CHAPTER II

EXPERIMENTALS

2.1 Chemicals and Instruments

Acryloyl chloride and triethylamine were obtained from Fluka and freshly distilled prior to use.

Pentachlorophenol, 2,4,5-trichlorophenol, 2,4,6-trichlorophenol, 4-chloro-3-methylphenol and dodecane were obtained from Fluka and used without further purification.

Methyl methacrylate was also obtained from Fluka and purified by shaking with 5 wt % aqueous sodium hydroxide solution. It was then washed several times with distilled water, dried over anhydrous sodium sulfate and distilled prior to use.

Toluene, methanol, n-hexane were commercial grade, but benzene and chloroform were reagent grade and obtained from Merck. All of them were distilled before used.

Melting points were determined by John-Fisher electrothermal melting point apparatus with a cover glass sample holder and are uncorrected.

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The GLC analysis was carried out by using Gas Chromatograph, Shimadzu model GC-RIA.

The following instrument was used to obtain the spectroscopic data.

UV-Visible Spectrophotometer, Shimudzu, model UV 240 with 10 nm matched quartz cells,

Infrared Spectrophotometer, Perkin-Elmer, model Perkin-Elmer 780, with grating infrared spectrophotometer,

Fourier-transform NMR spectrometer, Jeol, model JNX-FX 90 Q.

2.2 Syntheses of the Chlorophenyl Acrylate Monomers

2.2.1 General Procedure

The chlorophenolic compound (0.15 mole), tolulene (400 mL) and triethylamine (0.12 mole) were charged into a three-necked round bottom flask (500 mL) equipped with a condenser, a nitrogen inlet and a mechanical stirrer. The solution was cooled at 25°C then acryloyl chloride (0.18 mole) was slowly added dropwise to the cooling solution while it was stirring. During the reaction, white solid of triethylaminehydrochloride immediately precipitated. After about 3 hours, the mixture was filterred to remove triethylaminehydrochloride and several washing of the filtrate with distilled water removed all unreacted acryloyl chloride. The toluene layer was dried over anhydrous sodium sulphate and toluene was removed by reduced pressure distillation to yield a solid or a liquid. The synthesized compound was purified by either recrystallization from methanol as a solvent or column chromatography using silica gel as an adsorbent and benzene as an eluent. The yields were varied from 80% to 90%. Structures of the synthesized products are elucidated by the infrared and the proton NMR spectroscopy.

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2.2.1.1 Pentachlorophenyl Acrylate (PCPA)

Pentachlorophenyl acrylate was prepared by the reaction of pentachlorophenol and acryloyl chloride in the

presence of triethylamine (20). A round-bottomed flask (500 mL, equipped with a condenser and nitrogen blanket) was previously charged with toluene (400 mL), pentachlorophenol (40.0 g, 0.15 mole), and triethylamine (24.9 mL, 0.18 mole). The flask was then maintained at 20-25°C while acryloyl chloride (14.6 mL, 0.18 mole) was added sequentially by equalizing funnel. White triethylamine hydrochloride (m.p. 253°C) immediately precipitated. After 3 hours, the ice bath was removed and the flask was allowed to warm to room temperature. After warming, the toluene solution was filtered and washed several times with distilled water (400 mL) to remove unreacted acryloyl chloride. The benzene layer was dried by anhydrous sodium sulphate, then removed by vacuum rotaevaporation. The remaining brownish solid was recrystallized from methanol to yield 40 g (83%) pentachlorophenyl acrylate as a white solid, m.p. 75° C.

Spectroscopic data :

IR (KBr) ν_{max} (cm^{-1}): 3049 (w), 1750 (s), 1635 (m), 1405 (m), 1390 (s), 1365 (s), 1140 (s), 1120 (s), 982 (m), 800 (m), 790 (m) and 720 (m)

^{1}H-NMR ($CDCl_3$) δ (ppm): 6.08-6.86 (m, 3H)

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2.2.1.2 2,4,5-Trichlorophenyl acrylate (2,4,5-TCPA)

Similarly, 2,4,5-trichlorophenyl acrylate was prepared by the reaction of 2,4,5-trichlorophenol (30 g ,0.15 mole) and acryloyl chloride (14.6 mL , 0.18 mole) in toluene (400 mL) and in the presence of triethylamine (24.9 mL , 0.18 mole). Other procedure was carried out in the same way as that of Section

2.2.1.1. A colourless needle solid of 2,4,5-trichlorophenyl acrylate was ultimately obtained to yield 33 g (86%), m.p. 63°C.

