).
Common bands	• 2925–2850 (m-s), 1430 (s) and 1500 (s)		
For two units	Backbone and side-chain C—H stretching and bending bands in CH, CH_2 and CH_3 groups		

sterically bulky propane sulfonic group in the copolymer increases the T_g of the copolymer. Data analysis was carried out with the Setaram software package. As seen from DSC curves, the intensity and position of the broad endo-peaks around 160–185°C, which are associated with the melting point, significantly depend on the monomer unit ratios in the copolymers, especially on the CrA content. It is known that the higher melting points of polymers are associated with many factors including inter- and intramolecular interactions through hydrogen-bonded functional linkages and structural regularity and rigidity of macromolecules (16). These results indicate that T_m significantly depends on the strong acidic CrA and AMPS unit content in polymers. These allows to propose that the observed characteristic melt phase is the result of formation and melting of the strong hydrogen-bonded physically network structure, which does not show reversible crystallization behavior after cooling. In the observed abnormal strong melting transition with higher values of enthalpy ($\Delta H_o^d = 40.38–90.11\ J \cdot g^{-1}$) for the copolymers. The enthalpy changes (ΔH_o^d) and heat capacity (ΔCp) during thermal degradation obtained from the DSC thermograms of polymers are given in Table 6. The gradual increase of (ΔH_o^d) and (ΔCp) of the polymers for melting point were observed with an increase in the mol percent of CrA unit in the copolymer. (ΔH_o^d) and (ΔCp) were significantly increased with CrA content depending on the endothermic

Table 2. Monomer compositions in feed and copolymer

Sample code no	Feed composition in mole fraction		Conversion (%)	Elementel (%) N	Copolymer composition in mole fraction	
	AMPS(M_1)	CrA(M_2)			AMPS(m_1)	CrA(m_2)
P-1	1.00	—	—	—	1.00	—
P-2	0.80	0.20	8.6	6.30	0.85	0.15
P-3	0.75	0.25	7.9	6.06	0.78	0.22
P-4	0.70	0.30	8.8	5.80	0.71	0.29
P-5	0.60	0.40	10.4	5.44	0.63	0.37
P-6	0.50	0.50	8.4	5.20	0.58	0.42
P-7	0.40	0.60	9.2	4.98	0.54	0.46
P-8	0.30	0.70	9.0	4.40	0.44	0.56
P-9	0.25	0.75	7.8	4.18	0.40	0.60
P-10	0.20	0.80	8.0	3.60	0.32	0.68
P-11	—	1.00	—	—	—	1.00

Table 3. F-R and K-T parameters for copoly(AMPS/CrA) system

Sample code no.	$F = M_1/M_2$	$f = m_1/m_2$	$G = F(f-1)/f$	$H = F^2/f$	$\eta = G/(\alpha + H)$	$\xi = H/(\alpha + H)$
P-2	4.0000	5.6667	3.2942	2.8235	0.9587	0.8218
P-3	3.0000	3.5455	2.1538	2.5385	0.6836	0.8056
P-4	2.3333	2.4483	1.3803	2.2238	0.4867	0.7841
P-5	1.5000	1.7027	0.6190	1.3214	0.3201	0.6833
P-6	1.0000	1.3810	0.2759	0.7241	0.2064	0.5418
P-7	0.6667	1.1739	0.0988	0.3786	0.0997	0.3820
P-8	0.4286	0.7857	−0.1169	0.2338	−0.1381	0.2763
P-9	0.3333	0.6667	−0.1667	0.1667	−0.2139	0.2139
P-10	0.2500	0.4706	−0.2813	0.1328	−0.3774	0.1782

