

# Preparation and Characterization of Nitrile Butadiene Rubber (NBR)/Polyoxymethylene (POM) Blends Compatibilized by Maleic Anhydride Grafted POM (MAH-g-POM)

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**Abstract:** Maleic anhydride grafted POM (MAH-g-POM) was prepared and used as the compatibilizer for nitrile butadiene rubber (NBR) modified by acetal copolymer (POM) in different proportions. It was found that MAH-g-POM had good compatibilization effect for the blends. Both DSC and XRD results indicated that the process of blending POM with NBR considerably influenced the crystallization of POM. The scanning electron microscopy (SEM) study of tensile fracture surfaces of the blends clearly showed that the compatibility of NBR/POM blends was enhanced with increasing POM content. Mechanical properties indicated that NBR/MAH-g-POM had relatively higher elongation at break but lower tensile strength than NBR/POM blend. The present work will supply a good insight into the formula design and further optimization of polymer composites or blends.

**Keywords:** Nitrile butadiene rubber (NBR), Acetal copolymer (POM), maleic anhydride grafted POM (MAH-g-POM), Mechanical properties, Compatibility.

## 1. INTRODUCTION

Nitrile butadiene rubber (NBR), which has excellent oil resistance, aging resistance and mechanical properties, is widely employed in the manufacture of oil resistance products, a variety of oil resistant gaskets, casting and so forth. As is known, the performance of NBR will be considerably influenced by the content of acrylonitrile (AN). With increasing AN content, improved oil resistance, heat resistance and mechanical properties can be achieved [1-5]. On the other hand, the relatively poor performance (such as, resistance to low temperature, ozone resistance, compression strength, etc.) has limited the further application of NBR to an extent. Generally speaking, NBR can be modified by other materials by preparing composites or blends with desirable physical and mechanical properties.

In recent years, a great deal of effort has been made to modify NBR with other polymers, especially the thermoplastics, for example, PVC [6-8], PP [9-11], PS [12-13], etc. Huang *et al.* [7] reported that NBR/PVC composite with 30 wt. % PVC showed the

best synthetic mechanical and tribological properties, and the lubricating effect of PVC played an important role in decreasing the friction coefficient. Ismail *et al.* [11] observed that the vulcanized PP/NBR blend had perfect tensile properties and oil resistance, and it was able to withstand serious natural weathering conditions. There were some other researchers, who blended NBR with engineering plastics (e.g., polyamide [14]) or natural substances (e.g., starch [15]). Albano *et al.* [15] argued that the thermal stability of NBR was considerably increased with the addition of coconut flour. Since these systems are incompatible ones, it is crucial to find a suitable compatibilizer to modify NBR. Recently, the use of maleic anhydride (MAH) grafted PP or PVC [16, 17], or adding a third component (e.g., epoxy resin [6, 9]), have been employed as the compatibilizer for these systems. Ismail *et al.* [16] found that the presence of MAH increased the interfacial interactions between PVC and NBR phases, which significantly improved the mechanical properties of the blends.

Polyoxymethylene (POM) is presently one of the most commonly used engineering plastics with high crystallinity and high melting point, primarily due to its relatively well-defined molecular chain structures. So far, there is limited literature available on the modification of NBR with POM. Jiang *et al.* [18] reported that the hardness of NBR/POM blends

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increased and the elongation at break reduced with increasing POM content. Wang *et al.* [19, 20] found that phenolic resin can play a role as a good compatibilizer for NBR/POM blend.

In this work, NBR/POM blends were prepared using a BANBURYING mixer, with maleic anhydride grafted POM (MAH-g-POM) prepared and used as compatibilizers. Our aim is to investigate the compatibilization effect of MAH-g-POM on the compatibility, thermal behavior and mechanical properties of NBR/POM blends so as to optimize the formula design of NBR/POM blends. The current study will be instructive to an improved understanding of the relationship among “processing-structure-property” of polymer composites or blends.

