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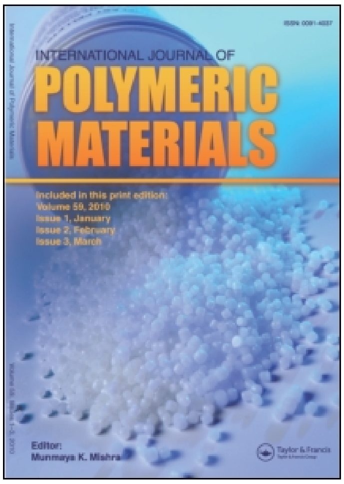

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Synthesis and Properties of N-cyclohexylmaleimide-Containing Graft-type MCS Resin

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Synthesis and Properties of N-cyclohexylmaleimide-Containing Graft-type MCS Resin

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N-cyclohexylmaleimide-containing Graft-type MCS resin was synthesized by suspension polymerization. Effects of graft conditions, such as initiator and monomer mixture composition, on graft efficiency (GE) were studied. GE has maximum value when the initiator concentration is 0.3% of the monomer content, and increases with increasing of cyclohexylmaleimide (ChMI) content at fixed initiator concentration. Effects of monomer mixture composition on the properties of the copolymers were also investigated. The results show that the molecular weight ($\bar{M}_n$ *and* $\bar{M}_w$)*,* T_g*,* T_{Vicat}*, and degradation temperature of the copolymers increased with increasing ChMI moieties in the copolymer. The mechanical properties (impact strength and tensile strength) were increased due to the presence of ChMI. The apparent viscosity* η_a *and the non-newtonian flow parameter power n increase with increasing content of ChMI, and the melt is a pseudoplastic liquid. The morphological structure of graft-type MCS resin was investigated by scanning electron microscopy (SEM).*

Keywords: MCS resin; Graft copolymer; N-cyclohexylmaleimide

INTRODUCTION

MCS resin is a terpolymer of methyl methacrylate (MMA), chlorinated polyethylene (CPE), and styrene (St), which possesses characteristics of fire resistance, weather fastness, and some transparency. The blending of the copolymer of MMA and St (PMS) with chlorinate

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CPE and graft copolymerization are the two main synthesis methods to produce MCS resin [1–3]. However, the blending method can not produce uniform phase because CPE possesses high melt viscosity, which not only affects the mechanical properties of the material, but also makes the blending process of the MCS resin difficult. Graft copolymerization could overcome the above deficiencies.

In recent years, N-substituted maleimides have been found to be the most valuable monomers, because they contain a rigid planar ring, which can effectively enhance the thermal properties of copolymers [4–6]. Taking the property and price into account ChMI was found to be the best one among these monomers. In the past, methyl acrylate modified graft-type MCS had been synthesized by suspension polymerization, the effects of different composition of monomers on mechanical properties, rheological behavior, and morphology had been investigated [7]. However, the synthesis and the properties of N-cyclohexylmaleimide-containing graft-type MCS resin have not been reported until now.

In this article, the ChMI was incorporated into graft-type MCS resin as a modifier. The relationship between composition of the monomer mixture and graft efficiency, molecular weight, T_g, T_{Vicat}, degradation temperature, rheological behavior, mechanical properties, and morphological structure is discussed.

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EXPERIMENTAL

Material

The monomers methyl methacrylate (MMA) and Styrene (St) were supplied by Beijing Chemical Reagent Co. N-cyclohexylmaleimide was supplied by ZhuoZhou Haihui Chemicals Co. Acetone, tetrahydrofuran, sodium dodecysulfate (SDS), potassium persulfate ($K_2S_2O_8$), $Al_2(SO_4)_3 \cdot 18H_2O$, benzoyl peroxide (BPO), azobisisobutyronitrile (AIBN), and Na_2CO_3 were all of analytically pure grade and supplied by Tianjin Chemical Reagent Co. Polyvinyl alcohol (PVA), chlorinated polyethylene (degree of chlorination 35%), tribasic lead sulfate (TLS), dibasic lead phosphite (DLP), barium stearate (BS), and paraffin wax were all of commercial grade.

Synthesis of Emulsion Copolymer

The copolymer PCMS of N-cyclohexylmaleimide, methyl methacrylate, and Styrene was synthesized by emulsion polymerization. The monomers, MMA and St, were freshly distilled and N-cyclohexylmaleimide was recrystallized twice from a mixture of alcohol and water. SDS was used as an emulsifier, and $K_2S_2O_8$ was used as initiator. Distilled deionized water (DIW) was used in polymerization. All components were purged under N_2 for 20 min. The 0.72 g of SDS and 36 g of DIW were charged into a 100 ml four-necked glass flask. The reaction vessel was heated to 80°C with continuous stirring. Then the monomer mixture (MMA/St/ChMI = 25/25/25, by weight) was added gradually to the flask together with the prepared solution of 0.04% $K_2S_2O_8$ over the course of 1.5 h under a nitrogen atmosphere. The reaction was held at 80°C for an additional 0.5 h and then at 90°C for another 1 h. The copolymer was precipitated by 3% $Al_2(SO_4)_3 \cdot 18H_2O$ solution and purified by washing with warm DIW, then it was dried in vacuum. The conversion ratios of monomers were over 98%.

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Synthesis of Graft-Type MCS Resin

A series of graft-type MCS resin was synthesized by suspension polymerization. MMA and CPE were fixed at 25% (by wt), the content of ChMI in graft-type MCS was increased from 0, 5, 10, 15, 20, to 25% (St decreased form 50% to 25% accordingly, by wt). After the mixture of MMA, St, ChMI, and initiator (BPO or AIBN) swelled the chlorinated polyethylene for 12 h in a four-necked glass flask, 0.4% polyvinyl alcohol solution was added into the swelling system (oil/water = 1/5). Then the reaction vessel was heated to 80°C in N_2 atmosphere, stirring continuously. The reaction was held at 80°C for 4 h, and then at 90°C for additional 3 h. The product was purified by washing with warm DIW, and then dried in vacuum. The conversion ratios of monomers were over 98%.

