

Compatibility Study in Natural Rubber and Maize Starch Blends

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ABSTRACT: Blends containing various ratios of natural rubber (NR) and maize starch (MS) were prepared on a two roll mill. The effect of starch contents on physico-mechanical properties and curing characteristics of the prepared blend vulcanizates was investigated. The data indicate poor mechanical properties, delayed cure rate index, and decreased maximum torque with increasing starch content in the blend formulation. This indicates that the interfacial interaction between the blend components was poor. Various contents of the compatibilizers, maleic acid anhydride (MAH) and glycidyl methacrylate (GMA), were mixed with the blend NR/MS (90/10). The effect of the compatibilizer contents on the physico-mechanical properties and curing

characteristics of the binary blend was investigated. Compatibilized blends with GMA (1 phr) showed an improvement in the physico-mechanical properties in comparison with uncompatibilized blend samples. Blends with MAH exhibited higher modulus and hardness values with respect to GMA blends. The efficiency of the compatibilizers was also evaluated by studies of phase morphology (scanning electron microscope), Fourier transform infrared spectroscopy, and thermal stability (thermogravimetric analysis). © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 125: 959–967, 2012

Key words: compatibility; blends; mechanical properties; curing of polymers; rheology

INTRODUCTION

With the extensive use of synthetic polymers, petrochemical energy shortage and environmental pollution has become increasingly serious. The use of polymer materials prepared by natural renewable resources has become the focus subject in current basic and applied research.^{1,2}

The blending of polymers with starch, as wide spread natural resources, has attracted considerable interest in industry and academic for developing biodegradable polymers with a reasonable cost and desired combination of properties.^{3–7} This is based on the advantages that starch is an inexpensive, abundant supply, renewable, environmental amity, and fully biodegradable material. In the plastics industry, a variety of biodegradable plastic products have been developed from a large number of applications of starch and its derivatives.^{8–13} Also, in the rubber industry, the development of starch has attracted more attention.^{14,15}

In general, there are three approaches to prepare rubber/starch compounds. The first is to modify a polymer matrix and then mix starch with the modi-

fied matrix. The matrix is functionalized (i.e., grafted) with acrylic acid¹⁶ or with maleic anhydride after plasma treatment¹⁷ to improve the compatibility and interfacial adhesion between the constituents. This method is relatively expensive but it can obtain better mechanical, thermal, and biodegradation properties of the modified matrix/starch composite as compared with unmodified one. Furthermore, its lower viscosity makes it easier to process. The second approach is to modify starch and mix the modified starch with matrix because carbon black as conventional filler for elastomer reinforcement is wholly dependent on the nonrenewable petroleum resource. Starch paste was chemically modified with esterification,¹⁸ and by grafting with polybutylacrylate.¹⁹ The modified starch was investigated as reinforcing filler of rubber through mixing and co-coagulating with rubber latex.¹⁹ A novel starch modification for filler-reinforced elastomer was developed using two types of modifiers, i.e., resorcinol–formaldehyde (RF) and a silane surfactant (S). When the starch was co-modified by both, the mechanical properties improvement of styrene–butadiene rubber was superior to that reinforced by carbon black.²⁰ *In situ* modified starch with combination of (RF) and (S) during latex compounding process was developed as a new strategy to strengthen the interfacial interaction.²¹ Modification of starch such as gelatinization (combined with co-coagulating rubber latex)²² and acid hydrolysis (to prepare starch nanocrystals)²³ have been

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TABLE I
Formulations of Rubber Compounds of the Blends

Ingredients	Compound No.						
	N1	N2	N3	N4	N5	N6	N7
	Content (phr) ^a						
NR	100	90	80	70	60	50	40
Zinc oxide	5	4.5	4	3.5	3	2.5	2
Sulfur	2.5	2.25	2	1.75	1.5	1.25	1
Stearic acid	2	1.8	1.6	1.4	1.2	1	0.8
PβN ^b	1	0.9	0.8	0.7	0.6	0.5	0.4
CBS ^c	1	0.9	0.8	0.7	0.6	0.5	0.4
MS	0	10	20	30	40	50	60

^a Parts per hundred parts of rubber.

^b Phenyl-β-naphthylamine.

^c N-Cyclohexyl-2-benzothiazole sulphenamide.

examined. The modifier is designed to interact with starch and rubber to prevent hydrogen bonding and crystallization of starch and to improve compatibility between starch and rubber. The third approach is to use compatibilizer, maleated natural rubber was prepared and used as a blending composition and a compatibilizer for blending of natural rubber (STR5L) and cassava starch.²⁴ Polyethylene-octene/maleic anhydride graft copolymer (POE-MA) was prepared with grafting percentage of about 0.78% and used as compatibilizer for POE elastomer and waxy corn starch blends.²⁵ Thermoplastic starch/linear low-density polyethylene blends were prepared using LLDPE-g-MA as compatibilizer.²⁶ The addition of compatibilizer can lower the size of starch phase with homogeneous dispersion throughout the matrix leading to better mechanical properties comparing with blends without compatibilizer. This method is simple, but depends on the availability and efficiency of the compatibilizers.