## 2. EXPERIMENTAL PART

### 2.1. Materials

Nitrile butadiene rubber (NBR) used in this study was NBR-2865 (acrylonitrile content 28 %, Mooney viscosity [ML 1+4] at 100°C: 65), which was kindly supplied by the *Nantex Co., Taiwan*. Acetal copolymer (POM), Grade: M90-44, was manufactured by the *Dupont Inc., U.S.A.*, with a density of 1.41 g/cm<sup>3</sup>. Maleic anhydride (MAH), was provided by the *Tianjin Guangfu Fine Chemical Research Institute, China*. Dicumyl peroxide (DCP) was obtained from the *Sinopharm Chemical Reagent Co., China*. Besides, sulfur, stearic acid, zinc oxide and N-cyclohexane-2-benzothiazole sulfonamide (CZ) were all obtained from the *Ambang Chemical Co., China*.

### 2.2. Samples Preparation

#### 2.2.1. Preparation of Maleic Anhydride Grafted POM (MAH-g-POM)

Maleic anhydride grafted polyoxymethylene (MAH-g-POM) at a ratio of 100/3 (in weight percent) with 0.3

wt.% DCP was prepared through melt extrusion using the *HAAKE PolyLab OS of Thermo Scientific Co., Germany*. The maximum processing temperature is 170 °C, at a screw speed of 15 rpm.

#### 2.2.2. Preparation of NBR/POM Blends

The formulae of the blends are detailed in Table 1. NBR/POM blends were initially prepared by charging POM into the mixing chamber in a *HAAKE* rheometer, and followed by the addition of NBR with MAH-g-POM or MAH at 170 °C at 40 rpm. The mixing time was 15 min. The blends were removed from the mixer, and once again blended with vulcanization additives at 40 °C for 10 min, then sheeted on a two-roll mill. Later on, the samples vulcanized on a vulcanizing machine (Model: QLB-350, *Shanghai Rubber Machinery Co., China*), at 170 °C under 10 MPa for 10 min.

### 2.3. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR characterization was carried out on a spectrometer Model: VERTEX 80+ HYPERION 2000, *Brucker Optics, Co., U.S.A.* The spectrum resolution was 0.1 cm<sup>-1</sup> and the scanning range was from 500 to 4000 cm<sup>-1</sup>. All samples for FTIR measurement were run through the use of attenuated total reflectance (ATR) technique.

### 2.4. X-Ray Diffraction (XRD) Characterization

XRD characterization was conducted at the ambient temperature on a Rigaku Dmax III diffractometer using a Cu (*K<sub>α</sub>*) radiation ( $\lambda=1.54 \text{ \AA}$ ), with an electron accelerating voltage of 36 kV and a scanning range of 5°~80° at a speed of 4 ° per min.

### 2.5. Differential Scanning Calorimetry (DSC) Characterization

The melting and crystallization behaviors were characterized on a differential scanning calorimeter

**Table 1: Formulae of NBR/POM Samples Used in this Study (Unit: wt.%)**

Samples	NBR	POM	MAH-g-POM	MAH	S <sup>a</sup>	Zinc Oxide <sup>a</sup>	Stearic acid <sup>a</sup>	CZ <sup>a</sup>	DCP <sup>a</sup>
A	100	0	0	0	1.5	5	1	1	1
B	80	20	0	0	1.5	5	1	1	1
C	80	0	20	0	1.5	5	1	1	1
D	80	20	0	3	1.5	5	1	1	1
E	90	10	3	0	1.5	5	1	1	1
F	80	20	3	0	1.5	5	1	1	1
G	70	30	3	0	1.5	5	1	1	1

<sup>a</sup>Vulcanization additives used for NBR.

(Model: TA Q-2000, TA Instruments Inc., U.S.A.). Under the nitrogen ( $N_2$ ) atmosphere, each sample was initially heated at 20 °C/min, then kept at 250 °C for 5 min, and subsequently cooled at 20 °C/min. The measuring temperature ranged from -80 to 250 °C.