Measurement of Graft Efficiency of Monomers

Graft-type MCS resin was extracted in a Soxhlet's Extractor (Beijing, China) [8], using a mixture of acetone and tetrahydrofuran (3:1 by volume) as the volatile extraction solvent. Graft copolymer and CPE in the graft-type MCS would remain after the extraction. Graft efficiency (GE) was calculated by:

$$\text{graft efficiency(GE)} = \left(1 - \frac{W_0 - W_1}{M}\right) \times 100\%$$

where W_0 is the total weight of graft-type MCS resin, W_1 is the residual weight of graft copolymer, and M is the weight of monomers.

At the same time, PCMS copolymer and CPE were extracted in the same way.

Equipment and Characterization Procedures

About 0.2% lubricant (BS, wax) and 5% stabilizer (TLS, DLP) of CPE were added to the graft-type MCS resin. All components were first mixed in a mixer and then milled on a laboratory two-roll mill at a temperature of 180°C for 10 min. Five millimeter thick plaques were pressed at a temperature of 170–190°C. After cooling in the press, the plaques were tempered for 10 h at 60°C. The rheological behavior of graft-type MCS resin was investigated at a fixed temperature of 180°C and different pressures by a Shimadzu koka flow tester (Toynaka, Japan) [9]. The diameter and length of nozzle was 1 mm and 10 mm, respectively. The weight of specimen was 1.3 g.

The $\overline{\mathrm{M}}_\mathrm{n}$ and $\overline{\mathrm{M}}_\mathrm{w}$ of the ungrafted copolymer were determined using a P200 gel permeation chromatogragh (GPC). Tetrahydrofuran was used as the flowing phase and the flow rate was 1.00 mL/min. The column was calibrated by using commercially available narrow-distribution polystyrene. The $\overline{\mathrm{M}}_\mathrm{n}$ and $\overline{\mathrm{M}}_\mathrm{w}$ were calculated by

$$\overline{\mathrm{M}}_\mathrm{n} = \frac{\sum H_i M_i}{\sum H_i} \qquad \overline{\mathrm{M}}_\mathrm{w} = \frac{\sum H_i}{\sum H_i / M_i}$$

in which H is the peak height of the GPC spectrum.

The Fourier transform infrared (FTIR) analysis was conducted on an FTS-40 IR spectrophotometer (BIO-RAD Co., USA).

Thermogravimetric analysis (TGA) was carried out on a Shimadzu DT-40 thermal analyzer. The initial thermal degradation temperature ($\mathrm{T_i}$) and the half weight loss temperature ($\mathrm{T_{50}}$) value were determined at a heating rate of 10°C/min in an air atmosphere. The glass transition temperature ($\mathrm{T_g}$) was measured using a differential scanning calorimeter at a heating rate of 20°C/min from -80°C to 175°C under a nitrogen flow of 40 mL/min. The Vicat softening point ($\mathrm{T_{vicat}}$) were measured by using Vicat softening point tester at a heating rate of 2°C/min. The temperature at which the needle at the load of 1 kg pushed into the sample 1 mm was taken as the $\mathrm{T_{vicat}}$.

The tensile tests were performed using an electronic tensile tester. They were conducted at an extension rate of $12.0\,\mathrm{mm}\cdot\mathrm{min}^{-1}$ at 25°C and 50% relative humidity. The notch impact tests were determined on an XCJ-40 impact tester. The fractured surfaces of the tensile specimens were observed by scanning electron microscopy (SEM) using a method in the literature [10].

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RESULTS AND DISCUSSION

Graft Efficiency

When graft-type MCS, CPE, and PCMS were extracted by acetone and tetrahydrofuran mixture for 48 h, the weight of graft-type MCS resin decreased, the pure PCMS was all dissolved and the weight of CPE remained unchanged. This shows that ungrafted polymers can be resolved by acetone and tetrahydrofuran mixture but the polymers grafted onto CPE substrate would remain unaffected.

When the monomer ratio was kept unchanged (MMA/St/ChMI = 5/7/3, by weight), the graft efficiency was estimated at different concentrations of BPO. When the BPO concentration was 0.1%, 0.3%, 0.5%, 0.7% of the monomer feed content, the graft efficiency was 23.7%, 30.5%, 30.2%, 24.8%, respectively. The graft efficiency has maximum value when the concentration of BPO is 0.3%. This is due to the increase of free radical with increasing BPO concentration, which will lead to an increase of graft efficiency on the one hand, but will also cause the increase of coupling termination of radical group and decrease the graft efficiency on the other hand. The concentration of initiators in the following experiment was all 0.3% of monomer feed content.

When MMA and CPE were all fixed at 25% (by wt), the content of ChMI in graft-type MCS changed from 0, 5, 10, 15, 20, to 25% (by wt), and other conditions kept unchanged, graft efficiency of using AIBN and BPO as initiator was estimated. The results are shown in Figure 1. The monomer reactivity ratios are summarized in Table 1. It was found that ChMI copolymerized alternatingly with electron-donating styrene although charge-transfer complex is not formed, so it is a species with a very weak interaction between St and ChMI [11]. St and MMA also have the tendency of copolymerizing alternatingly, and the activity of MMA is greater than that of ChMI in the MMA-ChMI system and radicals preferentially add to MMA instead of ChMI just as the reactivity ratios show [12]. There is weak interaction between ChMI and CPE, so the charging of ChMI make it easy for monomers to graft onto CPE substrates. The reactivity ratios and interactions among reactants can explain the fact that GE decreases first because of the feeding of ChMI and then increases with the increasing of ChMI content. ChMI and MMA have the tendency of copolymerizing alternatingly and the activity of MMA is greater than that of ChMI, so that a homopolymer of MMA existed in the resin when the content of ChMI was high. The graft efficiency of the resin initiated by BPO was higher than that initiated by AIBN under the