Spectroscopic data:

IR (KBr) ν_{max} (cm^{-1}): 3095 (s), 1755 (s), 1635 (m), 1460 (m), 1410 (m), 1350 (m), 1300 (m), 1245 (m), 1125 (m), 1140 (m), 995 (m), 895 (m), 870 (m), 855 (w), 795 (m), 765 (w) and 680 (w)

^{1}H-NMR ($CDCl_3$) δ (ppm): 6.00-6.80 (m, 3H), 7.34 (s,1H), 7.55 (s,1H)

2.2.1.3 2,4,6 Trichlorophenyl acrylate (2,4,6-TCPA)

Like Section 2.2.1.1, 2,4,6-trichlorophenyl acrylate was prepared from the reaction of 2,4,6-trichlorophenol (30 g, 0.15 mole) and acryloyl chloride (14.6 mL , 0.18 mole) in toluene (400 mL) and the presence of triethylamine (24.9 mL, 0.18 mole). Eventually, the colourless liquid was obtained and purified by column chromatography using silica gel as an adsorbent and benzene as an eluent to yield 35 g (91%) 2,4,6-trichlorophenyl acrylate.

Spectroscopic data:

IR (NaCl) ν_{max} (cm^{-1}): 3090 (w), 1760 (s), 1638 (m), 1570 (s), 1450 (s), 1410 (s), 1390 (m), 1225 (s), 1200 (w), 985 (m), 860 (m), 825 (m), 810 (w) and 775 (m)

^{1}H-NMR ($CDCl_3$) δ (ppm): 6.04-6.84 (m, 3H), 7.36 (s,2H)

2.2.1.4 4-Chloro-3-methylphenyl acrylate (4-Cl-3-MPA)

In the same manner, the reaction of 4-chloro-3-methylphenol (21.7 g,0.15 mole) and acryloyl chloride (14.6 mL, 0.18 mole) in the presence of triethylamine (24.9 mL,0.18 mole) was carried out in toluene (400 mL). After purification from column

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chromatography, a pale yellow liquid of 4-chloro-3-methylphenyl acrylate (26 g, 87%) was finally obtained.

Spectroscopic data :

IR (NaCl) ν_{max} (cm^{-1}): 3040 (m),2930 (w), 1760 (s), 1640 (m), 1480 (s), 1405 (s), 1298 (m),1250 (m), 1230 (s), 1160 (s),1050 (m), 1030 (m), 990 (m), 900 (m), 805 (m), 735 (w), 685 (m)

^{1}H-NMR ($CDCl_3$) δ (ppm) : 2.34 (s, 3H), 5.87-6.53 (m,3H), 6.69-7.38(m, 3H)

2.3 Homopolymerization of the Chlorophenyl Acrylate Monomers

The chlorophenyl acrylate monomer was dissolved in benzene (10 mL) in a reaction vessel. Then α,α′- azobisisobutyronitrile (1 mole %) was added. After the solution was purged with nitrogen for 10 minutes, it was heated in an oil bath whose temperature was maintained at 50°C. When the reaction was carried out for a certain length of time, it was cooled to room temperature. The cool solution was then added dropwise into 400 mL of hexane with vigorously stirring. The precipitate was filtered, redissolved in benzene and reprecipitated in methanol until no contamination of the monomer was detected as revealed by thin layer chromatography and infrared spectroscopy. The white powder was dried in vacuo at 50° C to constant weight. The homopolymerization data of the chlorophenyl acrylate are listed in Table 2.1 .

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Table 2.1 Homopolymerization data of the chlorophenyl acrylates

Monomer	Weight (g)	Time (hr)	Polymer (g)	Conversion (%)
PCPA*	3.2090	5.30	1.5194	47.30
2,4,5-TCPA	2.5187	5.20	0.4836	19.28
2,4,6-TCPA	2.5937	14.30	1.8604	71.72
4-Cl-3-MPA	1.9688	11.30	1.2948	65.76

* Benzoyl peroxide was used as the intiator.

2.4 Copolymerization of the Chlorophenyl Acrylate Monomer and Methyl Methacrylate

2.4.1 Low-Conversion Copolymerization

Copolymerization was carried out by thoroughly mixing certain amount of methyl methacrylate with the chlorophenyl acrylate in 20 mL of dry benzene in the reaction cell. For each chlorophenyl acrylate, seven reaction cells were prepared and contained different mole fractions of the chlorophenyl acrylate comonomer in feeding, i.e. 0.7,0.6,0.5,0.4,0.3,0.2, and 0.1. It was then added 0.0164 g of α,α'-azobisisobutyronitrile into each reaction cell. After heating at 50°C for a certain length of time, the solution was cooled and added dropwise into 600 mL of hexane with vigorously stirring. The white powder of the copolymer was filtered off, redissolved in benzene, reprecipitated into methanol and filtered then dried under vacuum at 50°C to constant weight.