## 2.6. Scanning Electron Microscopy (SEM) Study

The morphological features of the tensile-fractured surfaces of the samples were examined and analyzed using a scanning electron microscope (SEM), Model: S-4800, Hitachi Inc., Japan with electron accelerating voltages of 1 kV or 5 kV (dependent on magnification). The fractured ends of the specimens were cut into 3.0 mm and mounted on aluminum stub.

## 2.7. Mechanical Property Testing

Tensile properties were examined with a computer-controlled tensile instrument (Model: UTM-2000, SANS Co. Ltd., China). All measurements were carried out according to the GB/T 528-1998. Dumb-bell specimens with a thickness of 2 mm were cut from the compression-molded sheets. At least five specimens were tested in each case for better repeatability of the measured data.

## 3. RESULTS AND DISCUSSION

### 3.1. Characterization of POM, MAH-g-POM and NBR/POM Blends

Figure 1 shows the FTIR spectra of POM and MAH-g-POM. MAH-g-POM was purified by soxhlet extraction, using acetone as an extractant. Then, it was dried for the removal of acetone. It can be seen that both curves display the C-H stretching vibration around 3000  $cm^{-1}$ , the C-O-C stretching vibration around 1100  $cm^{-1}$ , as well as the C-H out-of-plane bending vibration around 900  $cm^{-1}$ . In the spectrum of MAH-g-POM, the absorption of carbonyl (C=O) at 1734  $cm^{-1}$  indicated the presence of anhydride groups from MAH. However, the absence of C-C double bond at 1600  $cm^{-1}$  of MAH structure might be associated with the C-C radical of MAH grafted with POM. The infrared absorption peak area ratio of characteristic groups of the graft with a specific internal standard was adopted to characterize the rate of the graft [21]. In this study, we used the stretching vibration absorption peak of carbonyl from MAH as graft peak, and the stretching vibration absorption peak of C-O-C from POM as internal standard. According to  $R=A_{1734}/A_{1100}$  [21], the degree of grafting ( $R$ ) was 0.8 % in the present work.

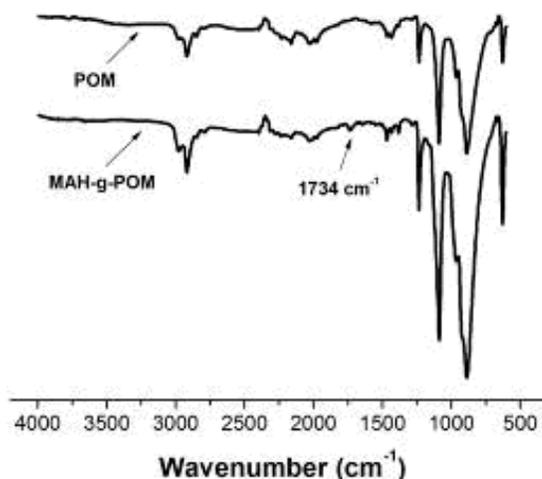


Figure 1: FTIR spectra of POM and MAH-g-POM.

The XRD results were used to analyze the crystal structure of POM and MAH-g-POM. The unit cell of crystalline POM is hexagonal [22], and the diffraction peaks from the (100), (105) and (110) planes were observed at 23.1°, 34.6° and 48.2°, respectively. Figure 2 shows that the three diffraction peaks were observable for POM and MAH-g-POM. By comparison of results, the use of similar results [23] indicating that plane distance of crystalline POM almost remained constant, namely, the crystalline lattices of POM did not undergo an obvious change. In addition, the intensities of the diffraction peaks of POM decreased after grafted with MAH, which suggested that the degree of crystallinity and crystallite size were both altered after grafting reaction. Various researchers [24, 25] have studied the relation between crystallize and the intensity of the sharp diffraction peaks. The relative intensity of the sharp diffraction peaks of MAH-g-POM was smaller than neat POM, indicating that the degree of crystallinity of MAH-g-POM was reduced, which is similar with the reported before.

