

# Methyl Methacrylate–*N*-Chlorophenyl Maleimide Copolymers: Effect of Structure on Properties

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**ABSTRACT:** The article describes the preparation of cast acrylic sheets by copolymerizing methyl methacrylate (MMA) with varying mole fractions of *N*-*o*-chlorophenyl maleimide (OC), *N*-*m*-chlorophenyl maleimide (MC), and *N*-*p*-chlorophenyl maleimide (PC) using benzoyl peroxide as an initiator. The effect of incorporation of varying mole fraction of *N*-chlorophenyl maleimides in poly(methyl methacrylate) backbone on the optical, physicomechanical, and thermal properties of cast acrylic sheets were evaluated. Vicat softening temperature, glass transition temperature ( $T_g$ ) and thermal stability of the copolymers increased with an increase in the maleimide content. The solar transmittance and percentage of transmittance of the copolymer sheets having low mole fractions of *N*-chlorophenyl maleimides (i.e., 0.025–0.085) were found to be comparable with that of PMMA. Further increase in the comonomer content resulted in a decrease in the percentage of transmittance. Copolymer sheets having low mole fractions of *N*-chlorophenyl maleimides (0.025–0.108) have tensile strength comparable to that of PMMA, whereas the percentage of elongation decreased. Tensile modulus increased with increasing mole fraction of *N*-chlorophenyl maleimides. © 1998 John Wiley & Sons, Inc. *J Appl Polym Sci* 68: 527–534, 1998

**Key words:** copolymerization; methyl methacrylate; *N*-*o*-chlorophenyl maleimides; *N*-*p*-chlorophenyl maleimides; MMA copolymers

## INTRODUCTION

Methyl methacrylate (MMA) has been copolymerized with a variety of vinyl monomers to yield polymers with improved melt flow indices, better impact strength, abrasion resistance, and heat characteristics.<sup>1–8</sup> The ability to tailor the rigid and stable methacrylates alone or in combination with acrylates to fit specific application requirements, such as outstanding clarity and dimensional stability, and the unusual chemical and light stability of this class of materials has in-

creased its growth and have made methacrylic esters a prime candidate for numerous applications. These copolymers find applications in paints, plastics, automotive coatings, viscosity index improvers, medicines, adhesives, and substitutes for glass in solar collectors.<sup>9–12</sup> Poly(methyl methacrylate) (PMMA) sheets have been used as a substitute for glass in solar collector covers primarily because of their light weight, excellent weather resistance, light transmittance, and good insulating properties. However, one of the limitations of PMMA is its low softening point ( $\sim 90^\circ\text{C}$ ), due to which it undergoes distortion when used as inner glazing material.<sup>2,3</sup>

The main objective of the present work was to modify the structure of PMMA so that its  $T_g$  can

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be enhanced without affecting its useful properties. In our earlier articles,<sup>13,14</sup> we have reported the solution copolymerization of *N*-aryl maleimides [having electron releasing ( $-\text{CH}_3$ ) and electron withdrawing ( $-\text{Cl}$ ) groups] with MMA using free radical initiator. An increase in  $T_g$  and thermal stability have been observed upon such copolymerization. Although extensive investigation has been carried out on the copolymerization behavior of MMA with several vinyl monomers, very few reports are available on the physico-mechanical properties of these copolymers. Hence, it was considered of interest to examine systematically the effect of comonomer structure (that is, *o*-, *m*-, and *p*-chlorophenyl maleimide) and composition on the physico-mechanical, thermal, and optical properties of PMMA. Various copolymer sheets were therefore fabricated using varying mole fractions of comonomers in the initial feed.

## EXPERIMENTAL

### Materials

Methyl methacrylate (CDH) was purified by washing with dilute solution of sodium hydroxide followed by water. It was dried over anhydrous sodium sulphate and distilled under reduced pressure. *N*-*o*-chlorophenyl maleimide (OC), *N*-*m*-chlorophenyl maleimide (MC), and *N*-*p*-chlorophenyl maleimide (PC) were prepared according to the method reported in our earlier article.<sup>13</sup> Benzoyl peroxide (BPO) (E. Merck) was recrystallized from chloroform.