The aim of this work is to study the potential utilization of maleic acid anhydride (MAH) and glycidyl methacrylate (GMA) as compatibilizers for NR/MS blend vulcanizates and investigate systematically their effect on the compatibility, physico-mechanical properties, curing characteristics, morphology, and thermal stability.

EXPERIMENTAL

Materials

- Natural rubber (NR): ribbed smoked sheets (RSS-1) with specific gravity 0.913, Mooney viscosity ML (1 + 4), 60–90 at 100°C, and T_g -75°C was product from Malaysia.
- Maize starch (MS) was supplied by El Nasr Pharma Central Chemical Company, Abu Zabal, Egypt.

- Stearic acid with white flakes and specific gravity 0.9–0.97 at 15°C was supplied by Aldrich Company, Germany.
- Zinc oxide (ZnO) with specific gravity 5.55–5.61 was supplied by Aldrich Company, Germany.
- The antioxidant used was phenyl β-naphthylamine (PβN) with specific gravity 1.18–1.24 and melting point 105–106°C supplied by Aldrich Company, Germany.
- Elemental sulfur (S) with fine pale yellow powder and specific gravity 2.04–2.06 was supplied by Aldrich Company, Germany.
- N-Cyclohexyl-2-benzothiazole sulphenamide (CBS) with specific gravity 1.27–1.31, melting range 95–100°C, and pale grey powder was supplied by Aldrich Company, Germany.
- MAH with melting point 54–56°C and number average molecular weight of 98.06 g mol⁻¹ was purchased from Merck, Darmstadt, Germany.
- GMA with boiling point 189°C and a density of 1.042 g/mL at 25°C was purchased from Merck, Darmstadt, Germany.

Compounding and testing

Compounding of NR was carried out on a two-roll open mill according to ASTM D 3182-2007. The formulations of the mixes are given in Table I. The rheograph of the mixes and their cure characteristics were determined with an oscillating disc rheometer 100 (Monsanto) according to ASTM-D 2084-2007. The mixes were cured in a hydraulic press at 142°C for the optimum cure time. The mechanical properties (i.e., tensile strength, elongation at break, and Young's modulus) of vulcanized sheets were determined with Zwick 1425 testing machine (Germany). The compressed sheets were cut into dumbbell-shaped specimens with appropriate punching dies