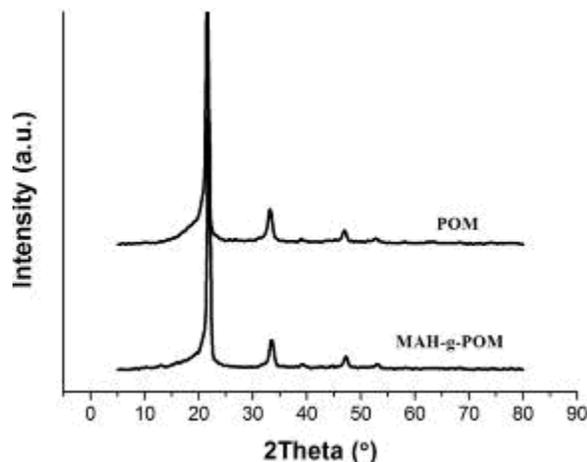


Figure 2: XRD spectra of POM and MAH-g-POM.

Figure 3 suggests that the (100) diffraction peak of POM had the highest intensity. However, it was greatly weakened when POM and MAH-g-POM were blended with NBR. These all showed that the crystal structure of POM was changed after melt blending; while there was no significant difference between NBR/MAH-g-POM and NBR/POM blends.

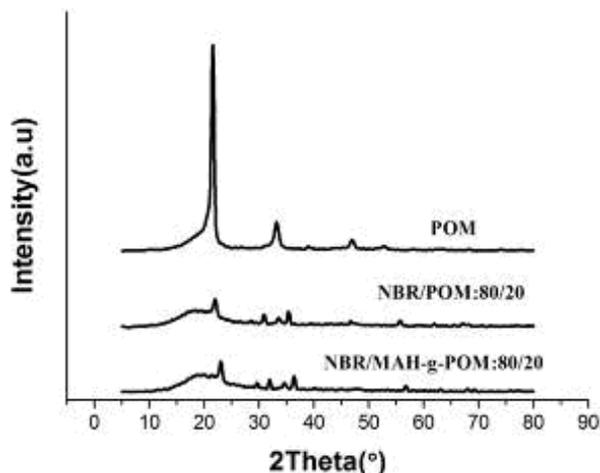


Figure 3: XRD spectra of POM, NBR/POM and NBR/MAH-g-POM.

The thermal behaviors of POM and MAH-g-POM were characterized by DSC. The DSC diagrams of POM and MAH-g-POM are presented in Figure 4. The melting point ( $T_m$ ), crystallization temperature ( $T_c$ ) and the crystallization enthalpy ( $\Delta H_c$ ) are listed in Table 2. The degree of crystallinity was evaluated by the following equation [26]:

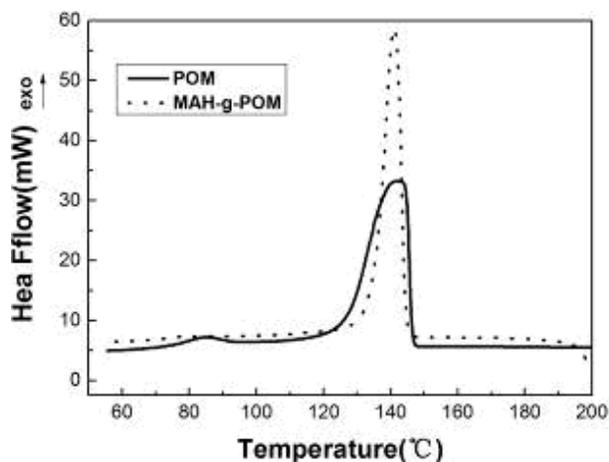


Figure 4: DSC spectra of POM and MAH-g-POM.

$$X_c = \frac{\Delta H}{\Delta H_0} \times 100\% \quad (\Delta H_0 = 248 \text{ J/g})$$

It was found that when POM was grafted with MAH,  $\Delta H_c$  decreased significantly, but  $T_m$  and  $T_c$  decreased

slightly. The degree of crystallinity of neat POM was 56.0 %, and it decreased after grafting with MAH. The reason is mainly owing to the linear molecular structure of POM, which is favorable to easier crystallization and a higher crystallinity. Due to the steric hindrance caused by MAH, both crystallinity and  $T_m$  of MAH-g-POM decreased.