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Finally, the dried precipitate was chromatagraphed on tlc plate comparing with the corresponding chlorophenyl acrylate and using methanol as the developing solvent. It revealed that neither monomer contaminated with the precipitate. The polymerization for each monomer feeding ratio of the chlorophenyl acrylate and methyl methacrylate was repeated twice. The polymerization data of each chlorophenyl acrylate and methyl methacrylate were listed in Table 2.2-2.5 .

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Table 2.2 Data for copolymerization of pentachlorophenyl acrylate and methyl methacrylate

Poly (PCPA-co-MMA)	PCPA (g)	MMA (g)	Benzene (mL)	Time (hr)	Copolymer (g)	Conversion (%)
first run						
PC-1-I	0.8100	2.2561	25	10.42	0.2620	8.54
PC-1-II	1.6079	2.0106	25	12.00	0.3877	10.71
PC-1-III	2.4061	1.7653	25	10.10	0.6216	14.91
PC-1-IV	3.2183	1.5069	25	10.00	0.6847	14.49
PC-1-V	4.0137	1.2621	25	9.20	0.7917	15.00
PC-1-VI	4.8223	1.0109	25	9.20	0.6263	10.75
PC-1-VII	5.6143	0.7631	25	9.00	0.1714	2.68
second run						
PC-2-I	0.7990	2.2493	25	11.00	0.4500	14.80
PC-2-II	1.2852	1.6074	20	11.00	0.0406	1.40
PC-2-III	1.9303	1.3951	20	9.00	0.1332	4.00
PC-2-IV	2.5696	1.2062	20	8.30	0.2966	7.85
PC-2-V	4.0143	1.2654	25	9.10	0.2300	4.35
PC-2-VI	3.8578	0.8087	20	9.00	0.5210	11.16
PC-2-VII	5.6264	0.7639	25	9.00	0.9587	15.00

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Table 2.3 Data for copolymerization of 2,4,5-trichlorophenyl acrylate and methyl methacrylate

Polymer	2,4,5-TCPA (g)	MMA (g)	Benzene (mL)	Time (hr)	Copolymer (g)	Conversion (%)
first run						
TC-1-I	0.6365	2.2589	25	5.00	0.3396	11.72
TC-1-II	1.2552	2.0170	25	4.10	0.3347	10.22
TC-1-III	1.8890	1.7593	25	4.10	0.2800	7.67
TC-1-IV	2.5156	1.5071	25	4.10	0.1411	3.50
TC-1-V	2.5287	1.0017	20	6.00	0.1730	4.90
TC-1-VI	3.7792	1.0111	25	5.20	0.2059	4.30
TC-1-VII	4.4007	0.7520	25	7.00	0.5481	10.64
second run						
TC-2-I	0.5069	1.8136	20	6.00	0.3481	15.00
TC-2-II	1.2719	2.0085	25	4.00	0.3388	10.32
TC-2-III	1.8874	1.7482	25	4.00	0.2990	8.22
TC-2-IV	2.5206	1.5248	25	3.30	0.1297	3.21
TC-2-V	2.5203	1.5177	25	4.00	0.5676	14.00
TC-2-VI	2.5106	1.0035	20	6.20	0.2416	6.87
TC-2-VII	3.0204	0.8200	20	7.10	0.5416	14.10

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Table 2.4 Data for copolymerization of 2,4,6-trichlorophenyl acrylate and methyl methacrylate

Polymer	2,4,6-TCPA (g)	MMA (g)	Benzene (mL)	Time (hr)	Copolymer (g)	Conversion (%)
first run						
TP-1-I	0.5047	1.7992	20	16.00	0.2339	10.14
TP-1-II	1.5184	1.3848	20	10.07	0.2985	10.28
TP-1-III	2.0126	1.1954	20	11.20	0.3353	10.45
TP-1-IV	2.5158	1.0156	20	15.00	0.2800	7.92
TP-1-V	3.0256	0.8019	20	9.00	0.2775	7.25
TP-1-VI	3.5210	0.6156	20	9.20	0.2758	6.67
second run						
TP-2-I	0.5035	1.8081	20	9.00	0.2192	9.48
TP-2-II	1.5092	1.4032	20	10.00	0.3780	12.97
TP-2-III	2.0204	1.2496	20	9.20	0.1151	3.52
TP-2-IV	2.5201	1.0479	20	10.00	0.3153	8.83
TP-2-V	3.0218	0.7961	20	9.50	0.2952	7.73
TP-2-VI	3.5258	0.6027	20	9.40	0.3205	7.76