### Fabrication of Copolymer Sheets

#### Preparation of Prepolymer Syrup

Prepolymer syrup was prepared by bulk polymerization of MMA (inhibitor-free) using benzoyl peroxide (0.3% w/w) as an initiator. The reaction was carried out by heating the mixture at 90°C in nitrogen atmosphere for 15–20 min. The reaction was arrested by cooling the mixture in ice cold water. Required quantities of MMA prepolymer syrup, comonomer, UV stabilizer (benzotriazole), releasing agent, and BPO were mixed thoroughly using a mechanical stirrer for 30 min. Filtration was done using cheesecloth for removing suspended particles. It was then degassed to remove the trapped air bubbles.

### Preparation of the Mold

A poly(vinyl chloride) (PVC) gasket of 3 mm diameter was placed between two toughened glass plates of  $35 \times 30 \text{ cm}^2$ . The gasket was held in place by thin wires, and the glass plates were clamped together. One side of the glass plate was kept open for pouring the prepolymer syrup. A small strip of mylar sheet was used as a funnel for filling the mold.

### Casting of Sheets

The deaerated syrup was poured into the mold. The wires holding the PVC gasket were pulled out to remove any air bubbles that may be present in the mold. The filled mold was then placed in a preheated water bath at 70–80°C for 4 h. The mold was removed, and post-curing was carried out in the hot air oven at 90–120°C for 3 h. Later, it was allowed to cool. The clamps were then removed, and copolymer sheets were taken out. Sixteen copolymer sheets were prepared by varying the mole fraction of *N*-chlorophenyl maleimides (OC, MC, and PC) in the feed from 0.025 to 0.171. Samples were designated by adding a prefix "S" to the letter designation of the *N*-chlorophenyl maleimide monomer and a suffix indicating the mol % of the *N*-chlorophenyl maleimide taken in the initial feed. For example, a copolymer sheet prepared by taking 2.5 mol % of PC, MC, or OC has been designated as SPC-2.5, SMC-2.5, and SOC-2.5, respectively.

## CHARACTERIZATION

Intrinsic viscosity of the copolymer sheets was determined in chloroform at  $30 \pm 0.1^\circ\text{C}$  using Ubbelohde suspension level viscometer. Density of the polymer sheets was determined by displacement method in accordance to ASTM D-792.

### Thermal Characterization

Vicat softening temperature (VST) was measured according to ASTM D-1525 on a Ceast 6505/111 instrument using VST measurement assembly. A flat-ended needle loaded with specified mass was placed in direct contact with test specimen having dimensions of  $1 \times 1 \times 0.3 \text{ cm}^3$ . The specimen needle was heated at a rate of 120°C/h. The temperature at which the needle penetrates to the depth of 1 mm was recorded as the VST. Two

specimens were used to test each sample, and the average value was reported.

The glass transition temperature was determined by differential scanning calorimetry (DSC) using a DuPont 9900 thermal analyzer having a 910 DSC module. A sample size of  $6 \pm 1$  mg and a heating rate of  $10^\circ\text{C}/\text{min}$  were used. For recording DSC scans, powdered samples were obtained by dissolving a small piece of copolymer sheet in chloroform, followed by precipitation in methanol. In order to have the same thermal history, the powdered samples were heated in a DSC cell from 50 to  $150^\circ\text{C}$  in a static air atmosphere at a heating rate of  $10^\circ\text{C}/\text{min}$  and then allowed to cool in the DSC cell. The same sample was then reheated to  $250^\circ\text{C}$ , and a DSC scan was recorded at a heating rate of  $10^\circ\text{C}/\text{min}$ . The glass transition region was characterized by noting the following temperatures:  $T_e$ , extrapolated onset temperature;  $T_g$ , midpoint temperature; and  $T_f$ , extrapolated endset temperature.

Thermal stability of the samples was determined by recording TG/DTG traces in nitrogen atmosphere (flow rate was  $60 \text{ cm}^3/\text{min}$ ) using a DuPont 9900 thermal analyzer having a 951 TG module. A heating rate of  $10^\circ\text{C}/\text{min}$  and a sample size of  $10 \pm 1$  mg was used in each experiment.