Table 2: Summary of Melting Temperature, Crystallization Temperature and the Crystallization Enthalpy of POM and MAH-g-POM

Materials	$T_m$ (°C)	$T_c$ (°C)	$\Delta H_c$ (J/g)
POM	175.4	142.0	138.9
POM-g-MAH	172.1	141.1	109.8

Figure 5a shows that the glass transition temperature ( $T_g$ ) of NBR slightly increased when it was blended with POM. It can be found that  $T_g$  of NBR almost remains unchanged between NBR/MAH-g-POM/POM and NBR/POM blend, indicating that the effect of MAH-g-POM on the properties of NBR/POM blend is limited, such as, flexibility and processing property. Figure 5b shows that the melting temperature of NBR/MAH-g-POM/POM blend is higher than NBR/POM blend. The total of POM of NBR/POM blend was increased when MAH-g-POM was added, which increased the hybrid entropies of mixing. But the heating rate was too fast, and there is a hysteresis of the melting process. Thus, the melting temperature of POM increased. Figure 5c shows that when POM or MAH-g-POM was blended with NBR, the crystallization peak became not obvious, coupled with a decreased crystallization temperature. This could possibly be due to the influence of the rubber segments on the blending of POM with NBR, which further decreased the ordered stack structure of molecular chains.

### 3.2. Morphological Analysis of Compatibility

Figure 6 shows a comparison of tensile fracture surfaces between NBR/POM and NBR/MAH-g-POM (80/20). Figure 6a shows a coarse and unstable particle structure and an uneven distribution of the dispersed phase of POM, which indicated that the poor interphase adhesion gave rise to poor stress transfer across the interfaces. Figure 6b shows that the fracture surfaces only had several particles on the surface, and the others might be pulled out during the stretching process. The compatibility of NBR/MAH-g-POM was improved as compared to the NBR/POM (80/20) blend,