Table 4. Extended K-T parameters for copoly(AMPS/CrA) system

Sample code no.	ζ_1	ζ_2	Z	G	H	η	ξ
P-2	0.0884	0.0624	1.4365	3.2487	3.7462	0.9747	0.8239
P-3	0.0805	0.0681	1.1896	2.1397	2.5052	0.6920	0.8102
P-4	0.0886	0.0845	1.0516	1.3772	2.2138	0.4917	0.7904
P-5	0.1068	0.0940	1.1430	0.6148	1.3032	0.3253	0.6895
P-6	0.0914	0.0662	1.3997	0.2722	0.7049	0.2107	0.5457
P-7	0.1103	0.0626	1.8067	0.0963	0.3596	0.1017	0.3799
P-8	0.1159	0.0632	0.8863	−0.1136	0.2208	−0.1406	0.2734
P-9	0.1079	0.0540	2.0587	−0.1619	0.1573	−0.2176	0.2114
P-10	0.1133	0.0601	1.9369	−0.2733	0.1254	−0.3837	0.1761

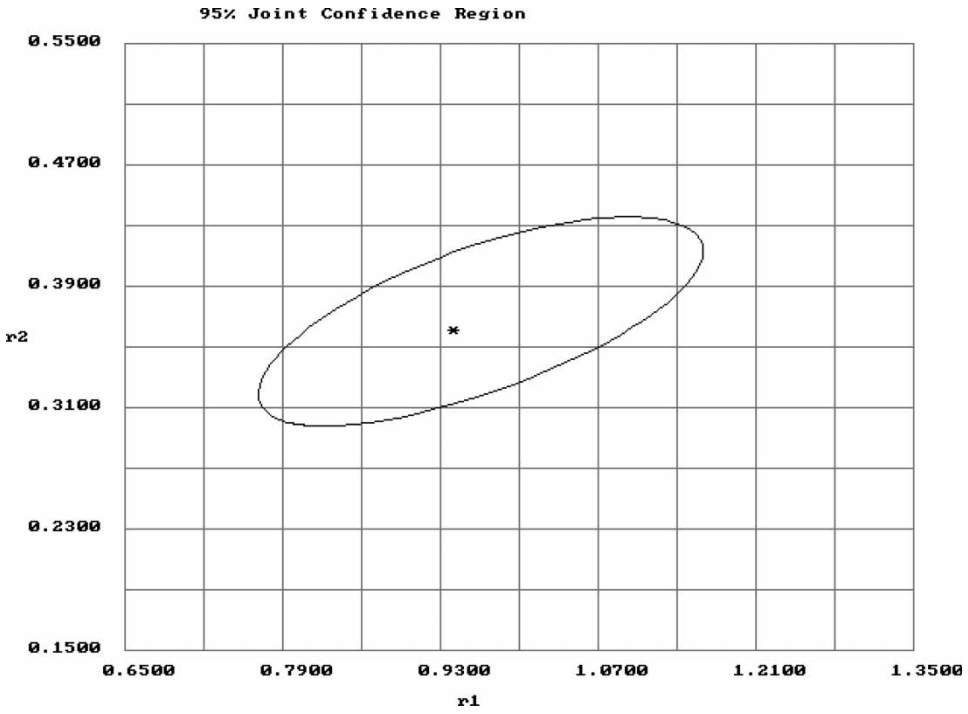


Fig. 3. 95% joint confidence region of r_1 and r_2 values by RREVM for AMPS-CrA copolymer system.

Table 5. Copolymerization parameters for the free radical copolymerization of AMPS with CrA

Method	r_1[a]	r_2[a]	r_1r_2
Fineman-Ross	1.0814	0.4604	0.4978
Kelen-Tüdös	0.9824	0.3662	0.3597
Extended Kelen-Tüdös	0.9832	0.3504	0.3445
RREVM	0.9427	0.3606	0.3399

[a] r_1 and r_2 are the monomer reactivity ratios of AMPS and CrA, respectively.

peak area and mass of sample. Also the gradual increase of T_g of the polymers were observed with an increase in the mol percent of AMPS unit in the copolymer.