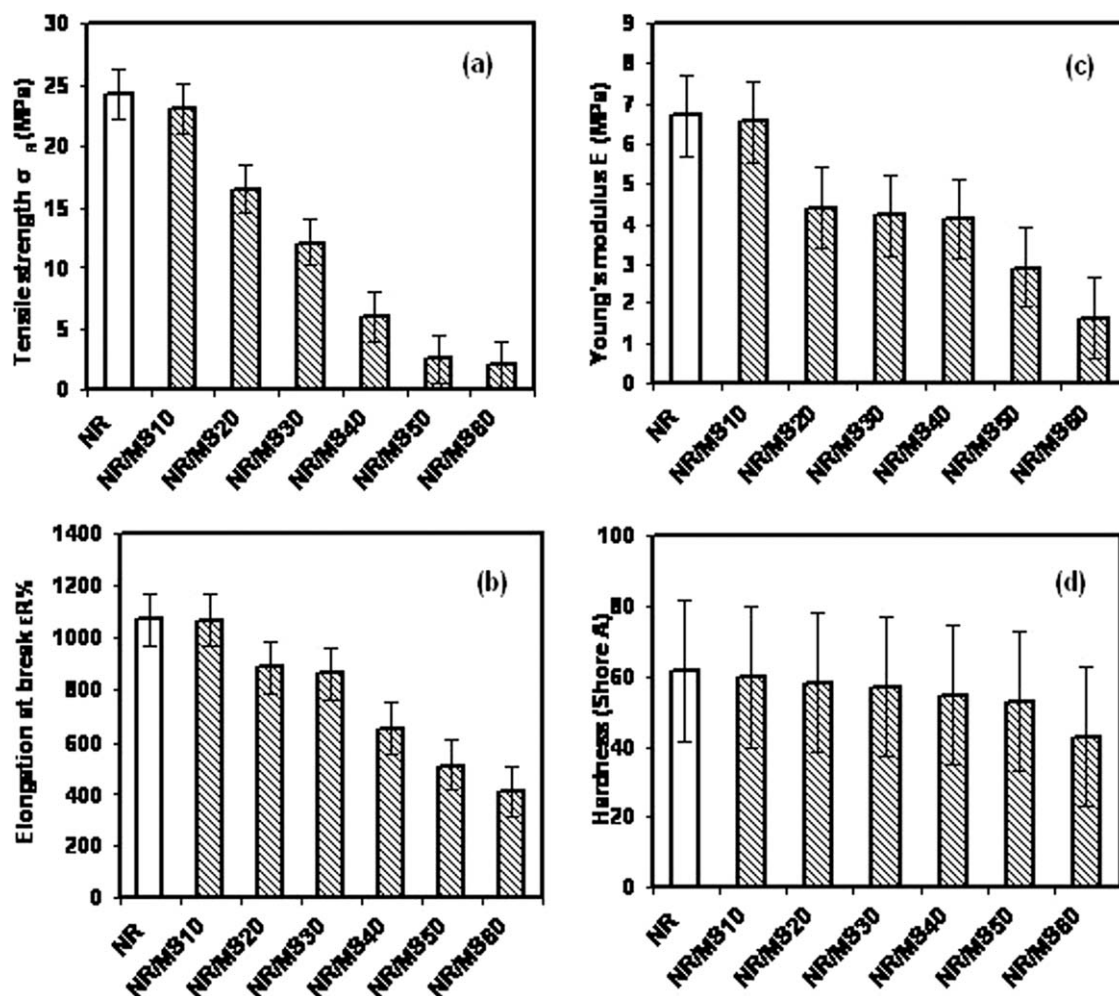


Figure 1 Physico-mechanical properties as a function of starch content, (a) tensile strength (Mpa), (b) elongation at break (%), (c) Young's modulus (MPa), and (d) hardness (Shore A).

with a width of 4 mm (DIN 53504 STABIN \wedge EF). The specimens, with a width of 4 mm, a neck length of 15 mm, were tested at a crosshead speed of 500 mm/min and with a load cell of 10–25N according to ASTM D 624-2007. The hardness of test specimens at least 6-mm thick was measured with a Shore A durometer (Hartepuffer; DIN53505) according to ASTM D 2240-2007. All these tests were performed at room temperature ($25 \pm 1^\circ\text{C}$), and the reported results were averaged from a minimum of five specimens. The surface morphology of the samples was examined through scanning electron microscope (SEM, JEOL 5300). The samples were coated with a very thin layer of gold. The middle part of the cross-section of the specimens was chosen as representative of the overall morphology. The Fourier transform infrared (FTIR) spectra of samples were obtained at room temperature using a Jascow FTIR 430 series infrared spectrophotometer equipped with KBr discs. The scanning range was from 520 to 4000 cm^{-1} . Thermogravimetric analysis (TGA) was conducted with Shimadzu TGA-50H. A typical sample

weight was about 8–10 mg and the analysis was performed at a heating rate of $10^\circ\text{C}/\text{min}$ from 50 to 600°C under nitrogen atmosphere.

RESULTS AND DISCUSSIONS

Uncompatibilized NR/MS blends

Effect of starch contents on physico-mechanical properties

Figure 1(a–d) summarizes the effect of maize starch, MS, content on tensile strength, σ_R , elongation at break, ε_R , Young's modulus, E , and hardness Shore A of blend samples as compared with those of pure NR. Mechanical tests were not done for the pure starch because it was not possible to prepare suitable films with the process used here.

It was found that the tensile strength, σ_R , Figure 1(a), drastically decreased with the increase of MS content. This was in harmony with the results presented by Nakason et al.²⁷ and was also consistent with the data of Ichazo et al.²⁸ A possible explanation

TABLE II
Curing Characteristics of the Mixes at $142 \pm 1^\circ\text{C}$

Curing characteristics	Compound No.						
	N1	N2	N3	N4	N5	N6	N7
t_{s2} , (min)	6.3	6.2	6.2	6.2	6.2	6.1	6.2
t_{c90} , (min)	9.3	9.5	10	10.7	11.1	11.5	11.9
CRI, (min^{-1})	33.33	30.30	26.32	22.22	20.41	18.52	17.54
M_L (dN.m)	2.5	2.5	2.0	2.0	2.0	2.0	2.0
M_H (dN.m)	37	36	35.5	32	30	28	26.9
$M_H - M_L$ (dN.m)	34.5	33.5	33.5	30	28	26	24.9

Scorch time (t_{s2}), optimum cure time (t_{c90}), cure rate index (CRI), minimum torque (M_L), maximum torque (M_H).

for the decrease of σ_R with the incorporation of starch into NR could be attributed to the low interfacial interaction between the components of the blends which would lead to mechanical rupture at the blend interface. Another factor to account for this observation was the relatively low extensibility of starch particles in NR matrix which would cause a low strain at break. The drop in σ_R was in accordance with the decrease in the effective cross-sectional area of NR matrix. Further evidence on the dispersion or agglomeration of starch particles in the NR matrix will be provided by SEM analysis.