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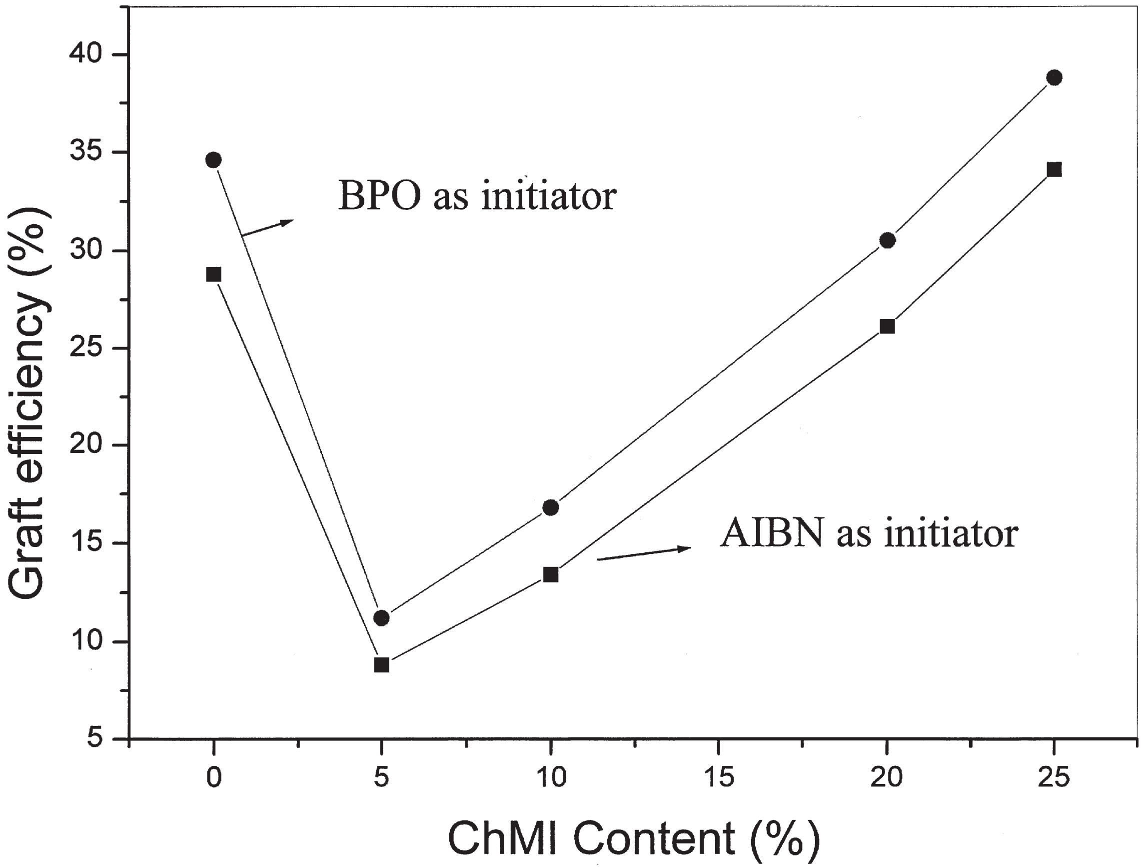

FIGURE 1 Graft efficiency of graft-type MCS resin using AIBN and BPO as initiator.

TABLE 1 Reactivity Ratios of Monomers

M_1	M_2	r_1	r_2
St	MMA	0.52	0.46 [14]
St	ChMI	0.11	0.02 [11]
MMA	ChMI	1.4	0.24 [11]

same conditions. This may be because peroxide initiator has stronger ability of abstracting hydrogen from CPE substrate than azo initiator. As a result, much more graft points formed, which in turn leads to a higher graft efficiency [13].

A change in the proportion of St and ChMI, did not affect the numbers of graft points. It is assumed that the length of molecular chain of the graft copolymer increases as the graft efficiency increases [14]. For testing this assumption, the molecular weight of ungrafted polymers was determined as shown in Table 2. The $\overline{M}_n$ and $\overline{M}_w$ increased with an increase of ChMI content [15]. Polydispersity decreased because of the feeding of ChMI, but in PMMA homopolymer the molecular weight distribution widens at high ChMI content. The increase of $\overline{M}_n$ and $\overline{M}_w$ results in better compatibility, but the increase of PMMA homopolymer results in contrary effect, so when the content of ChMI was 20% it had good compatibility whereas the compatibility got poor at 25% ChMI content as shown in the DSC figure (Figure 3) and SEM photos (Figure 7).

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FTIR

Figure 2 is the infrared spectrum of extracted graft-type MCS resins and PCMS. It is observed from FTIR spectrum of PCMS (Figure 2D) that the characteristic absorption peak of ester group in MMA is $1725\,cm^{-1}$ [16], the absorption peaks of phenyl group are 1600, 760, and $700\,cm^{-1}$ [16], and the absorption peaks of carbonyl groups in ChMI are 1709 and $1770\,cm^{-1}$ [17]. Figure 2 A, B, and C are the FTIR

TABLE 2 Molecular Weight and Polydispersity

ChMI content (wt %)	0	10	15	20	25
$\overline{M}_w$ ($\times 10^{-5}$)	2.24	2.61	3.18	3.9	4.9
$\overline{M}_n$ ($\times 10^{-5}$)	0.98	1.31	1.65	2.11	2.13
$\overline{M}_w/\overline{M}_n$	2.7	2.35	2.28	2.18	2.3