### Mechanical Properties

Tensile properties were measured according to ASTM D-638, using an Instron tensile tester model 4301. The samples were conditioned for 48 h at  $25^\circ\text{C}$  and a relative humidity of 50% before testing. Dumbbell-shaped specimens (length, 112 mm; width of narrow section, 6 mm; gauge length, 30 mm) were made from the cast sheet by cutting rectangular pieces and shaping them using a metal mold. The edges were smoothed using fine sand paper. For each composition, a minimum of five samples were tested, and the average value was taken.

Flexural properties were determined in accordance to the ASTM D790-81 using an Instron tensile tester model 1121. The specimens having dimensions of  $120 \times 12.5 \text{ mm}^2$  were used. A span length of 60 mm, a full-scale load of 50 kg, a chart speed of 100 mm/min, and a crosshead speed of 5 mm/min was used.

Impact strength of notched specimens was measured according to ASTM 256 using impact tester Ceast 6545/000. Samples were conditioned for 48 h before testing. Five samples for each com-

position were used for testing, and an average value is reported.

### Optical Properties

Solar transmittance measurement was done using D & S Alphotometer, which is a miniaturized pyranometer. The amount of solar energy transmitted through the material was compared with the amount of incident solar energy to determine the transmittance of a material.

Turbidity ( $\tau$ ) of the sheets was calculated using the Beer-Lambert extinction law, as follows:

$$\tau = -(1/x) \ln T$$

where  $x$  is the thickness of the sheet, and  $T$  is the fraction of light transmitted by the sheet. The ASTM D-1003 test procedure was used to measure the percentage of transmittance and the percentage of haze of the sheets using XL-211 Hazegard system.

### Chemical Resistance

The chemical resistance of the copolymer sheets was evaluated in accordance to the ASTM D-543. Weighed samples having dimensions of  $1 \times 1 \times 0.3 \text{ cm}^3$  were immersed in different solvents, such as  $\text{H}_2\text{O}$ , 15%  $\text{HCl}$ , 25%  $\text{H}_2\text{SO}_4$ , 25%  $\text{CH}_3\text{COOH}$ , 10%  $\text{NH}_3$ , 5%  $\text{H}_2\text{O}_2$ , and  $\text{CCl}_4$ . The change in weight and thickness of sheets after 7 days was noted.

## RESULTS AND DISCUSSION

All the sheets were clear, transparent, and free from surface defects. A yellow tinge was observed in the cast sheets having 0.108 mole fraction of *N*-chlorophenyl maleimides. The intensity of the color increased as the maleimide content in the copolymer sheets increased. The monomers OC, MC, and PC were yellow in color, and this coloration in the sheets could be due to the presence of either unreacted monomer or oligomers. This aspect was investigated by dissolving the sheet samples in chloroform and reprecipitating in

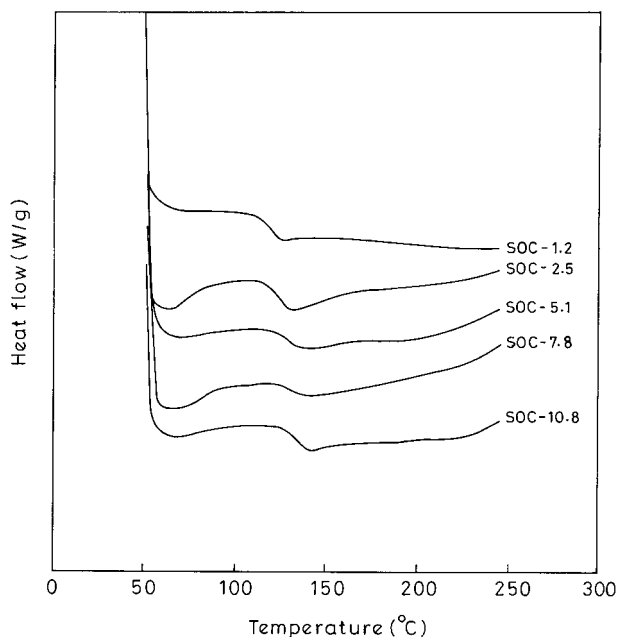
**Table I** Fabrication of Sheets of MMA and *N*-Aryl Maleimides