As evidenced from Figure 4, TGA curves have characteristic three-step decomposition regions. The first weight loss region appears around 120–190°C associated with dehydration of partially degradation of amide groups; secondary weight loss occurring around 210–300°C can be related to possible decarboxylation and/or other reactions of side-chain units and degradation of sulfonic groups; at last weight loss around 315–450°C indicate the main-chain degradation reactions and breakdown of the polymer backbone (20). Copolymers show a very high thermal stability which increases with increasing acidic AMPS unit content in copolymers. The increasing thermal stability at higher temperatures may be due to the presence of $-SO_3H$ and -COOH groups in the side chain, which form cross-links. It is well known that H-bonds, as a variety of intermolecular interaction, exert essential influence on kinetic and elementary actions of radical (co)polymerization (16, 30). An H-bond involves two functional groups in the same or in different molecule(s), i.e., between proton-donor (-COOH, -OH, $-CO-NH_2$, -CO-NHR, etc.) and proton -acceptor (-C═O, -OR, $-NH_2$, -NHR, etc) groups. Due to the formation of H-bonds, physical, physico-chemical properties, spectral (UV, FTIR, NMR, etc.) and structural parameters of (co)polymers can be changed essentially (31, 32).

Table 6. Thermal behavior of copolymers

AMPS(m_1)		DSC					TGA % weight loss at		
	T_g (°C)	ΔH_o^d (J/g)	ΔCp (J/g · K)	T_m (°C)	ΔH_o^d (J/g)	ΔCp (J/g · K)	200°C	300°C	400°C
0.85	123	0.9428 (Endo)	−0.1968	192	40.3784 (Endo)	−0.2281	18	47	65
0.71	120	0.8082 (Endo)	−0.1622	188	45.2376 (Endo)	−0.3885	12	50	67
0.63	116	0.5808 (Endo)	−0.1022	186	48.7947 (Endo)	−0.7336	20	48	70
0.54	113	0.5520 (Endo)	−0.0816	182	70.6866 (Endo)	−0.7535	21	51	74
0.40	110	0.4933 (Endo)	−0.0805	179	90.1172 (Endo)	−0.7968	20	52	77

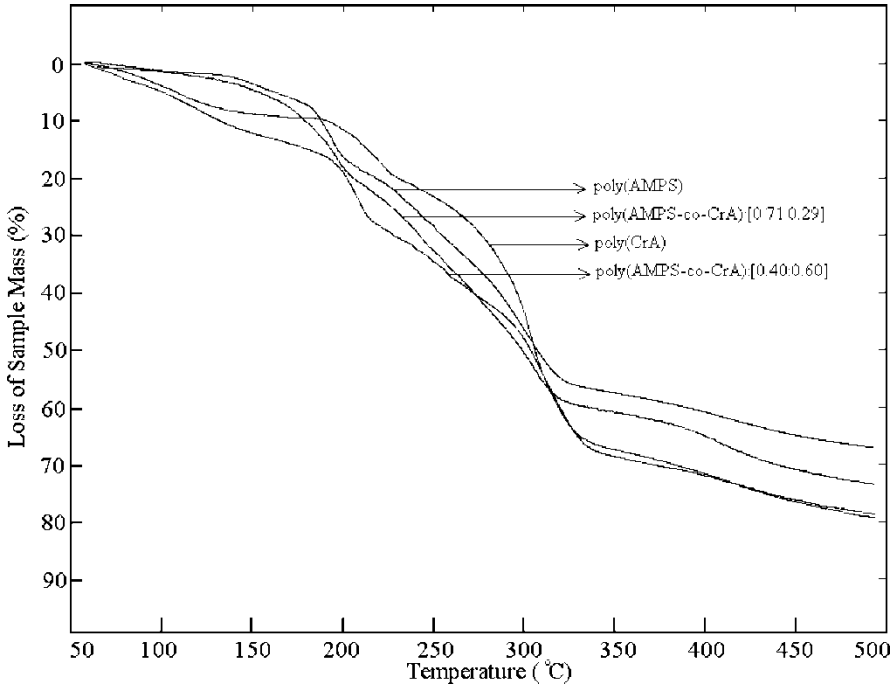
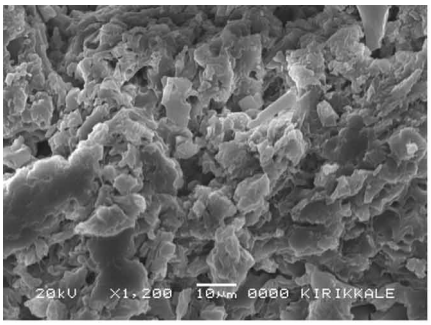
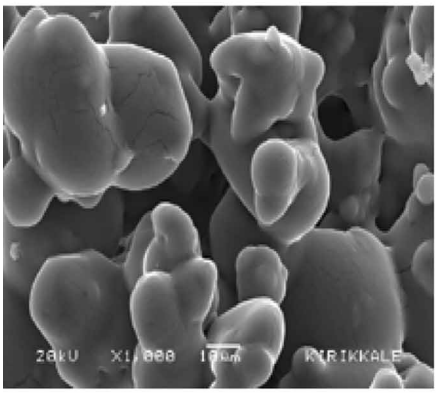