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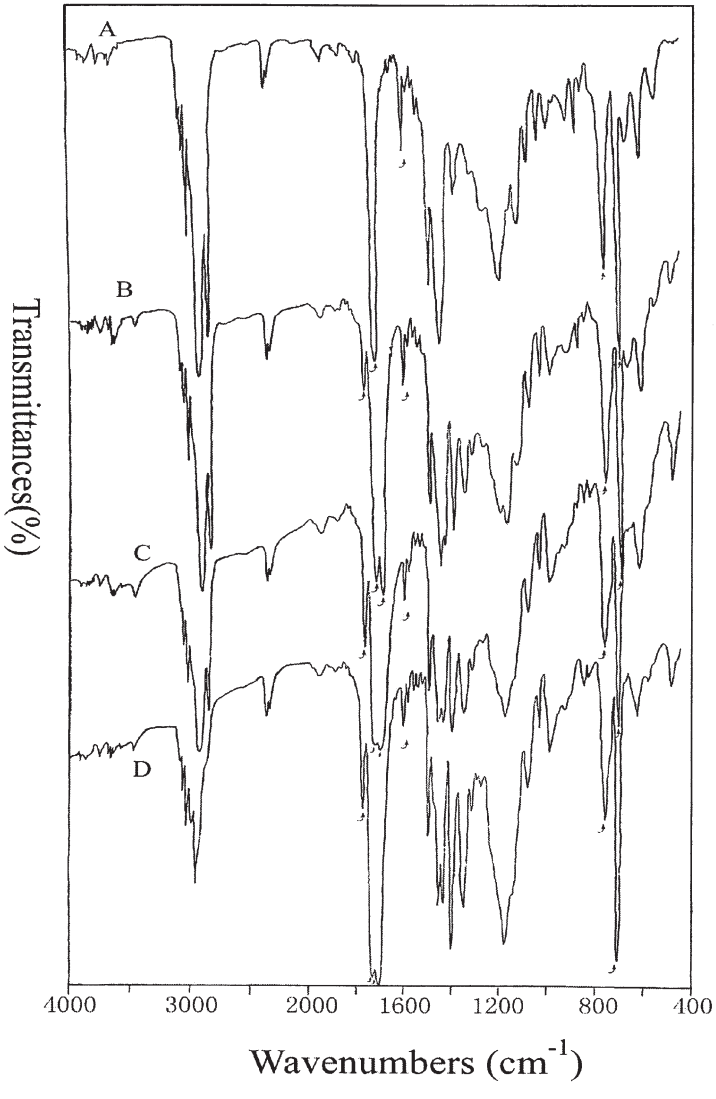

FIGURE 2 Infrared spectra of extracted graft-type MCS resins and PCMS.

spectra of MCS resins with ChMI content of 0%, 15%, 25% (by weight), respectively. The abosorption peaks of carbonyl group at 1709 and 1770 cm^{-1} increased with increasing ChMI content, whereas the absorption peak of phenyl group at 1600, 760, and 700 cm^{-1} decreased because of the decrease of St content, compared with the absorption peak of easter group in PMMA at 1725 cm^{-1}. When no ChMI is fed,

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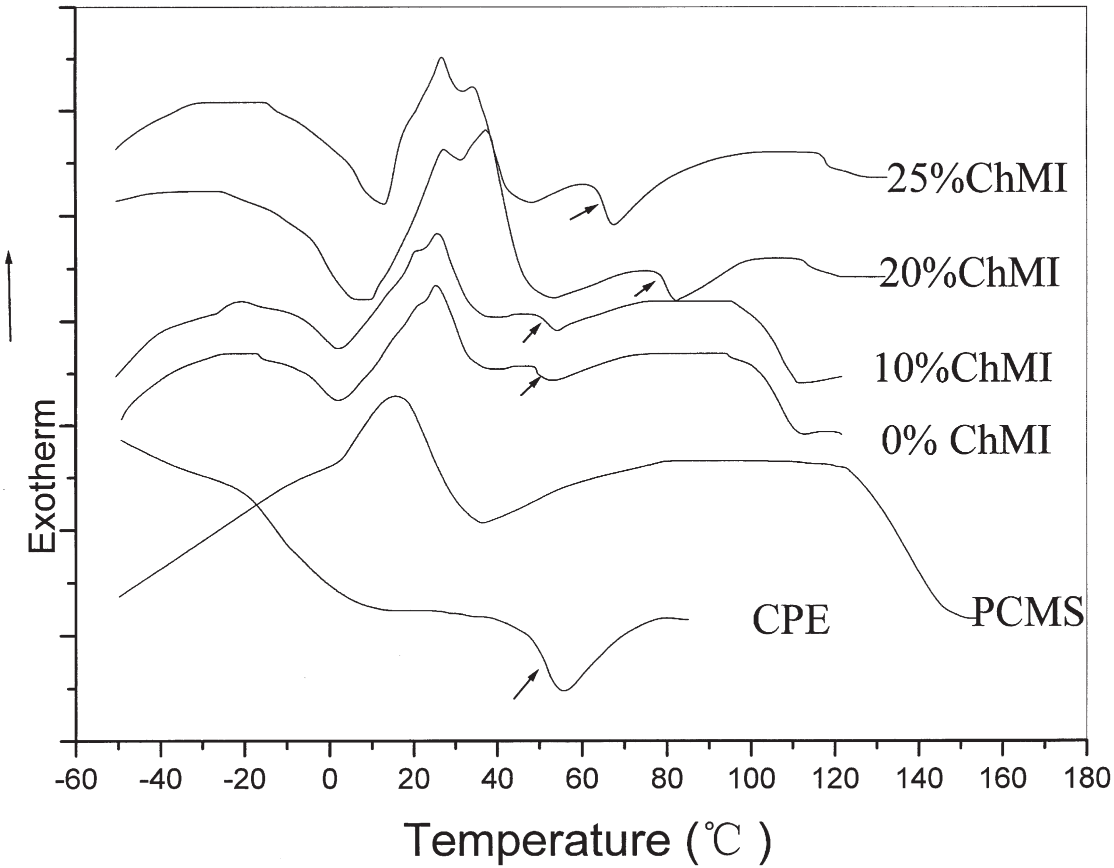

FIGURE 3 T_g of graft-type MCS resins and PCMS.

there is no absorption at 1709 and 1770 cm^{-1} in the FTIR spectrum. The result shows that the three monomers can graft onto CPE substrate successfully, and the monomer content that graft onto CPE increased with increasing the monomer feed content.