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Table 2.5 Data for copolymerization of 4-chloro-3-methylphenyl acrylate and methyl methacrylate

Polymer	4-Cl-3-MPA (g)	MMA (g)	Benzene (mL)	Time (hr)	Copolymer (g)	Conversion (%)
first run						
CM-1-I	0.7928	1.6051	20	7.20	0.3068	12.79
CM-1-II	1.1892	1.4078	20	8.00	0.2242	8.63
CM-1-III	1.5800	1.2080	20	7.30	0.3453	12.38
CM-1-IV	1.9700	1.0097	20	8.00	0.3209	10.76
CM-1-V	1.9731	1.0035	20	8.00	0.4413	14.82
CM-1-VI	2.4273	0.8023	20	9.00	0.2826	8.68
second run						
CM-2-I	0.8005	1.6109	20	9.20	0.3606	14.95
CM-2-II	1.1825	1.3964	20	10.00	0.1316	5.10
CM-2-III	1.5793	1.2058	20	5.55	0.3652	13.11
CM-2-IV	1.9662	1.0074	20	8.40	0.3410	11.46
CM-2-V	1.9704	1.0063	20	8.00	0.2897	9.73
CM-2-VI	2.3608	0.8024	20	7.30	0.2100	6.63

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2.4.2 High-Conversion Copolymerization

In the same manner, The high-conversion copolymerization of the chlorophenyl acrylate and methyl methacrylate was carried out in 10 mL of benzene and in the presence of α, α'-azobisisobutyronitrile at 50° C and under nitrogen atmosphere. In this case, n-dodecane (0.3513 mole/l, 2 mL) was added into the solution as an internal standard. For each chlorophenyl acrylate, seven reaction cells were prepared. Each reaction cell contained different mole fractions of the chlorophenyl acrylate comonomer in feeding, i.e. 0.8, 0.4 and 0.1. After heating for a certain length of time, the reaction was stopped and the monomer-feed composition before and after heating were determined by gas-liquid chromatography (see Section 2.6).

2.5 Determination of Copolymer Composition by UV-Visible Spectroscopy

A certain amount of each copolymer and each homopolymer was individually dissolved in chloroform (Table 2.6-2.13). A wavelength was sought at which one homopolymer and its corresponding copolymer showed a characteristic peak and the other constituents did not absorp. It was found that the wavelength at 241-243 nm is suitable one for each case. By preparing solution of various concentrations of one homopolymer, different UV-Visible spectra were recorded. A concentration-absorption profile of the homopolymer were then obtained by plotting the absorbance against the concentration of each homopolymer solution (Figure 2.1-2.4). The absorbance at wavelength 241-243 nm of the known concentration of the corresponding copolymeric samples was then obtained from their UV-Visible

spectra. Thus the concentration of the desired groups in the co-polymer was determined from the calibration curve.

Table 2.6 Relationship between concentration and ultraviolet absorption of PCPA

Concentration ($\times 10^{-5}$ M)	Absorbance at 242 nm
13.7610	0.973
10.3200	0.761
6.8810	0.529
3.4400	0.278

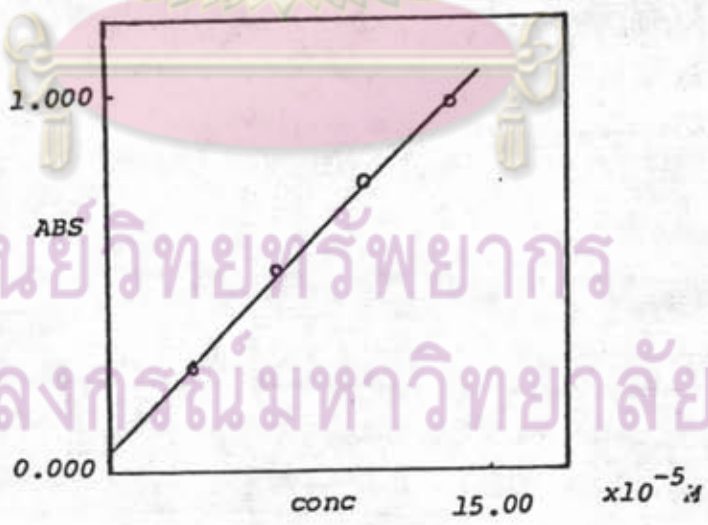

Fig. 2.1 Concentration-Absorption profile of PCPA in chloroform

$conc = (14.85 \times ABS - 0.833) \times 10^{-5}$ M

corr. coef. = 0.999

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Table 2.7 Relationship between concentration and ultraviolet absorption of poly (PCPA-co-MMA)