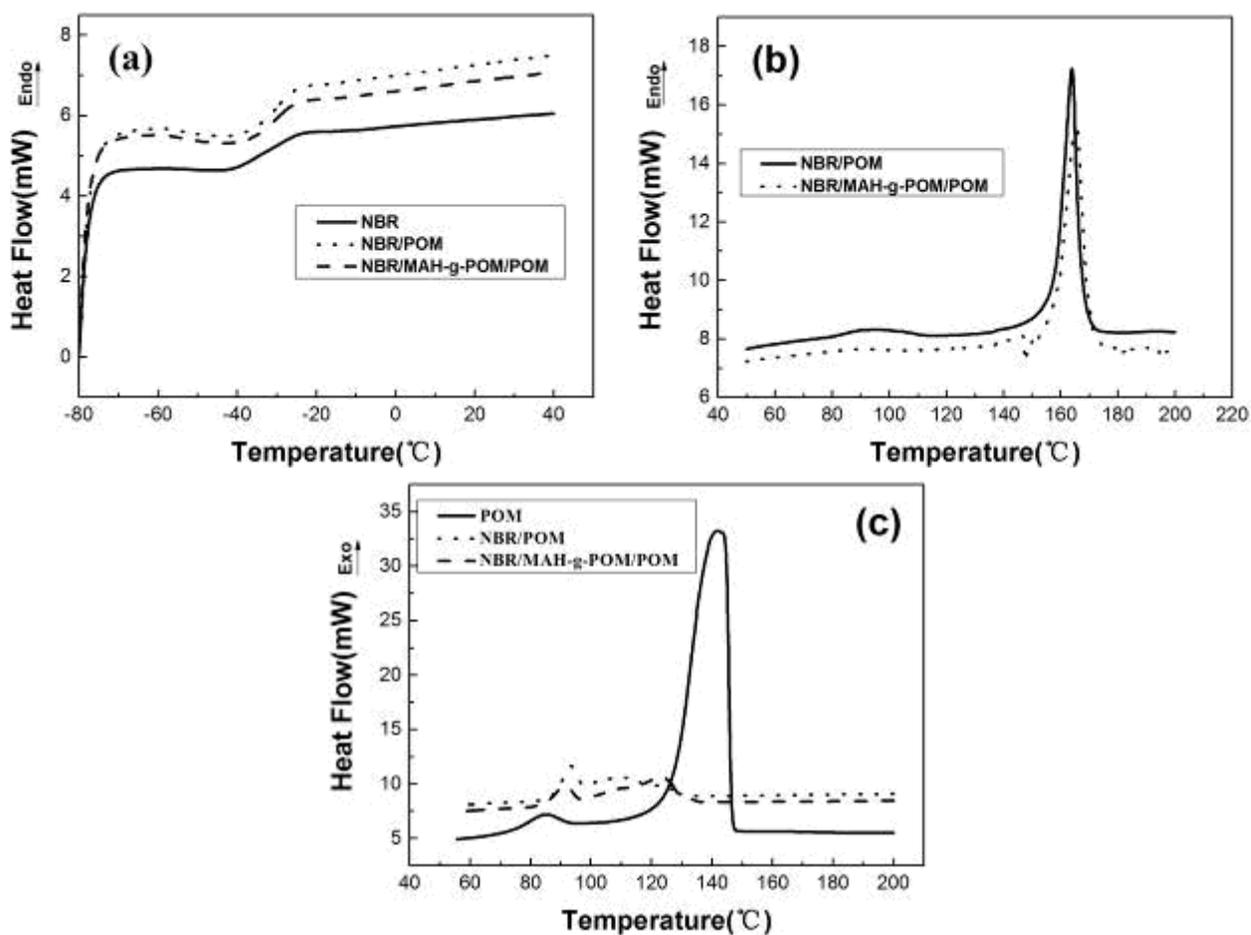


Figure 5: DSC spectra of POM, NBR, NBR/POM (80/20) and NBR/MAH-g-POM/POM (80/3/20).