Fig. 4. TGA curves of investigated polymers.



(a)



(b)

Fig. 5. Scanning electron microscope (SEM) image of the investigated polymers (a) poly(AMPS), (b) poly(AMPS-co-CrA)[0.71:0.29].

3.4 Morphologies of Polymers

Scanning electron microscopy of copolymers was performed using a Jeol model JSM−5600 scanning electron microscope at 20 kV with varying levels of magnification. In poly(AMPS) (Figure 5(a)) micrographs have the morphology with standard cauliflower structure. SEM micrograph of poly(AMPS-co-CrA)s (Figure 5(b)) reveal big droplets, whereas it is a cauliflower-like structure in pristine polymers. Microscopic observation suggested that the poly(AMPS-co-CrA)s were immersed in a partially haematic exudate. SEM revealed an absence of the organized fibrin clot, matrix which is usually evident as a AMPS and CrA unit after implant. Platelet adhesion and aggregation involved the whole gel surface.

3.5 Antimicrobial Activities

A set of assay tubes containing only inoculated broth was kept as control. The tubes were incubated 37 $\pm$ 1°C for 24 h for bacteria and 25 $\pm$ 0.1°C for 24 h for the yeasts. The MICs were recorded by visual observation after incubation periods. The data reported in Table 7 are the average data of three experiments. All studied compounds were activity against the microorganism. In general, the compounds inhibitory properties against microorganism were lower that of the standards drug. The obtained data reported that compounds were able to inhibit the growth of the microorganisms *in vitro* showing MIC values between 512–4 μg ml^{-1}. It can be inferred from the data that, in the present copolymers as the percentage of AMPS increases, the antimicrobial activity increases. The poly(AMPS-co-CrA) is includede CrA, which is known for strong herbicidal properties; in polymers, CrA replaceable anion (COO^-) showed lower effective and selective antimicrobial activities. It is also possible that the conformation of the polymers acquired under experimental conditions may also be a factor for their anti-growth activity. This study, however, is beyond the scope of this investigation (33, 34). MIC were determined by a standarts dilution technique after antimicrobial activities of compound were determined by using a filter paper disk

Table 7. *In vitro* antimicrobial activity results of compounds and standarts (MIC μg ml^{-1})

Sample code no.	S.a	B.s	B.m	K.p	E.a	E.c	P.a	C.t	C.g
P-1	512	512	512	512	512	256	256	512	512
P-2	512	512	512	512	512	256	256	512	512
P-3	512	512	512	512	512	256	256	512	512
P-4	512	512	512	512	256	256	256	256	512
P-5	256	256	256	512	256	128	128	128	256
P-6	256	256	256	256	512	512	256	256	512
P-7	256	256	256	128	128	128	64	128	256
P-8	128	256	256	256	256	128	128	128	256
P-10	64	64	128	64	128	64	128	128	64
P-11	64	64	128	64	32	32	128	128	64
Ampicilin	2	2	2	2	2	2	2	–	–
Fluconazol	–	–	–	–	–	–	–	8	8

S.a: Staphylococcus aureus ATCC 6538, B.s: Bacillus subtilis ATCC 6633, B.m: Bacillus megaterium DSM 32, K.p: Klebsiella pneumonia FMC5, E.a: Enterobacter aeroginosa CCM 2531, E.c: Echerichia coli ATTC 25922, P.a: Pseudomonas aeroginosa DSM 50071, and yeast; C.t: Candida tropicalis ATCC 13802, C.g: Candida globrata ATCC 66032.