Sample	<i>N</i> -Aryl Maleimide	Mole Fraction of <i>N</i> -Aryl Maleimide	Thickness of Sheet (mm)	$\rho$ (g/cm <sup>3</sup> )	$[\eta]$ (dL/g)
PMMA	—	0.00	2.81	1.186	3.84
SPC-2.5	PC	0.025	2.72	1.196	3.5
SPC-3.9	PC	0.039	2.75	1.206	3.24
SPC-5.1	PC	0.051	2.8	1.209	3.2
SPC-6.4	PC	0.064	3.21	1.216	2.98
SMC-2.5	MC	0.025	3.1	1.199	3.02
SMC-5.1	MC	0.051	3.12	1.211	2.87
SMC-7.8	MC	0.085	3.2	1.225	2.76
SMC-10.8	MC	0.108	3.46	1.239	2.48
SMC-13.8	MC	0.138	3.41	1.247	2.4
SMC-17.1	MC	0.171	2.77	1.251	2.34
SOC-1.2	OC	0.012	2.98	1.185	2.98
SOC-2.5	OC	0.025	3.02	1.190	2.42
SOC-5.1	OC	0.051	2.75	1.197	2.48
SOC-7.8	OC	0.085	3.12	1.219	2.13
SOC-10.8	OC	0.108	3.14	1.230	1.5

methanol. The reprecipitated polymer was colorless, indicating that the color was due to the unreacted monomer. Similar observation has already been reported in our earlier article.<sup>8</sup> Copolymer sheets having higher mole fraction of OC/MC/PC comonomers could not be prepared because of their limited solubility in MMA monomer. The position of the substituent also affected the solubility of *N*-chlorophenyl maleimides in MMA mono-

mer. PC monomer was least soluble; therefore, sheets having very low mole fractions (0.025–0.064) of PC could be fabricated (Table I).

The density of the cast sheets determined at a temperature of 30°C increased with increasing mole fraction of maleimide content. Intrinsic viscosity  $[\eta]$  of the copolymers were found to decrease with increasing amount of *N*-chlorophenyl maleimide in the initial feed (Table I).  $[\eta]$  values for the copolymer sheets are much higher than the

**Figure 1** DSC scans of MMA–OC copolymers.**Table II** Results of DSC Scans of Copolymer Sheets in Static Air Atmosphere (Heating Rate of 10°C/min)

Sample	$T_e$ (°C)	$T_g$ (°C)	$T_f$ (°C)
SPC-2.5	114	120	125
SPC-3.9	109	118	126
SPC-5.1	115	123	132
SPC-6.4	117	129	136
SMC-2.5	122	126	139
SMC-5.1	122	129	137
SMC-7.8	123	130	138
SMC-10.8	117	132	149
SMC-13.8	123	133	139
SMC-17.1	130	138	142
SOC-1.2	115	122	129
SOC-2.5	121	125	130
SOC-5.1	125	131	135
SOC-7.8	131	135	138
SOC-10.8	135	139	142

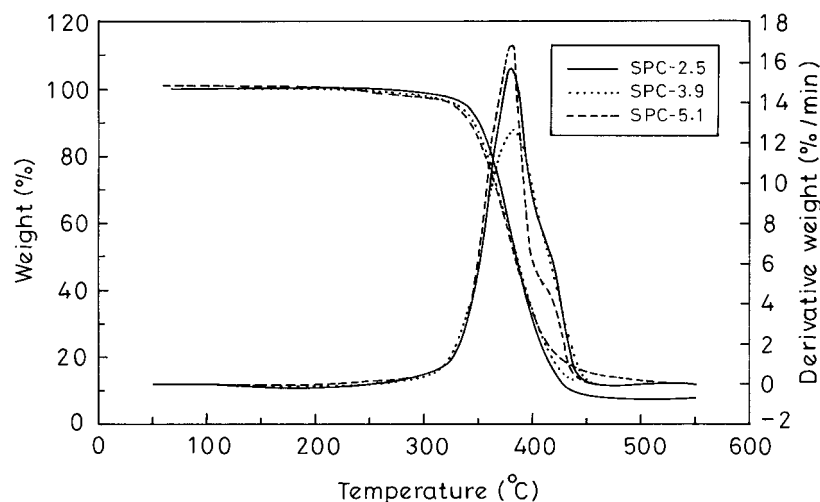


Figure 2 TG-DTG traces for MMA-PC copolymers.