Glass Transition Temperature and Vicat Softening Point

As seen in Figure 3, the glass transition temperature of high-chlorinated segments of CPE (noted with arrows) has an evident shift to higher temperature when the content of ChMI content was 20%, and then has a drop when the content of ChMI was 25%. This indicates that the resin has better compatibility at 20% ChMI content, but the compatibility got poor at 25% ChMI content because of the formation of PMMA homopolymer. There are exothermic peaks at 0–40°C among the DSC curves of PCMS and graft polymers, which *may* be caused by the release of intermolecular interaction energy. The glass transition of MMA, St, and ChMI copolymer (T_g') in graft-type MCS resin is over 90°C, and the glass transition temperature increases and gets less distinctive with increasing ChMI content. When the content of ChMI is 25%, the T_g' and the T_{Vicat} (as shown in Figure 4) of the resin increase 16°C and 35°C, respectively, compared with the resin whose content of ChMI is 0%. The T_g' and the T_{vicat} increase slightly when the ChMI content is low, but when ChMI content is over 10% they increase substantially. This indicates that when the ChMI content is over 10%, the compatibility of MCS resin increases leading to the increase of interaction of molecular chains, so the T_g' and the T_{Vicat} increase [18]. The incorporation of rigid polar ChMI units in the copolymer increases the molecular chains' rigidity and molecular interactions, so the glass transition temperature and the T_{Vicat} of the copolymer increase.

Thermogravimetric Analysis

The thermal stability of the copolymers is arbitrarily defined as a function of the initial thermal degradation temperature (T_i) and the half weight loss temperature (T_{50}) value. The TGA curves are shown in Figure 5. It can be observed from the TGA curves that when the content of ChMI is 25%, T_i and T_{50} increased 11°C and 8°C, respectively, compared with the general graft-type MCS resin. The residual mass of the plateau of the N-cyclohexylmaleimide-containing graft-type MCS resin is more than the general graft-type MCS resin. Obviously, it is the incorporation of ChMI units in the copolymer chain that improves the material's thermal stability [19].

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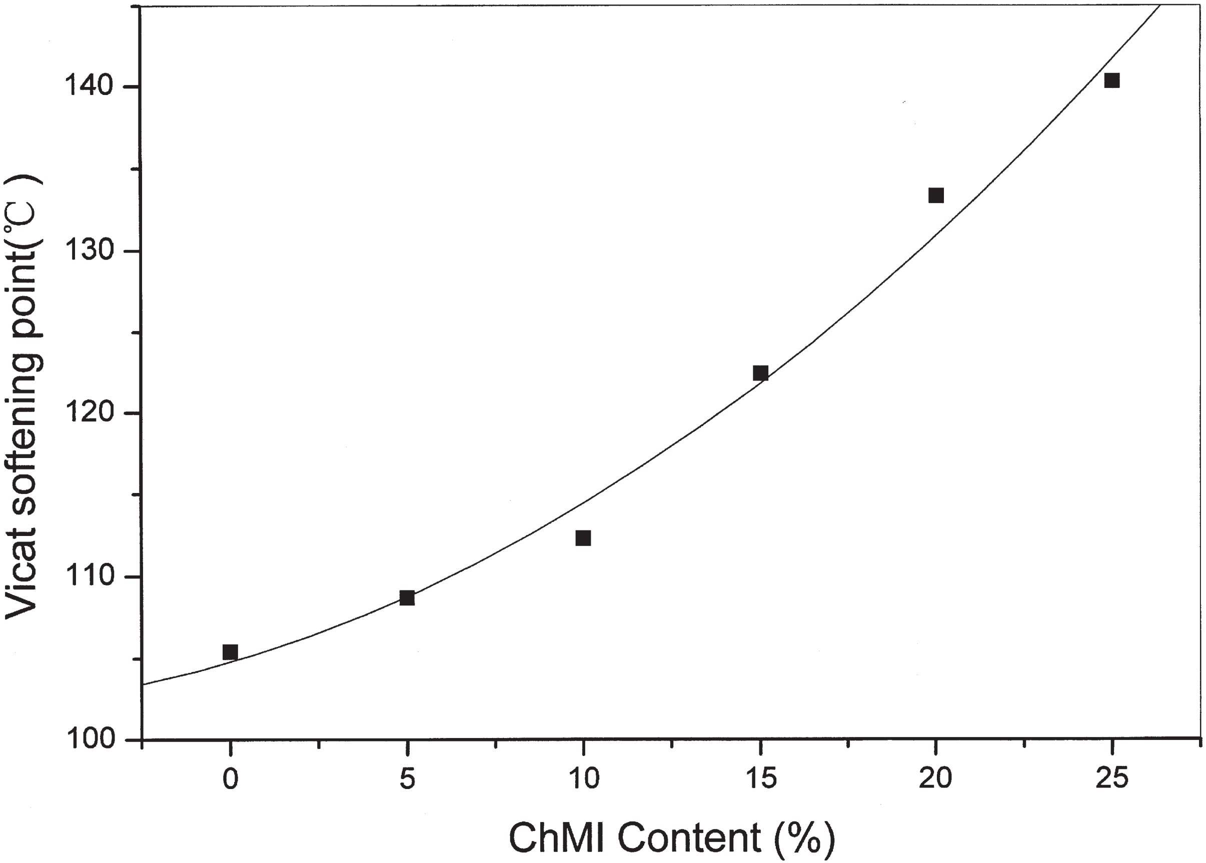

FIGURE 4 T_{Vicat} of graft-type MCS resins.