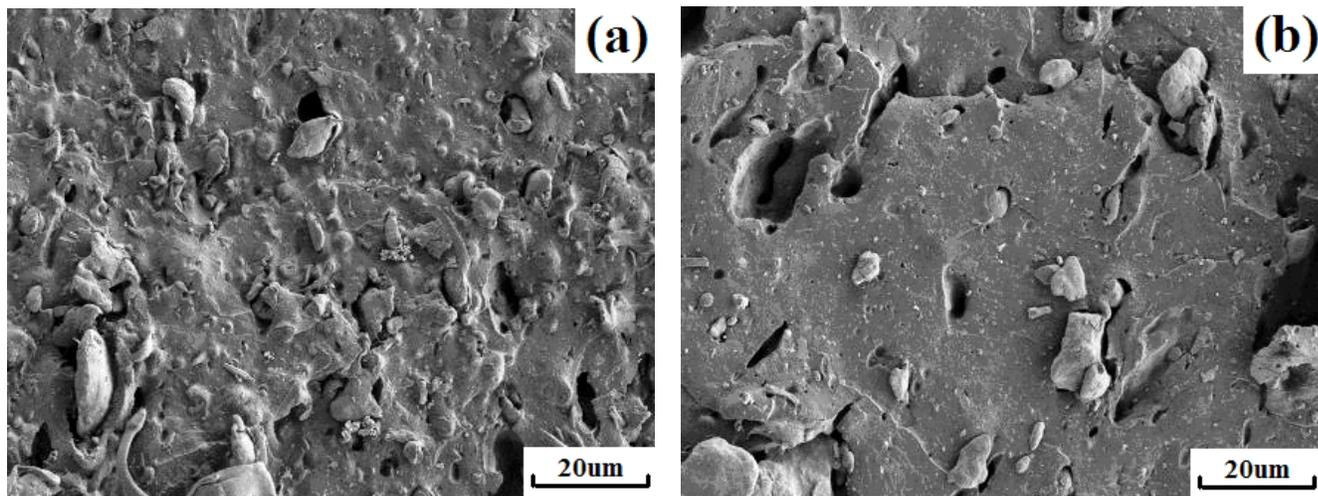
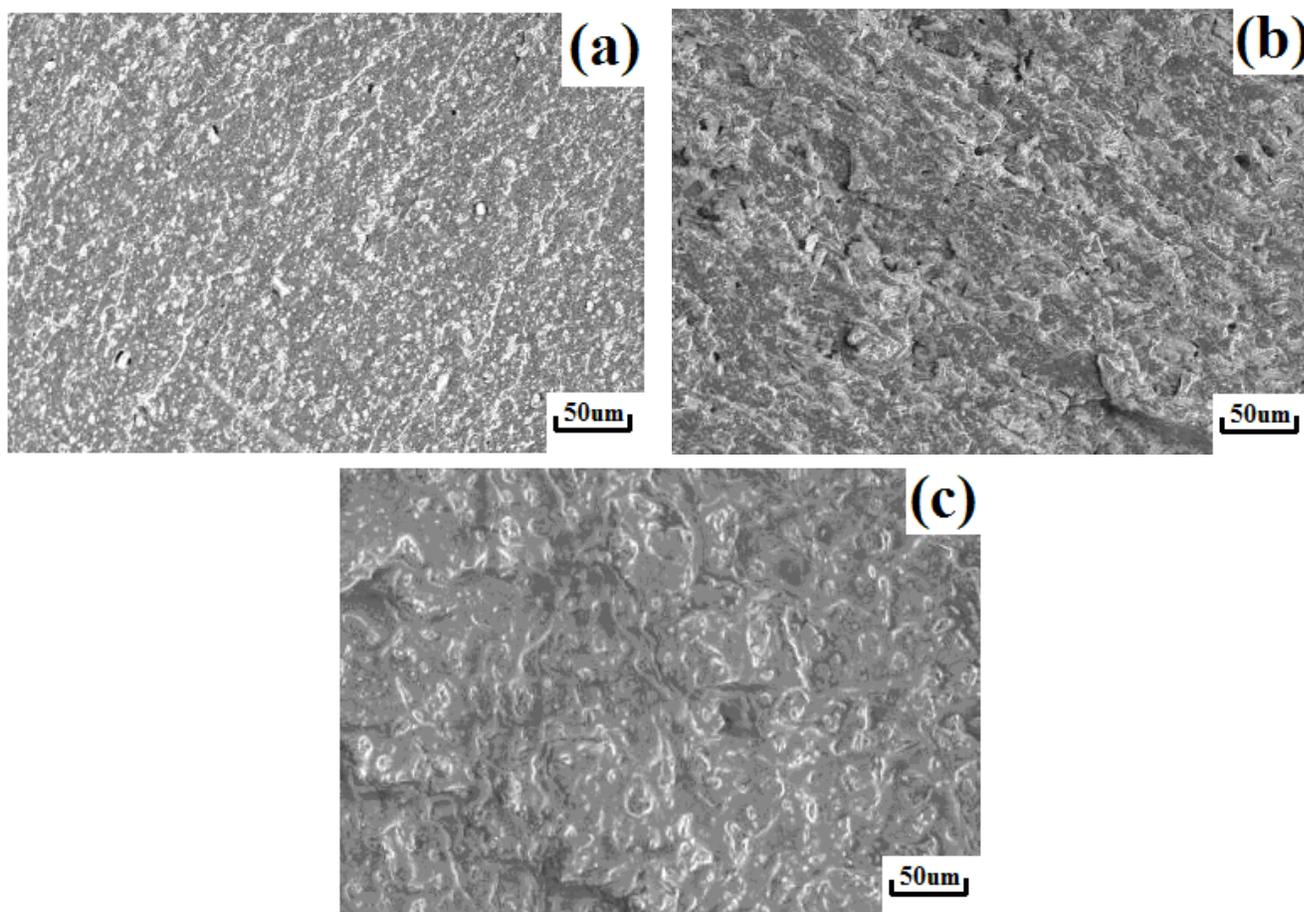


Figure 6: SEM images (x1000) for (a) NBR/POM (80/20), and (b) NBR/MAH-g-POM (80/20).

since better dispersion of POM in NBR was observed and the average size of the POM particles was smaller. Besides, little change in tear lines on the tensile-fractured surfaces between the two blends can be seen.