Poly (PCPA-co-MMA)	Weight[a] (g)	Absorbance[b]
PC-1-I	0.00340	0.729
PC-1-II	0.00250	0.850
PC-1-III	0.00160	0.676
PC-1-IV	0.00170	0.845
PC-1-V	0.00124	0.624
PC-1-VI	0.00090	0.664
PC-1-VII	0.00140	0.868
PC-2-I	0.00335	0.712
PC-2-II	0.00250	0.845
PC-2-III	0.00190	0.814
PC-2-IV	0.00154	0.755
PC-2-V	0.00120	0.616
PC-2-VI	0.00130	0.930
PC-2-VII	0.00172	0.983

a) 25 mL of $CHCl_3$ was used.

b) λ_{max} at 243 nm

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Table 2.8 Relationship between concentration and ultraviolet absorption of poly (2,4,5-TCPA)

Concentration ($\times 10^{-4}$ M)	Absorbance at 242 nm
6.9470	0.990
4.9622	0.754
3.9697	0.620
1.9849	0.278

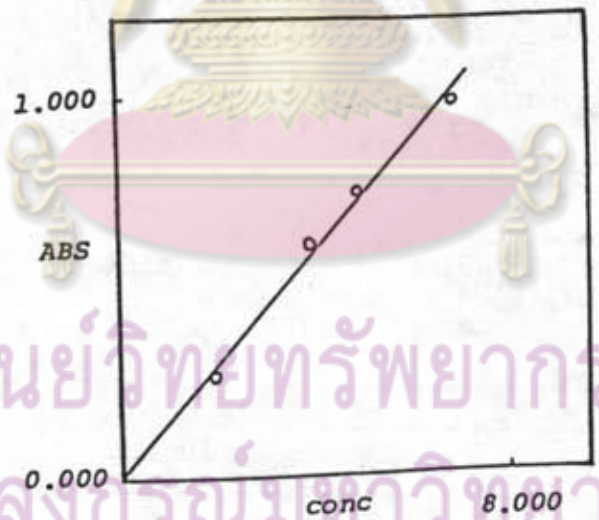

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Fig. 2.2 Concentration-Absorption profile of poly (2,4,5-TCPA) in chloroform: conc = (6.986xABS-0.149)x10^{-4}M

corr. coef. = 0.994

Table 2.9 Relationship between concentration and ultraviolet absorption of poly (2,4,5-TCPA-co-MMA)

Poly (2,4,5-TCPA-co-MMA)	Weight[a] (g)	Absorbance[b]
TC-1-I	0.0097	0.543
TC-1-II	0.0078	0.688
TC-1-III	0.0070	0.782
TC-1-IV	0.0054	0.710
TC-1-V	0.0047	0.750
TC-1-VI	0.0040	0.683
TC-1-VII	0.0047	0.831
TC-2-I	0.0117	0.648
TC-2-II	0.0058	0.521
TC-2-III	0.0052	0.597
TC-2-IV	0.0060	0.783
TC-2-V	0.0062	0.822
TC-2-VI	0.0042	0.650
TC-2-VII	0.0040	0.700

a) 25 mL of $CHCl_3$ was used.

b) λ_{max} at 242 nm

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Table 2.10 Relationship between concentration and ultraviolet absorption of poly (2,4,6-TCPA)

Concentration ($\times 10^{-3}$M)	Absorbance at 241 nm
2.3300	0.818
1.8640	0.703
1.3980	0.560
0.9320	0.394

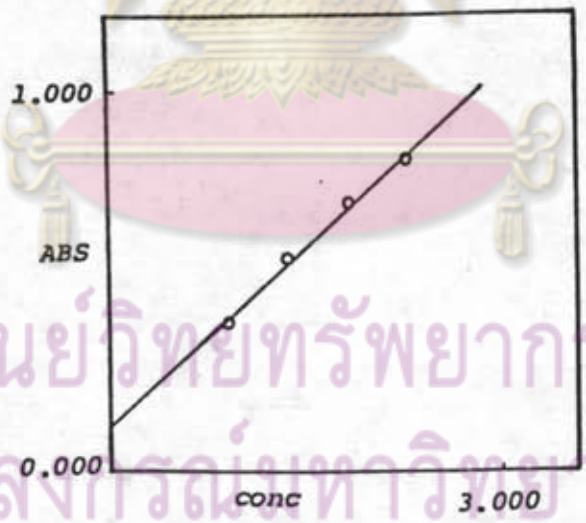

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Fig. 2.3 Concentration-Absorption profile of poly (2,4,6-TCPA) in chloroform: conc = (29.89xABS-1.969)x10^{-4}M

corr. coef. = 0.994

Table 2.11 Relationship between concentration and ultraviolet absorption of poly (2,4,6-TCPA-co-MMA)