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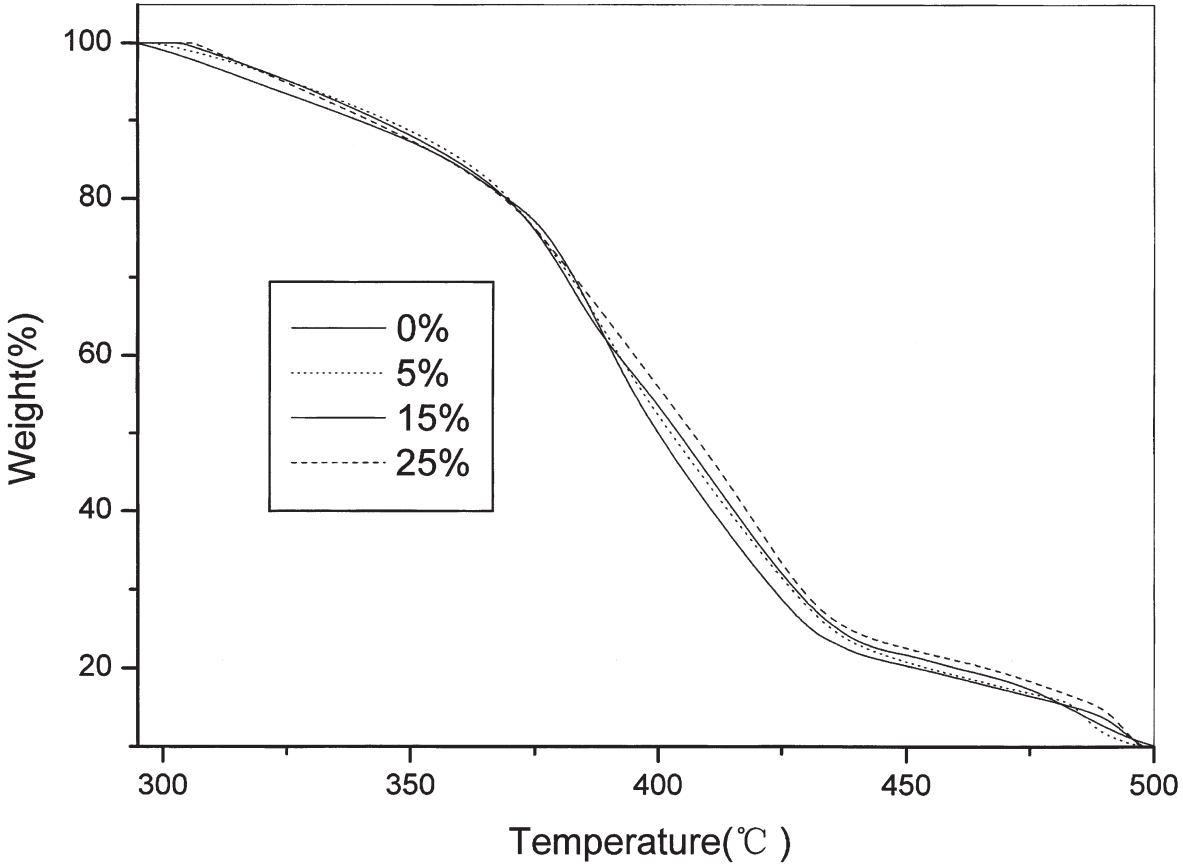

FIGURE 5 TGA curves of graft-type MCS resins.

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Rheological Behavior of Graft MCS Resin

The experimental temperature was fixed at 180℃, with experimental stresses of 140, 160, 180, 200, 220 kg/cm^2, respectively.

According to the Ostwald-De Waele power-law equation [20]:

$$\tau = k\dot{\gamma}^n \tag{1}$$

The non-Newtonian flow parameter n can be obtained from the plot of ln τ_w versus ln $\dot{\gamma}_w$, as seen in Table 3.

According to Reference [21], $\dot{\gamma}_w$, is improved for non-Newtonian in the following equation:

$$\dot{\gamma}'_w = \frac{3n+1}{4n}\dot{\gamma}_w \tag{2}$$

The apparent viscosity is given by:

$$\eta_a = \frac{\tau_w}{\dot{\gamma}'_w} \times 9.8 \times 10^5 \,(\text{Poise}) \tag{3}$$

The rheological behavior of graft-type MCS resin is estimated, and the plots of ln η_a vs. ln $\dot{\gamma}'_w$ are given in Figure 6. As seen from Figure 6, the apparent viscosity η_a decreases with increasing shear rate. It shows that the molten graft-type MCS resin is a pseudo-plastic liquid. ChMI is a stiff polar molecule, thus the interaction between molecules and molecular chain stiffness increases because of the feeding of ChMI, resulting in the increase of the apparent viscosity of the resin. Molecular weight and polydispersity have some effect on the apparent viscosity, and the increase of molecular weight is liable to increase the apparent viscosity of the resin, but the increase of polydispersity has the contrary effect. So the apparent viscosity of the resin increased first but when the content of ChMI was over 20%, the viscosity almost did not change. The non-Newtonian flow parameter power n of N-cyclohexylmaleimide-containing graft-type MCS resin is higher than that of general graft-type MCS resins. This can be attributed to the fact that the rigid maleimide structure increases the stiffness of macromolecular chains.