Figure 1(b) indicates that the ϵ_R was higher for NR and decreased markedly with the addition of MS. These poor strength properties may be attributed to the geometry and the concentration of the starch particles in the compounding formulation. The strength of the compounds decreases due to the inability of the starch particles to support stress transferred from the elastomeric phase.²⁷ In synthetic polymer blends with a ductile matrix, the ϵ_R is considered to be highly sensitive to the state of the interface.²⁹ The reduction in the ϵ_R of the NR/MS blends could be attributed to the lack of compatibility between hydrophobic NR and hydrophilic starch or the poor interfacial adhesion between NR and starch as discussed later by SEM.

The effect of starch content on Young's modulus is indicated in Figure 1(c). The modulus was decreased additively by the incorporation of MS. A similar observation could be seen in Figure 1(d) for hardness test results. The decrease in Young's modulus and hardness after addition of starch probably reflected the heterogeneous distribution of starch in NR and the low interfacial interaction between components of the blend that resulted in mechanical rupture at the blend interface.

Effect of starch contents on curing characteristics

Table II gives the results of curing characteristics of the mixes at different starch contents (viz., 0, 10, 20, 30, 40, 50, 60 phr). Little effect on the scorch time,

t_{s2} , was seen with increasing quantities of the MS in the blend formulation. An increasing trend of the optimum cure time, t_{c90} , was observed on increasing quantity of MS. These results were in agreement with Nakason et al.²⁴

Cure rate index, a kinetic parameter of vulcanization, is estimated as follows:

$$\text{CRI} = 100 / (t_{c90} - t_{s2})$$

The rate of cure delayed with decreasing concentration of NR (i.e., increasing starch content in the blend) as confirmed by an increase in the cure time t_{c90} . This decrease in cure rate caused the vulcanization reaction to decrease and create less active cross-link sites in the rubber compound. This effect may be attributed to decreasing amounts of the cure ingredients with a decreasing of NR in the blend formulation (see Table I). Since the starch is absent from vulcanizing site and it has form of particulates, the ingredients of vulcanization will be more concentrated in the NR region.

The M_H and M_L data indicate that Δ torque ($M_H - M_L$) value decreased with a slight decrease in sulfur and accelerator level, as would be expected, indicating no chemical interaction between the two phases. This was in accordance with decreasing Young's modulus and hardness with increasing starch content as shown before Figure 1(c,d).

Compatibilized NR/MS blend vulcanizates

Effect of the compatibilizers on physico-mechanical properties

Figure 2(a–d) summarizes the effect of adding MAH and GMA compatibilizers with various contents (i.e., 1, 3, 5, 7, 9 phr) on the physico-mechanical properties (viz., σ_R , ϵ_R , E , and hardness) of NR/MS (90/10) blends. Compatibilized blends with GMA (1 phr) showed an increase in both σ_R and ϵ_R , Figure 2(a,b), as compared with MAH (1 phr). The highest properties of 1 phr GMA compatibilized blend were due to the presence of epoxy group. The epoxy group