Poly (2,4,6-TCPA-co-MMA)	Weight[a] (g)	Absorbance[b]
TP-1-I	0.0205	0.865
TP-1-II	0.0106	0.788
TP-1-III	0.0103	0.867
TP-1-IV	0.0063	0.628
TP-1-V	0.0077	0.823
TP-1-VI	0.0074	0.805
TP-2-I	0.0199	0.841
TP-2-II	0.0112	0.827
TP-2-III	0.0104	0.876
TP-2-IV	0.0084	0.826
TP-2-V	0.0093	0.983
TP-2-VI	0.0085	0.925

a) 10 mL of $CHCl_3$ was used.

b) λ_{max} at 241 nm

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Table 2.12 Relationship between concentration and ultraviolet absorption of poly (4-Cl-3-MPA)

Concentration ($\times 10^{-4}$ M)	Absorbance at 242 nm
8.2690	0.921
6.2020	0.745
5.1690	0.599
3.1010	0.365

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Fig. 2.4 Concentration-Absorption profile of poly (4-Cl-3-MPA) in chloroform: conc = (9.184xABS-0.353)x10^{-4} M

corr. coef. = 0.995

Table 2.13 Relationship between concentration and ultraviolet absorption of poly (4-Cl-3-MPA-co-MMA)

Poly (4-Cl-3-MPA-co-MMA)	Weight[a] (g)	Absorbance[b]
CM-1-I	0.0046	0.410
CM-1-II	0.0066	0.711
CM-1-III	0.0065	0.794
CM-1-IV	0.0049	0.725
CM-1-V	0.0053	0.763
CM-1-VI	0.0049	0.753
CM-2-I	0.0065	0.605
CM-2-II	0.0060	0.682
CM-2-III	0.0055	0.713
CM-2-IV	0.0047	0.727
CM-2-V	0.0059	0.869
CM-2-VI	0.0046	0.704

a) 25 mL of $CHCl_3$ was used.

b) λ_{max} at 242 nm

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2.6 Determination of Monomer Feed Composition by Gas-Liquid Chormatography

A certain amount of the chlorophenyl acrylate monomer and methyl methacrylate was dissolved in benzene (10 mL). It was then added n-dodecane (0.3513 mole/1.2 mL) into the solution as an internal standard . The GLC condition was sought at which the chlorophenyl acrylate, methyl methacrylate, benzene and n-dodecane peak could completely be separated. By preparing solution of various concentrations of each standard monomer , different chromatogram were recorded . A concentration-peak area profile of each standard monomer was then obtained by plotting the peak area against its concentration (Table 2.14-2.18 and Figure 2.5-2.8).The concentration of each monomer feed liquor before and after heating was then investigated from the corresponding profile.

The gas chromatograph was equipped with a 2 m x 3 mm i.d. stainless steel column packed with 10% SE-30 on Chrom W-AWDMCS. All determinations were performed on 1 µL injection. The conditions for analysis were as follows : column temperature program , 40°C (held for 4 min) to 230°C (held for 4 min) at 16 °C/min; FID temperature , 230°C ; injection port temperature , 230°C ; detector attenuation range, $2^6 x 10^3$ mV.V ; carrier gas, nitrogen at 30 cm^3/min, respectively; detector gases with their respective flow rates, hydrogen at 0.5 kg/cm^2 and air at 0.3 kg/cm^2 .

Results from monomer feed determination by GLC are collected in Table 2.18-2.21 where A_0 and A_t are the area ratios between monomer and n-dodecane at the initial and final condition, respectively.