TABLE 3 Relationship Between n and ChMI Content

ChMI% (in wt.)	0	10	15	20	30
n	0.35	0.37	0.40	0.42	0.45

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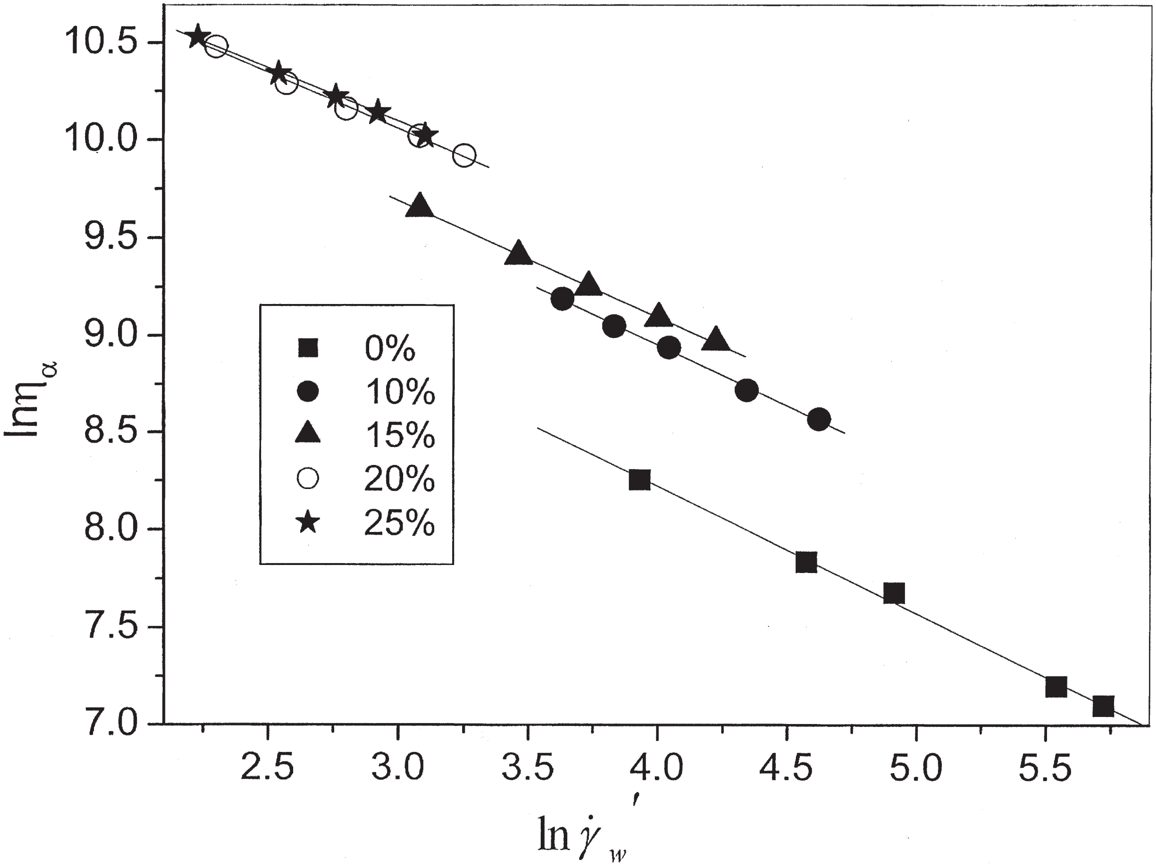

FIGURE 6 Rheological behavior of graft-type MCS resins.

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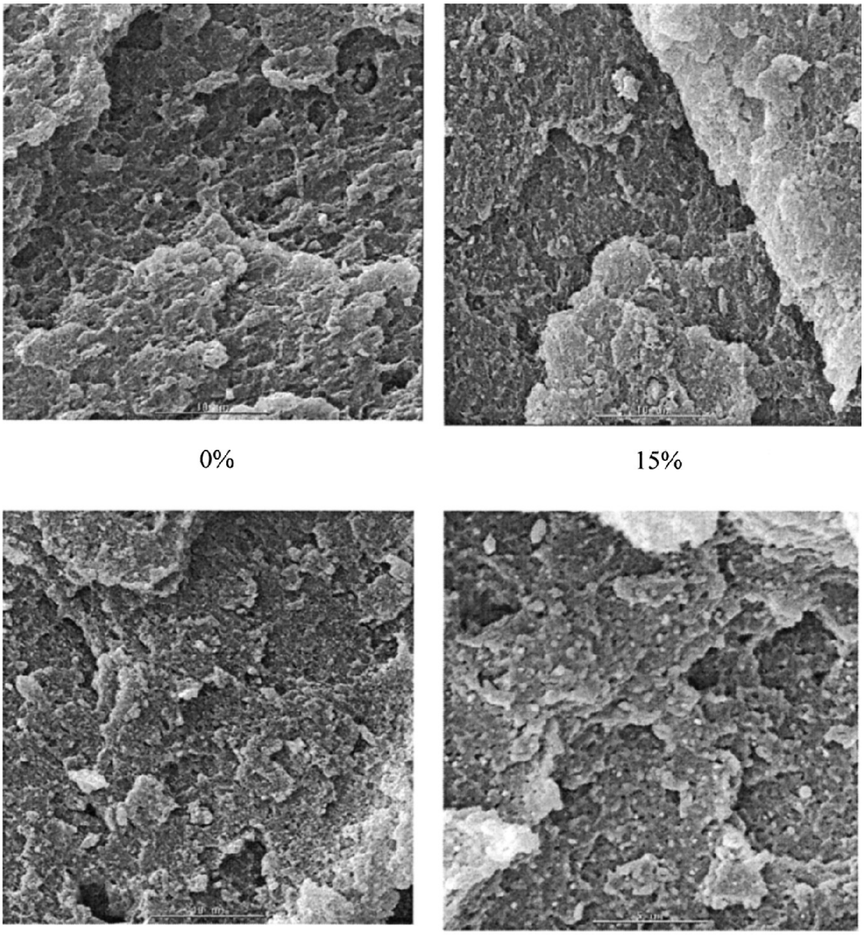
Mechanical Properties and Morphology