Figure 7 presents the comparison of tensile-fractured surfaces among various blends of NBR/MAH-g-POM/POM with the compositions of 90/3/10, 80/3/20 and 70/3/30 (all in weight percent). It can be seen that as the POM content increased, the fracture surface became rough. The change of morphological of



**Figure 7:** SEM images ( $\times 300$ ) for (a) NBR/MAH-g-POM/POM (90/3/10), (b) NBR/MAH-g-POM/POM (80/3/20), and (c) NBR/MAH-g-POM/POM (70/3/30).

fracture surface as a result of incorporation of plastic has been reported by various researchers [16]. Figure 7b shows an uneven distribution of the dispersed POM particles, which indicated the weak interface between the two phases. Figure 7c showed that no obvious phase separation phenomenon can be observed, also suggesting that a better compatibility between NBR and POM had been achieved. It is thus concluded that the compatibility of NBR/POM blends was significantly enhanced with increasing POM content, which was in accordance with previous publications [27, 28].

### 3.3. Tensile Property

Table 3 shows that when POM was added the tensile strength was significantly improved, while the elongation at break of NBR decreased. It indicated that the mechanical properties of NBR can be greatly improved with the introduction of POM. For NBR/MAH-g-POM blend, the elongation at break was higher than NBR/POM blend and neat NBR, indicating that the compatibility of NBR/MAH-g-POM was better than NBR/POM blend. Table 3 also showed that the tensile

strength of NBR/MAH-g-POM was significantly decreased, which could be caused by a certain degree of degradation of POM when it was grafted with MAH. At the same time, it was also found that the mechanical properties of NBR/MAH-g-POM/POM were weaker than those of NBR/MAH-g-POM, which could possibly be caused by the presence of macromolecular compatibilizer.

Overall, the compatibility of NBR/MAH-g-POM was found to be better than NBR/POM. However, there was still a certain degree of degradation of POM when it was grafted with MAH. Thus, to know how to prevent the degradation of POM during the grafting process will be insightful to the further modification of NBR (esp. for improvement on the comprehensive performance of NBR/POM blends).

### 4. CONCLUDING REMARKS

In this work, it was proved that POM can be successfully grafted with MAH. As compared to neat POM, both melting temperature and crystallinity of

Table 3: Experimental Data of Tensile Property for Various Samples

Samples	Elongation at break (%)	Tensile strength (MPa)
NBR	389.8	1.9
POM <sup>a</sup>	60.0	60.0
NBR/POM (80/20)	378.7	9.5
NBR/ MAH-g-POM (80/20)	393.0	4.1
NBR/MAH/POM (80/20/3)	301.2	4.3
NBR/ MAH-g-POM /POM (70/3/30)	284.3	6.7
NBR/ MAH-g-POM /POM (80/3/20)	326.6	6.2
NBR/ MAH-g-POM /POM (90/3/10)	363.2	4.5

<sup>a</sup>Data obtained from the material manufacturer.

MAH-g-POM were reduced to an extent. Meanwhile, the compatibility, thermal behavior, and mechanical properties of NBR/ POM composites were also studied. It was found that the more POM content, the better compatibility of NBR/POM blends. The compatibility of NBR/MAH-g-POM can be greatly improved as compared with the NBR/POM blends. The melting temperature of NBR/MAH-g-POM/POM blend was also higher than that of NBR/POM blend. The present study will be helpful to an improved understanding of the correlation among “processing-structure-property” of polymeric composites/blends.

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