Table 8. X-ray diffraction of the prepared copolymers

Poly(AMPS-co-CrA)[0.32:0.68]			Poly(AMPS-co-CrA)[0.71:0.29]		
2θ	d (Å)	I/I_0	2θ	d (Å)	I/I_0
21,12	4,2032	100	11,76	7,51913	46,78571
21,4	4,14883	96,60455	25,58	3,47956	68,65476
21,12	4,2032	100	25,94	3,43208	72,21429
20,84	4,25904	93,78882	27,06	3,29252	60,30952
20,48	4,33308	95,85921	30,56	2,92294	51,90476
20,1	4,41414	95,85921	52,4	1,74471	26,97619
19,8	4,48034	92,33954	61,5	1,50657	22,14286
19,66	4,51192	93,78882	64,2	1,44957	21,78571
18,92	4,68669	85,50725	20,12	4,40979	100
18,66	4,7514	84,05797	23,48	3,7858	91,66667
18,26	4,85458	81,98758	23,42	3,79536	86,5119
17,14	5,16919	82,81573	21,74	4,08471	82,14286
16,96	5,22364	78,67495	21,24	4,17972	85,71429
16,8	5,27303	77,84679	21,1	4,20714	86,10714
16,4	5,40073	71,01449	20,9	4,24695	84,52381
15,96	5,54862	60,66253	20,44	4,34147	82,14286
22,74	3,9073	96,48033	20,12	4,40979	100
23,58	3,76997	94,40994	19,82	4,47586	86,42857
23,92	3,71715	91,09731	19,58	4,53018	84,91667
26,2	3,39861	83,43685	19,34	4,58585	82,85714
27,9	3,19527	73,08489	17,94	4,94044	95,2381
31,8	2,81173	62,1118	17,84	4,96791	95,59524
33,8	2,64979	55,07246	17,72	5,00128	95,59524
35,42	2,53222	50,31056			
39,18	2,29743	51,75983			
44,18	2,04833	44,7205			
49,4	1,84341	35,81781			
53,16	1,72154	32,29814			
63,4	1,46592	29,60663			

agar-diffusion method. The synthesized compounds were dissolved in DMSO and absorbed onto a disk. DMSO disappeared with evparation. All compounds were in a inhibition zone.

3.6 X-ray Diffraction Analysis

The XRD data of the copolymers are given in Table 8. It can be seen that the patterns of the copolymers are similar. Both of them have a broad amorphous peak. The contact angle of the peaks is between 5° and 35°. It means that CrA incorporated through the covalent and hydrogen bond, and not simple mixing. The X-ray diffractions of the copolymers of CrA with the AMPS comonomer clearly reveal an increase in the degree of crystallinity, and an amorphous halo pattern takes place. Moreover, the degree of the crystallinity will be inversely proportional to the molar ratio of the copolymer feed with CrA. On the other hand, the X-ray diffraction curves throw light on the structure and the morphology of the obtained copolymer. The data indicates that the crystallinity of the obtained copolymers increases with increasing of molar ratio of CrA. As shown by the data, the copolymer show a moderate degree of crystallinity.

4 Conclusions

The antimicrobial activity on the homo- and copolymers of AMPS with CrA was obtained. As the percentage of AMPS in the copolymers increases, the effectiveness of the copolymers to inhibit the growth of the microorganisms increases. Although the sulfur and nitrogen content of the polymers appears to be the most important component to impart antimicrobial properties, it should be remembered that the conformation the polymers acquired under experimental conditions is a factor for their antigrowth activity. The presence of CrA in the copolymer increases the crystallinity of the copolymers obtained.

5 References

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