Figure 7 is the SEM photographs of tensile fractured surface for general graft-type MCS resin and N-cyclohexylmaleimide-containing graft-type MCS resin. As seen in Figure 7, the fracture surface of the general graft-type MCS resin exhibits somewhat tough break, but the fracture surfaces turn to brittle breaks and have some holes appear along with the increase in ChMI content. The compatibility



0%

15%

20%

25%

FIGURE 7 SEM photographs of the tensile fractured surface of graft-type MCS resins.

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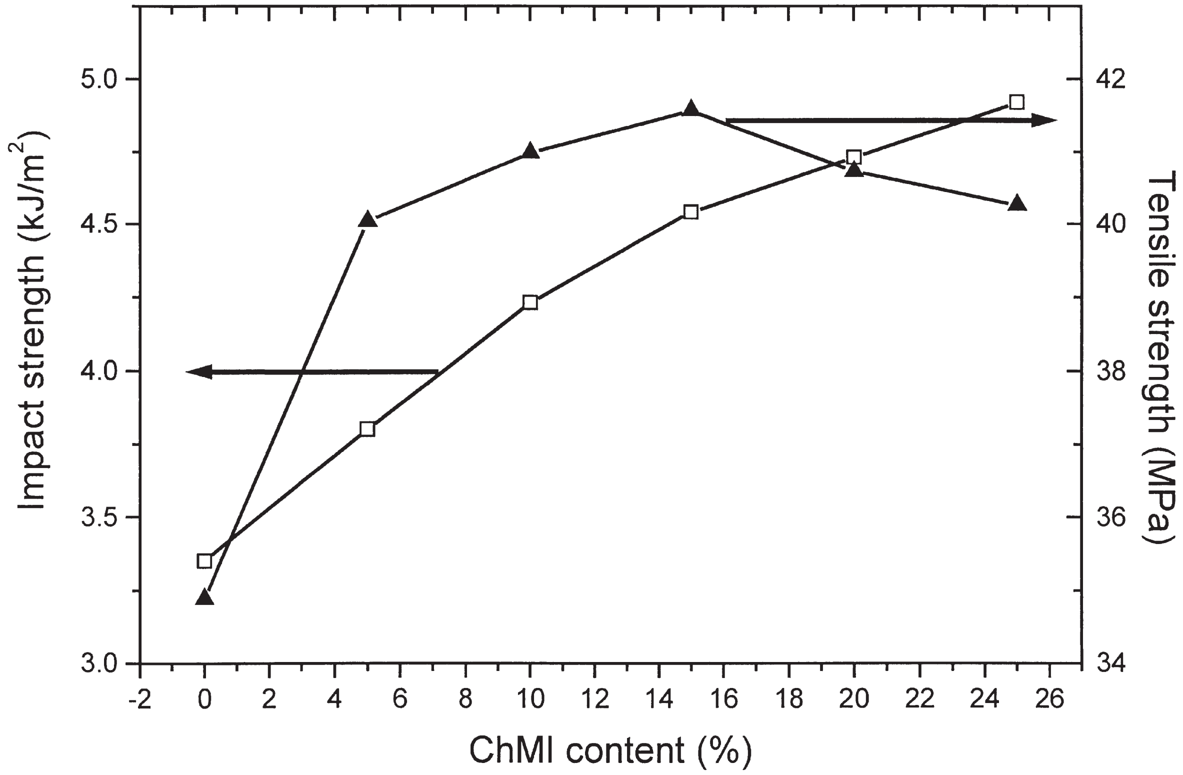

FIGURE 8 Mechanical properties of graft-type MCS resins.

of MCS resin got better along with the increase of ChMI content. Phase interface is blurred when the content of ChMI is 20%, which indicates the resin has better compatibility at this ChMI content.

The mechanical properties of graft-type MCS resin were estimated. The relationship between mechanical properties and the content of ChMI is shown in Figure 8. In general, the increase of impact strength will lead to the decrease of tensile strength, however, the tensile strength and the impact strength of MCS resin modified by ChMI all have increased with an increase of ChMI content. One reason for the increase of mechanical strength is the better compatibility caused by the feeding of ChMI. The holes seen in Figure 7 may be ChMI-containing copolymers, which can toughen the resin as brittle particles and cause the increase of impact strength. The incorporation of polar maleimide units in the backbones increases molecular interactions, so the tensile strength increases. But when the content of ChMI is over 15%, the entanglement between molecular chains decreases due to the increase of the rigid planar ring, so the tensile strength decreases.

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CONCLUSIONS

1. BPO as initiator had higher GE than AIBN, and GE had a maximum value when BPO concentration was 0.3% of monomer feed content.
2. Graft efficiency of graft-type MCS resin is dependent on the content of ChMI. Graft efficiency increased with increasing content of ChMI.
3. Molecular weight increased due to the feeding of ChMI, and polydispersity has minimum value at 20% ChMI content.
4. The glass transition temperature, and the T_{Vicat} of the copolymer increase because of the feeding of ChMI.
5. T_i and T_{50} increased 11°C and 8°C when the content of ChMI was 25%, and the residual mass of the plateau was higher than the general MCS resin.
6. The feeding of ChMI in graft-type MCS caused an increase of impact strength and tensile strength. But the tensile strength peaked when the content of ChMI was 15% (by weight).
7. The apparent viscosity and the non-newtonian flow parameter power *n* increased because of the introduction of ChMI.

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