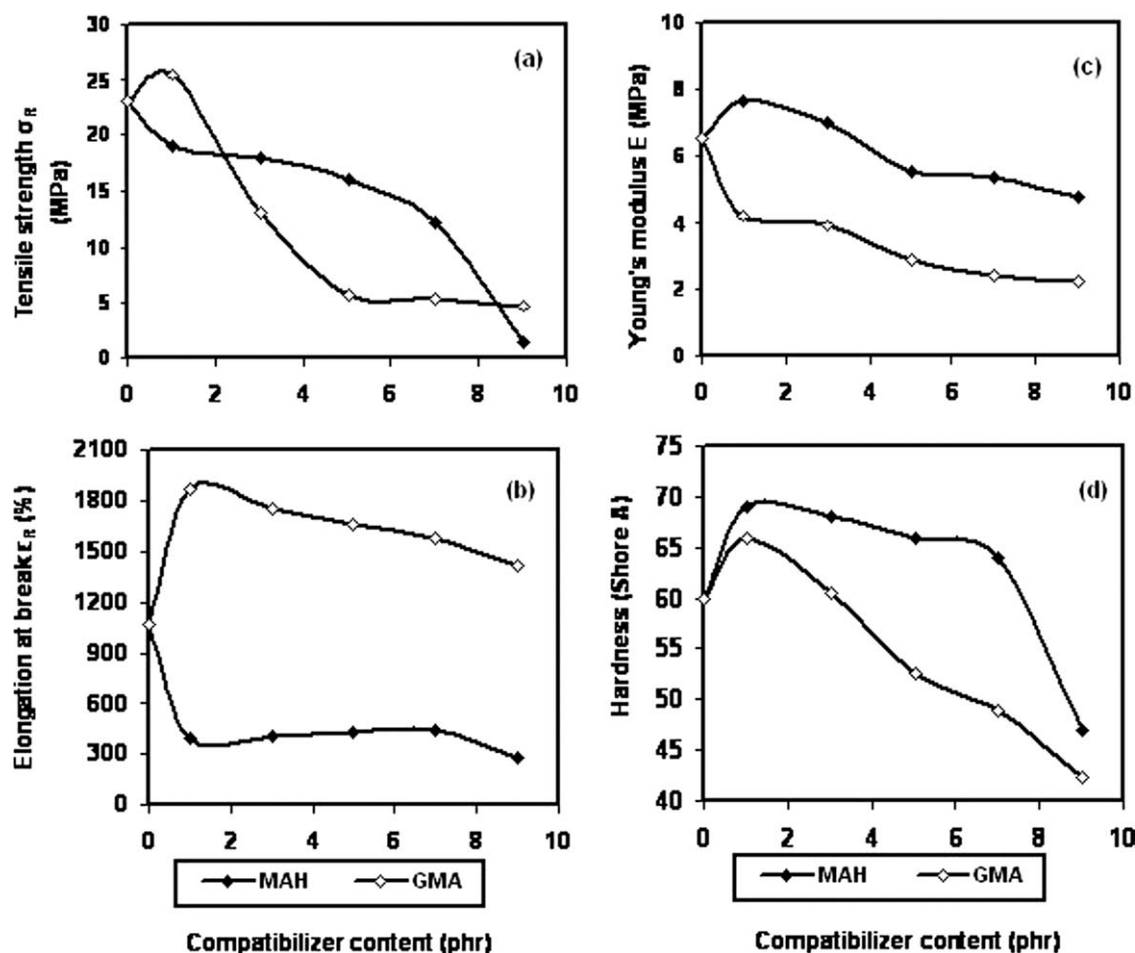


Figure 2 Physico-mechanical properties of NR/MS (90/10) blend versus compatibilizer content (a) tensile strength (Mpa), (b) elongation at break (ϵ_R (%)), (c) Young's modulus (Mpa), and (d) hardness (Shore A).

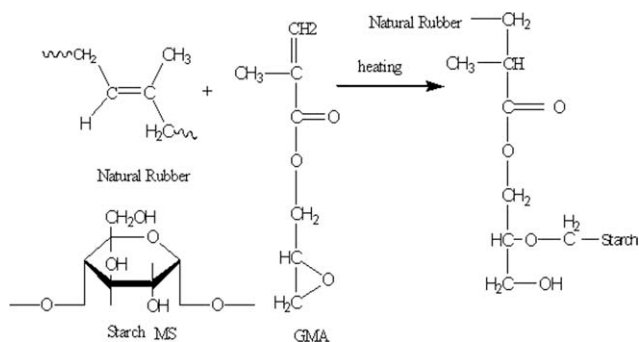
chemically interacts with the hydroxyl group of starch and the chemical reaction can be depicted as shown in Scheme 1.

This would promise a great decrease of cohesion energy and crystallization of starch. In other words; this would effectively prevent the gathering as well as regular packing of starch. Therefore, better dispersion of starch in NR matrix as well as better interfacial interaction between hydrophilic starch and hydrophobic NR could be expected, which would allow an improvement of mechanical properties of the composites. Thus, the presence of epoxy group in the compatibilizer structure increased its efficiency in enhancing the interaction between NR and starch phases. This increase in adhesion was evident from the scanning electron micrographs [Fig. 3(b)] with the lowest particle size of starch domains in this system, as explained later.

MAH compatibilized blend samples showed a decrease in tensile properties with a reduced flexibility with respect to GMA ternary samples. This was confirmed by SEM, showing no reduction in particle size at 1 phr MAH [Fig.3(c)], as explained later. Sim-

ilar results have been reported for the NR/cassava starch/maleated NR formulations.²⁸ It is believed that the polar part of MAH forms hydrogen bonds with the hydroxyl groups of starch in the interfacial region. Note that GMA and MAH with unsaturated double bonds may react with NR by the action of high temperature and stress.

The data in Figure 2(c,d) indicate the stiffness character of MAH compatibilized samples (i.e., higher



Scheme 1 Proposed chemical interactions between NR, MS and GMA.