values obtained for copolymers prepared by solution polymerization carried up to 10–15% conversion.<sup>13</sup> Fabrication of sheets was carried out by bulk polymerization, and the reaction was carried up to 100% conversion. In such polymerization systems, viscosity of the medium was much higher as compared to solution polymerization. Under such conditions, termination reactions are hindered due to the gel effect, which might be responsible for the higher values of  $[\eta]$ . A gel effect during the polymerization of MMA has also been reported above 15% conversion.<sup>15</sup>

The DSC scans of the MMA-OC copolymers are shown in Figure 1, and the results are summarized in Table II. Incorporation of even 0.026 mole fraction of PC-MC-OC in PMMA backbone resulted in an increase in  $T_g$ . The  $T_g$  of PMMA was

observed at 114°C. About 20°C, an increase in  $T_g$  was observed upon incorporation of 0.108 mole fraction of OC or MC (SOC-10.8 and SMC-10.8). Position of the chloro substituent in the *N*-chlorophenyl maleimide also affected the  $T_g$ . Comparison of  $T_g$  for the copolymers having equal mole fractions of OC, MC, or PC comonomers (that is, SPC-5.1, SMC-5.1, and SOC-5.1) showed higher values for SOC and lower values for SPC samples.

The thermal stability of the copolymers was determined by recording TG-DTG traces in  $N_2$  atmosphere. Typical TG-DTG traces for MMA-PC copolymers are shown in Figure 2, and the results are summarized in Table III. Thermal stability of the samples was compared by noting the initial decomposition temperature ( $T_i$ ), temperature of maximum rate of weight loss ( $T_{max}$ ), final

Table III Thermal Behavior of Copolymer Sheets in  $N_2$  Atmosphere (Heating Rate of 10°C/min)

Sample	$T_i$ (°C)	$T_{max}$ (°C)	$T_f$ (°C)	Percentage of Char at 500°C	Percentage of Weight Loss	
					250–350°C	350–500°C
SPC-2.5	345	381	413	8.59	10.16	81.25
SPC-3.9	347	383	416	12.5	10.94	76.56
SPC-5.1	353	381	410	14.1	10.94	75
SMC-2.5	323	338	345	6.69	46.46	46.85
	366	410	430			
SMC-5.1	338	367	414	5.04	20.86	74.1
SMC-7.8	316	343	359	7.04	43.89	48.55
	402	414	430			
SMC-10.8	326	354	396	10.55	32.82	56.63
SMC-13.8	329	358	395	8.84	33.86	56.3
SMC-17.1	320	347	388	13.04	40.58	46.38

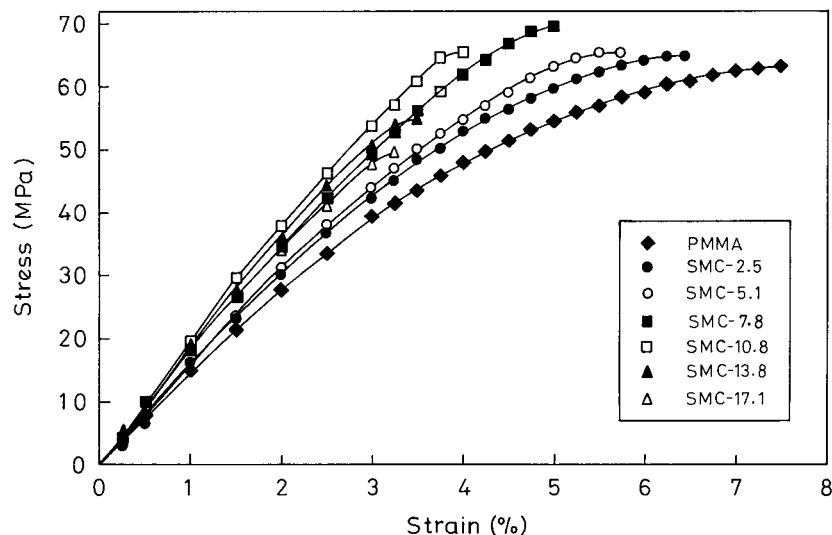


Figure 3 Stress-strain curves of MMA-MC copolymers.

decomposition temperature ( $T_f$ ), and percentage of char yield at 500°C. PMMA showed a three-step decomposition,<sup>13</sup> whereas a single-step decomposition was observed in all the copolymer samples except SMC-2.5 and SMC-7.8.  $T_i$ ,  $T_{max}$ , and  $T_f$  did not show any definite trend as a function of copolymer composition. However the percentage of char yield increased with increasing comonomer content. All the samples were stable up to 320°C; after that, they start losing weight.