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Table 2.14 Relationship between concentration and peak area of the standard pentachlorophenyl acrylate and methyl methacrylate

molar concentration		area	
[MMA]	[PCPA]	MMA	PCPA
1.0560	0.1616	5.0322	1.0522
0.7543	0.2500	3.4554	1.8385
0.4526	0.3901	2.0606	2.9884
0.3017	0.4941	1.3707	3.4362

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Fig. 2.5 Concentration-Peak Area profile of

a) MMA : conc = 0.2066xArea+0.0255 ; corr. coef. = 0.9994

b) PCPA : conc = 0.1369xArea+0.0050 ; corr. coef. = 0.9999

Table 2.15 Relationship between concentration and peak area of the standard 2,4,5-trichlorophenyl acrylate and methyl methacrylate

molar concentration		area	
[MMA]	[2,4,5-TCPA]	MMA	2,4,5-TCPA
0.4018	0.6511	1.7240	6.1254
0.6028	0.3954	2.6893	3.5701
1.0046	0.2118	4.5106	1.7483
1.2056	0.0956	5.5303	0.6791

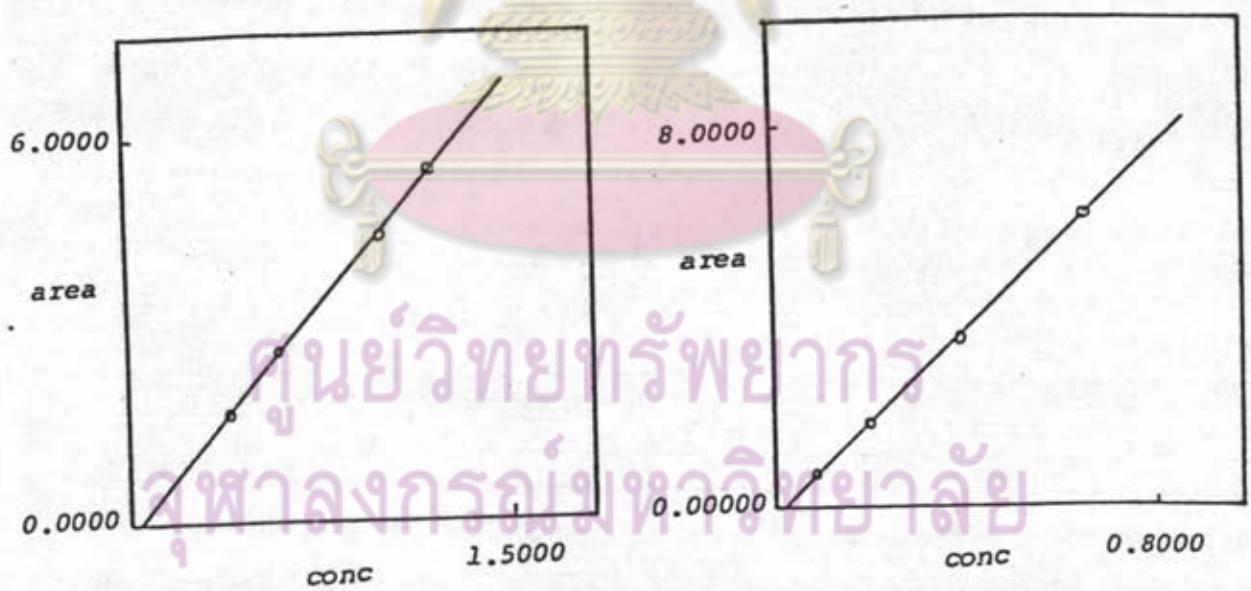

Fig. 2.6 Concentration-Peak Area profile of

a) MMA : conc = 0.2129xArea+0.0343 ; corr. coef. = 0.9998

b) 2,4,5-TCPA : conc = 0.1015xArea+0.0305 ;

corr. coef. = 0.9998

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Table 2.16 Relationship between concentration and peak area of the standard 2,4,6-trichlorophenyl acrylate and methyl methacrylate

molar concentration		area	
[MMA]	[2,4,6-TCPA]	MMA	2,4,6-TCPA
0.3896	0.7011	1.6699	4.7093
0.5194	0.5008	2.2924	3.4028
0.6493	0.4006	2.9168	2.6819
0.9090	0.3005	4.1022	2.0562