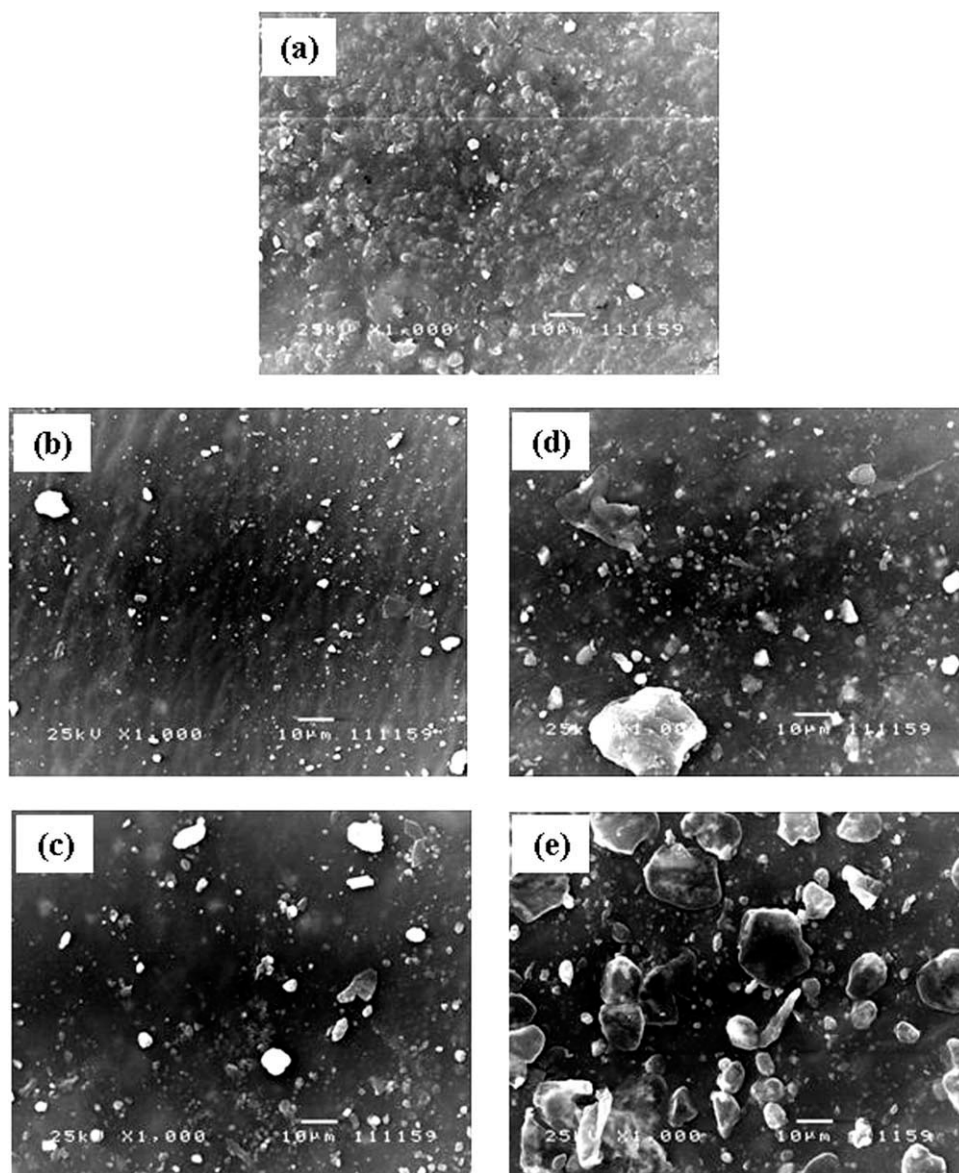


Figure 3 SEM of different composition surfaces: (a) NR/MS (90/10) blend sample without compatibilizer, (b) blend sample with GMA (1 phr), (c) blend sample with MAH (1 phr), (d) blend sample with GMA (7 phr), and (e) blend sample with MAH (7 phr).

Young's modulus and hardness values) with respect to GMA blends. The increase in Young's modulus and hardness with MAH clearly indicated that MAH increased rigidity of blend samples through the introduction of restrictions in the mobility or flexibility of polymer molecules.

On the other hand, at higher starch content (7 phr), the decrease in mechanical properties may be due to aggregation of starch and its inhomogeneous dispersion in rubbery matrix, as corroborated by results deduced from SEM images [Fig. 3(d,e)]. The formation of aggregates reduces the interface area between the matrix and starch particles and leads to the formation of stress concentration weak points in the matrix, consequently reducing the mechanical properties.

It is worth to find that the variation of the mechanical properties of the blends with different compatibilizers depends on the morphologies of the blends with respect to the size of the dispersed phase, interfacial adhesion, and dispersion of the minority phase in the matrix. Also, this variation depends on the chemical structure of the tested compatibilizer, which determined the main mechanisms of phase interaction.