Stability was also compared by comparing the weight loss in the temperature ranges of 250–350

and 350–500°C (Table III). In the case of MMA-PC copolymers, weight loss in the temperature range of 250–350°C was  $10.5 \pm 0.5\%$ , whereas it decreased in the temperature range of 350–500°C with increasing comonomer content. In the case of MMA-MC copolymers, the percentage of weight loss increased in the temperature range of 250–350°C, whereas, in the temperature range of 350–500°C, it decreased with increasing comonomer content. From these results, it can be concluded that the incorporation of maleimide monomers in the PMMA backbone results in a change

Table IV Mechanical Properties of Copolymer Sheets

Sample	Tensile Strength (MPa)	Percentage of Elongation	Tensile Modulus (MPa)	Flexural Strength (MPa)	Flexural Modulus (MPa)
PMMA	63	8.2	1187	102	5684
SPC-2.5	55	7.0	1110	103	5638
SPC-3.9	63	5.8	1376	112	5665
SPC-5.1	65	5.5	1472	111	5448
SPC-6.4	68	5.3	1680	115	5493
SMC-2.5	64	6.4	1627	114	5190
SMC-5.1	65	5.8	1687	117	5468
SMC-7.8	69	5.1	1799	119	5521
SMC-10.8	65	4.0	2131	122	5344
SMC-13.8	56	3.5	1877	101	5483
SMC-17.1	50	3.3	1597	113	5456
SOC-1.2	69	8.4	1557	111	4673
SOC-2.5	67	5.5	1763	110	5573
SOC-5.1	65	4.5	1745	106	5164
SOC-7.8	59	3.8	1845	101	4381
SOC-10.8	58	3.4	1942	95	4517

**Table V Izod Impact Strength and Vicat Softening Temperature of the Sheets**

Sample	Impact Strength (J/m)	Vicat Softening Temperature (°C)
PMMA	22.71	106.8
SPC-2.5	21.5	—
SPC-3.9	19.1	115.5
SPC-5.1	17.33	116.8
SPC-6.4	16.81	116.9
SMC-2.5	21.24	117.7
SMC-5.1	19.8	116.3
SMC-7.8	19.5	114.8
SMC-10.8	15.0	111.9
SMC-13.8	13.67	—
SMC-17.1	12.18	115.6
SOC-1.2	23.33	121.5
SOC-2.5	21.25	124.7
SOC-5.1	21.08	116
SOC-7.8	18.47	114.8
SOC-10.8	16.47	115.1

in degradation mechanism and eliminates the formation of weak linkages, such as head-to-head and unsaturated end groups.

The stress-strain curves of MMA-MC copolymers are shown in Figure 3, and the results of tensile properties are summarized in Table IV. Tensile strength of the copolymer sheets having low mole fractions of maleimides (0.025–0.108) were comparable to that of PMMA. A further increase in the maleimide content resulted in a de-

crease in tensile strength. Tensile modulus increased, whereas the percentage of elongation decreased with an increasing maleimide content in the copolymer sheets. All the copolymer sheets have lower flexural modulus as compared to PMMA. No definite trend was observed in the flexural modulus values as a function of copolymer composition. Flexural strength increased with increasing maleimide content in the copolymer samples, except for samples SMC-13.8 and SMC-17.1, where lower values were observed. Impact strength decreased with increasing maleimide content in the copolymers (Table V).

All the copolymers had Vicat softening temperature higher than PMMA. At low mole fractions (that is, up to 0.025) of OC, MC, and PC, a significant effect was observed as a function of the position of electron-withdrawing substituent. On further increase in comonomer content, the position of substituent did not affect the VST (Table V).