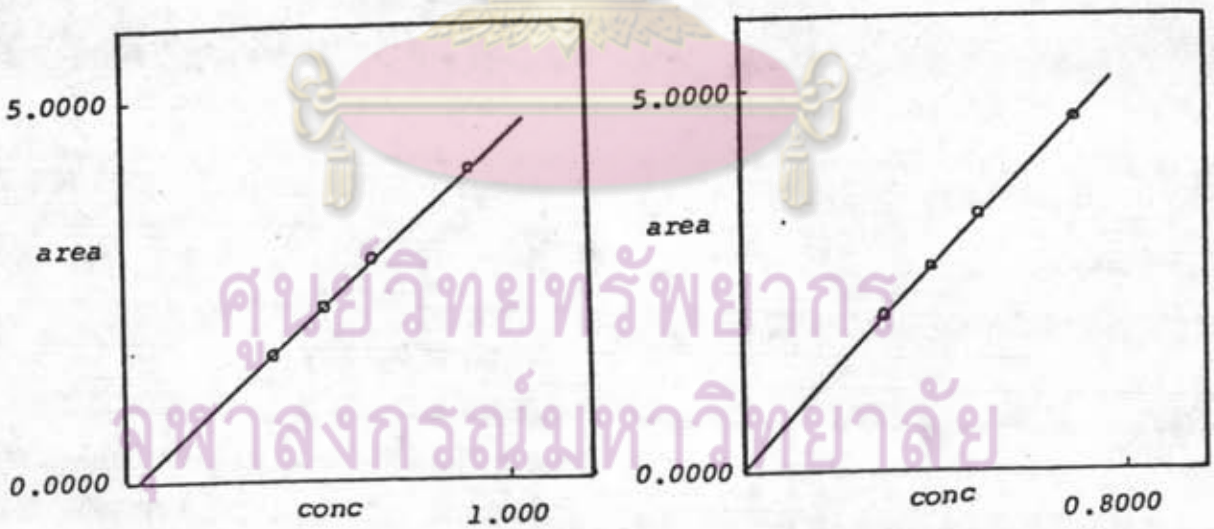

Fig. 2.7 Concentration-Peak Area profile of

a) MMA : conc = 0.2137xArea+0.0302 ; corr. coef. = 0.9999

b) 2,4,6-TCPA : conc = 0.1495xArea-0.0051 ;

corr. coef. = 0.9998

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Table 2.17 Relationship between concentration and peak area of the standard 4-chloro-3-methylphenyl acrylate and methyl methacrylate

molar concentration		area	
[MMA]	[4-Cl-3-MPA]	MMA	4-Cl-3-MPA
0.9101	0.1802	4.0470	1.1330
0.6500	0.3605	2.8630	2.3110
0.5200	0.4506	2.3340	3.1360
0.2600	0.6308	1.1370	4.1570

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Fig. 2.8 Concentration-Peak Area profile of

a) MMA : conc = 0.2240xArea+0.0040 ; corr. coef. = 0.9998

b) 4-Cl-3-MPA : conc = 0.1470xArea+0.0110;

corr. coef. = 0.9996

Table 2.18 GLC analytical data of the copolymerization of MMA with PCPA

	A_0		TIME	A_t	
	MMA	PCPA	(hr)	MMA	PCPA
I-5	4.8868	0.4177	12.00	3.5780	0.3002
II-5	4.8868	0.4177	13.30	2.7073	0.2388
III-15	3.9657	0.7674	11.00	2.6657	0.5170
IV-18	3.1410	1.0718	9.05	1.7524	0.6601
V-50	1.9179	3.3949	10.00	0.9886	2.5444
VI-50	1.9179	3.3949	11.00	0.8884	2.4801

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Table 2.19 GLC analytical data of the copolymerization of MMA with 2,4,5-TCPA

	A_0		TIME	A_t	
	MMA	2,4,5-TCPA	(hr)	MMA	2,4,5-TCPA
I-6	3.6519	0.2254	8.00	2.3832	0.0335
II-9	4.7412	0.7165	8.00	2.9939	0.3346
III-40	2.5340	3.6683	6.00	1.2842	1.9764
IV-60	1.8915	5.4636	6.00	0.7459	2.9183
V-65	1.2475	5.4193	6.00	0.5369	3.3681

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Table 2.20 GLC analytical data of the copomerization of MMA with 2,4,6-TCPA

	A_0		TIME	A_t	
	MMA	2,4,6-TCPA	(hr)	MMA	2,4,6-TCPA
I-10	5.1862	0.7879	13.00	4.7047	0.6983
II-10	5.1862	0.7879	14.00	4.3808	0.6448
III-50	1.2096	2.2394	10.00	0.3388	1.4067
IV-50	1.2096	2.2394	11.00	0.2344	1.0715
V-80	0.4450	2.9337	9.00	0.1624	2.2180

Table 2.21 GLC analytical data of the copolymerization of MMA with 4-Cl-3-MPA

	A_0		TIME	A_t	
	MMA	4-Cl-3-MPA	(hr)	MMA	4-Cl-3MPA
I-10	4.2178	0.5639	10.00	2.5848	0.2605
II-10	3.8580	0.6020	7.30	3.1129	0.4544
III-30	2.9933	1.8299	7.30	2.2165	1.3415
IV-40	2.1263	2.5674	9.30	1.2273	1.5109
V-80	0.5415	2.8742	9.30	0.0987	1.3612

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