Effect of the compatibilizers on curing characteristics

The cure characteristics of NR/MS blends (90/10) compatibilized with various contents of MAH and GMA are shown in Table III. For blends with MAH,

TABLE III
Curing Characteristics of NR/MS (90/10) Blend with and Without Compatibilizers

Blend ratio		Compatibilizer		Curing characteristics				
NR	MS	Type	Concentration (phr)	t_{s2} (min)	t_{c90} (min)	CRI (min^{-1})	M_L (dN.m)	M_H (dN.m)
90	10	MAH	–	6.2	9.5	30.30	2.5	36
			1	7.0	14.5	13.33	2.5	67
			3	7.5	15.5	12.5	2.8	67.4
			5	8.70	16.9	12.19	2.86	67.5
			7	9.15	17.75	11.63	2.9	71.5
		GMA	9	11.75	22.5	9.30	2.95	72.1
			1	6	8.88	34.72	2.56	38
			3	5.95	8.76	35.59	2.61	39
			5	5.87	8.64	36.10	2.63	39.2
			7	5.2	7.88	37.31	2.67	39.5
			9	4.92	7.56	37.88	2.68	39.9

it was found that t_{s2} and t_{c90} increased with increasing MAH content as compared with those with GMA. This reflected the decrease in CRI for compatibilized blends with MAH upon using CBS as an accelerator. The intensive retardation for the curing of CBS-accelerator was widely accepted because of the presence of acids or anhydrides in the rubber compound. A reaction between CBS and free maleic anhydride molecules and/or succinic acid (from a ring-opening reaction of the maleic anhydride) was the main reason for the delayed onset of vulcanization. This was consistent with the data of Nakason et al.²⁴ and Tchazo et al.²⁸ on using mercapto accelerators such as MBT and MBTS.

On the other hand, it was found that CRI increased with increasing GMA content and it supported the activation of the cure reaction with GMA compatibilizer.

M_H (maximum torque) generally correlates with hardness and or modulus.³⁰ The data indicate that the addition of a compatibilizer increased the torque values of blend samples as compared with uncompatibilized ones. Also, MAH compatibilized blend samples exhibited higher M_H values with respect to GMA samples; this was due to the stiffness character of MAH, as discussed before Figure 2(c,d). While M_L (minimum torque) values were not affected by the compatibilizer type or its content.

Morphological study

SEM micrographs of the fractured surface of NR/MS (90/10) with and without compatibilizers are shown in Figure 3(a–e). It was found that the dispersion of starch component in the blend was not uniform; Figure 3(a) indicates clear heterogeneous morphology of the blend. The distinct differences in polarity and surface free energy of starch and NR matrix could be cited as one of the possible causes for this observation. This supported our previous explanation that the interfacial interaction between starch and NR matrix was poor. Therefore, SEM observation of starch blends

provides visual evidence for the deterioration of mechanical properties of blends as indicated before.

The sample containing 1 phr GMA Figure 3(b) showed better starch dispersion with less undispersed agglomerates with respect to that containing 1 phr MAH blend Figure 3(c). At higher level of compatibilizer (i.e., 7 phr), the vulcanizates indicate a rough surface with many agglomerates due to phase separation as seen in Figure 3(d,e) for GMA and MAH compatibilized blends, respectively.

FTIR measurements

Figure 4(a–c) shows the FTIR spectra of NR/MS (90/10) blend with and without compatibilizers. FTIR measurements, Figure 4(a), indicate that no intermolecular interactions exist between the two polymers as no shift in the absorption peaks of the NR or starch in the blends was observed, which suggests that NR and starch are incompatible.

Compared the spectra of NR/MS/MAH blend and NR/MS/GMA blends, it can be seen that the absence of C–C double bond at about 1623 cm^{-1} of

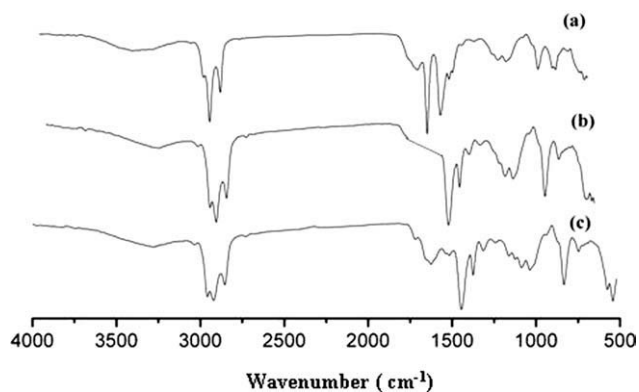


Figure 4 FTIR spectra: (a) NR/MS (90/10) blend sample, (b) blend sample with MAH (1 phr), and (c) blend sample with GMA (1 phr).

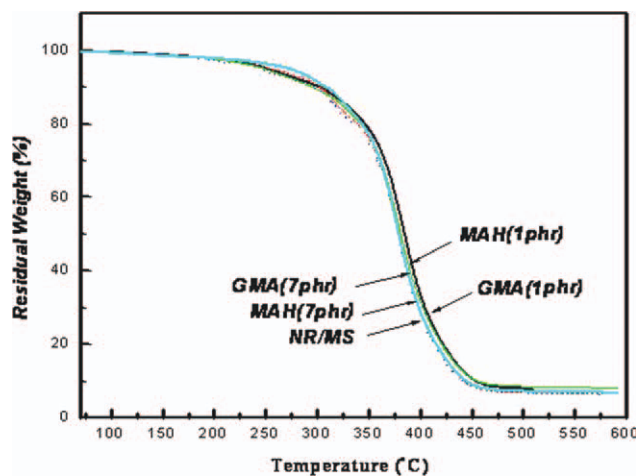


Figure 5 TGA curves of NR/MS (90/10) blend with and without compatibilizers. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

MAH structure,³¹ Figure 4(b), indicate that C—C radical of MAH was formed with NR, without an initiator, by the action of high temperature and stress. The presence of an absorption peak at 1700 cm^{-1} , assigned to the ester carbonyl stretching vibration in the copolymer.