The solar transmittance of the copolymer sheets (0.025–0.108 mole fraction) at 45° inclination were found to be comparable with that of PMMA (Table VI). Only in the case of the MMA-MC copolymer sheet having 0.138 mole fraction of MC (SMC-13.8) or more, a slight decrease in solar transmittance was observed. A marginal decrease in percentage of transmittance and an increase in turbidity was observed with an increase in the maleimide content in the copolymers. The percentage of transmittance and percentage of

**Table VI Optical Properties of Copolymer Sheets**

Sample	Solar Transmittance	Turbidity ( $\tau$ )	Percentage of Transmittance	Percentage of Haze
PMMA	0.88	0.048	91.0	1.7
SPC-2.5	0.86	0.055	92.4	0.0
SPC-3.9	0.87	0.051	91.9	0.0
SPC-5.1	0.86	0.054	91.9	0.9
SPC-6.4	0.85	0.051	91.3	3.7
SMC-2.5	0.87	0.049	92.4	4.2
SMC-5.1	0.86	0.048	91.7	6.4
SMC-7.8	0.86	0.047	90.0	13.6
SMC-10.8	0.83	0.054	83.0	20.0
SMC-13.8	0.78	0.073	75.4	6.2
SMC-17.1	0.73	0.113	67.2	22.5
SOC-1.2	0.88	0.043	92.6	1.6
SOC-2.5	0.87	0.046	92.2	1.9
SOC-5.1	0.87	0.051	91.4	3.4
SOC-7.8	0.86	0.048	90.4	3.1
SOC-10.8	0.84	0.056	86.8	6.5

**Table VII Percentage of Weight Change After 7 Days**

Sample	25% H <sub>2</sub> SO <sub>4</sub>	15% HCl	20% CH <sub>3</sub> COOH	H <sub>2</sub> O	10% NH <sub>3</sub>	5% H <sub>2</sub> O <sub>2</sub>	CCl <sub>4</sub>
PMMA	0.496	0.401	0.643	0.728	0.78	0.9	0.128
SPC-2.5	0.029	0.491	0.669	0.449	0.699	0.827	0.227
SPC-3.9	0.294	0.579	0.698	0.521	0.643	0.603	0.088
SPC-5.1	0.029	0.463	0.593	0.325	0.906	0.765	0.429
SPC-6.4	0.045	0.708	0.931	0.578	0.432	0.45	0.366
SMC-2.5	0.175	0.241	0.504	0.448	0.498	0.609	0.128
SMC-5.1	0.539	0.499	0.586	0.643	0.931	0.826	0.173
SMC-7.8	0.564	0.141	0.27	0.108	0.644	0.349	0.017
SMC-13.8	0.008	0.126	0.269	0.108	0.29	0.259	0.369
SMC-17.1	0.369	0.214	0.682	0.307	0.939	0.5	0.018
SOC-1.2	0.128	0.304	0.466	0.444	0.48	0.775	0.13
SOC-2.5	0.323	0.569	0.829	0.399	0.739	0.731	0.401
SOC-5.1	0.367	0.46	0.637	0.809	0.596	0.648	0.276
SOC-7.8	0.029	0.524	0.362	0.552	0.441	0.488	0.427
SOC-10.8	0.028	0.195	0.624	0.527	0.422	0.804	0.289

haze of the copolymer sheets having low mole fractions of maleimide (0.025–0.085) were comparable. An increase in the percentage haze was also observed for copolymer sheets having higher mole fractions of maleimide (Table VI).

The change in weight of the samples was determined after immersion in the chemical reagents for 7 days. The results are given in Table VII. It was observed that all the sheets showed a marginal increase in weight (that is, <1%) after immersion in different solvents for 7 days. The thickness of the samples was also noted before and after immersion. A change in sample thickness was observed after immersion in 5% H<sub>2</sub>O<sub>2</sub> and 15% HCl, whereas, in all other reagents, it remained unchanged.

## CONCLUSION

From these results, it can be concluded that transparent acrylic sheets having higher glass transition temperature and better thermal stability could be fabricated using very low mole fractions (up to 0.108) of *N*-chlorophenyl maleimide without affecting the mechanical properties.

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