In the spectrum of NR/MS/GMA, Figure 4(c), the absence of a C—C double bond peak at 689 cm^{-1} for GMA may be due to the reaction of GMA with NR through C—C double bond by the action of high temperature and without an initiator. It was also interesting to note that the characteristic peak of epoxy group in GMA (i.e., 912 cm^{-1})³² has disappeared with the incorporation of MS and the appearance of the ether link corresponding to an absorption peak at $1300\text{--}1000\text{ cm}^{-1}$. This may be attributed to the chemical reaction between the OH group of starch and the epoxy group of the compatibilizer during melt processing of NR/MS/GMA (90/10/1 phr).

Thus, FTIR was an evidence of the interaction of the tested compatibilizers with NR/MS blend vulcanizates.

Thermogravimetric analysis

The TGA results for the uncompatibilized NR/MS (90/10) blends and ternary ones with the tested compatibilizers at 1 and 7 (phr) under nitrogen

atmosphere at a heating rate of $10^\circ\text{C}/\text{min}$ are shown in Figure 5 and summarized in Table IV. The TGA results thermograms indicate the initial thermal degradation temperature (T_i) corresponding to $\sim 5\text{--}7\text{ wt } \%$ loss, after that degradation occurred rapidly. T_{25} , T_{50} , and T_{75} are the temperatures at which 25, 50, and 75 wt % loss occurred, respectively, and the final decomposition temperature (T_f) correspond to the temperature at maximum rate of weight loss.

The T_i value increased from 271 for uncompatibilized blends to 303 for MAH (1 phr) to 307 for GMA (1 phr) blends. Also, T_f for the compatibilized blends was higher than that for the uncompatibilized blends. T_f for the GMA (1 phr) blends was as high as 482°C with the highest residual weight percentage. Thus, the addition of a compatibilizer (1 phr) to the system brought the stability of the dispersed phase due to compatibilization. In the presence of GMA (1 phr) compatibilizer, a finer and stable morphology, Figure 3(b), was obtained and the thermal stability increased compared with that of MAH (1 phr) blends. On the other hand, the blends without compatibilizers tend to form a separate phase, thus destabilizing the phase morphology and lowering the thermal stability. This initiates degradation at a much lower temperature than expected.³³ Also, it has been observed that T_i and T_f for compatibilized blends with MAH or GMA (1 phr) occurred at higher temperatures compared with those of blend samples with 7 phr compatibilizer content. The same trend was also observed for T_{25} , T_{50} , and T_{75} . Thus, the data explain the decrease in thermal stability with increasing compatibilizer content in NR/MS blends. This was confirmed by blend morphology Figure 3(d,e) where the phase separation of blend components at higher compatibilizer content (7 phr) contributes to poor thermal stability.

CONCLUSIONS

From this study it could be concluded that:

1. The deterioration of the physico-mechanical properties of NR/MS blend vulcanizates with increasing MS content ranging between 10 and 60 phr in the blend.

TABLE IV
Thermal Stability of NR/MS (90/10) Blend with and Without Compatibilizers

Residual (%)	Weight-loss temperature ($^\circ\text{C}$)					Sample
	T_f	T_{75}	T_{50}	T_{25}	T_i	
7.6	462	404	377	354	271	NR/MS (90/10)
8.5	470	407	379	353	303	NR/MS/MAH (1 phr)
10.5	482	411	387	359	307	NR/MS/GMA(1 phr)
8.2	460	410	381	353	237	NR/MS /MAH (7 phr)
8.3	466	403	376	351	303	NR/MS/GMA(7 phr)

2. The retardation of the rate of cure reaction and a decreased trend in maximum torque were observed upon increasing starch content in the blend.
3. The compatibility of NR and MS is poor and can be enhanced by the addition of compatibilizers like MAH and GMA.
4. Blends with GMA (1 phr) showed an improvement in physico-mechanical properties (σ_R and ε_R), an increased cure rate index as confirmed by shorter optimum cure time with a smooth surface and fine dispersion, which in turn increased the thermal stability in comparison with those with MAH.
5. Minimum torque values were not affected by the compatibilizer type and content.
6. The addition of MAH or GMA at 7 phr, with a coarse morphology, did not improve the mechanical or thermal properties any further.

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