

Influence of Glycidyl Methacrylate Grafting on the Mechanical, Water Absorption, and Thermal Properties of Recycled High-Density Polyethylene/Rubber Seed Shell Particle Composites

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Rubber seed shell (RSS) was modified by grafting treatment using glycidyl methacrylate (GMA) at various concentrations. The RSS was then used to reinforce high-density polyethylene (HDPE). The effects of modification on the mechanical, water absorption, and thermal properties of the RSS/HDPE composites were studied using a mechanical testing instrument, weighing method, Vicat softening temperature (VST) testing, thermogravimetry, and dynamic mechanical analysis. The results showed that the GMA grafting produced an improvement in the flexural and tensile properties of the composites. The water absorption rate of the composites also had an obvious decrease. While a slight increase in VST was found, the various concentrations of GMA showed no improvement in VST. GMA modification also could elevate the thermal stability of the composites at the initial decomposition stage. The optimum grafting concentration of GMA (2.5%) led to the lowest thermal weight loss (37.07% and 26.56%) during the first and second decomposition stages. The E' values of the composites had a significant increase with the addition of GMA. There were two peaks of $\tan \delta$ for the untreated samples, but the modified samples exhibited a shift in the transition peak at higher temperatures; moreover, the second peak disappeared.

Keywords: High-density polyethylene; Rubber seed shell; Glycidyl methacrylate; Grafting

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INTRODUCTION

There has been a long history of rubber tree cultivation as an economic forest species in many low latitude tropical countries (Ng *et al.* 2014). In China, as rubber prices have tripled over the past decade, rubber tree plantations have experienced considerable growth mainly in the South and Southwest regions with plantation areas of 1.00 million ha (Sun and Jiang 2010; Xiao *et al.* 2014). The economical importance of rubber trees lies in the rubber latex. Rubber seed shell (RSS) as the primary by-products of the rubber tree has drawn little attention to explore their potential usefulness (Imanah and Okieimen 2003; Ismail and Shafiq 2014). In most cases, the RSS is just allowed to rot and then thrown away. In recent years, RSS has become of interest and is considered to be a promising source to produce activated carbon for the application of liquid and gas phase separation, deodorization, purification, storage, and catalysis (Nor *et al.* 2013; Xiao *et al.* 2014). Activated carbon has also been investigated for use as a reinforcing filler in various thermoplastic polymers (Ismail and Shafiq 2014).

Currently, waste plastic is generally considered as not only one of the major constituents of municipal solid waste but also as one of the most harmful components to the environment because of its non-biodegradable properties (Kumar *et al.* 2011). It has been reported that the plastic with the largest amount of consumption among polyolefins is high density polyethylene (HDPE), which constitutes one third of the world's plastic consumption (Chin *et al.* 2014). With the over-exploitation of natural resources and energy as well as the globally pervasive consciousness of environmental protection, high-valued utilization of agricultural and forestry by-products to develop novel materials has become popular. Combining natural plant fiber (wood, bamboo, sisal, jute, rice hull, *etc.*) instead of synthetic fiber (glass or carbon fiber) with recycled thermoplastic polymers is receiving more attention, due to the many advantages of natural fiber: enormous quantity, high specific strength and modulus, environmentally friendly characteristics, low cost, good machineability, and recyclability (Segerholm *et al.* 2012; Kaewkuk *et al.* 2013; Petchwattana and Covavisaruch 2013; Cavdar *et al.* 2014; Xue *et al.* 2014; Khan *et al.* 2015).

A recent study investigated the effects of RSS on the properties of polypropylene and polyethylene composites (Ismail and Shafiq 2014). Poor interfacial bonding between resin matrices and the RSS particles resulted in relatively low comprehensive physical and mechanical properties. This demonstrated that interfacial bonding promotion is necessary if the rubber seed shell is to be practically introduced into the composite products. When the interfacial bonding is strengthened, a RSS/polymer composite can be widely used in more products with a longer life, as it has acceptable comprehensive mechanical strength and is less susceptible to swelling, shrinking, and biodeterioration. Generally, there are some common methods for wood fiber pretreatment to enhance the interfacial interaction between the reinforcements and the plastic matrices, such as alkaline treatment, the addition of coupling agents or compatibilizers, as well as esterification and grafting treatment (Abdelmouleh *et al.* 2007; Wei *et al.* 2013).

Being similar to wood fiber, RSS can adopt an effective method for elevating its compatibility with wood fiber. It is well known that glycidyl methacrylate (GMA), having a reactive double bond and an epoxy group, is a very effective crosslinking monomer for graft polymerization (Devi and Maji 2007). GMA can be successfully grafted not only onto polymers but also onto natural fiber, *i.e.* cellulose. For instance, in several studies GMA was successfully grafted onto natural fiber or cellulose of plants, wood, as well as onto polypropylene and polyethylene to elevate the different physical and chemical functions of the materials (Devi and Maji 2007; Li and Wang 2007; Seko *et al.* 2010; Takácsa *et al.* 2012; Madrid *et al.* 2013; Ferreira *et al.* 2014). One study indicated that the monomer concentration had a significant effect on the grafting rate and the final properties of the grafted products (Wang and Han 2009). In this work's preceding study, a GMA grafting method was carried out to modify the surface properties of wood flour (WF)/chitosan (CS) mixtures to effectively reinforce the interfacial bonding properties of WF/CS/poly(vinyl chloride) composites (Xu *et al.* 2014).

The present study aims at investigating the effects of various GMA monomer concentrations on the mechanical properties, water absorption behavior, and comprehensive thermal performances of recycled HDPE/RSS composites by using an electronic universal mechanical testing instrument, weighing method, Vicat softening temperature testing (VST), thermogravimetry analysis (TGA), and dynamic mechanical analysis (DMA).

EXPERIMENTAL

Raw Materials

Recycled high density polyethylene (HDPE) pellets, with a melt flow index of 0.73 g/10 min and density of 960 kg/m³ was obtained from Xishuangbanna Huakun Biological Technology Co., Ltd, China. Rubber seed shell (RSS) particles with a size between 150 and 180 μm were kindly supplied by Xishuangbanna Huakun Biological Technology Co., Ltd, China. Glycidyl methacrylate (GMA) and ammonium ceric nitrate (ACN) were reagent grade without further purification and were purchased from Shanghai Saen Chemical Technology Co., Ltd, China. Other solvents and additives used were supplied by local chemical companies.

Modification of RSS

Prior to modification, the rubber seed shell (RSS) particles were dried at 105 °C for 48 h in an oven. The RSS particles were modified by chemical surface grafting according to a stated previous method with various concentrations of GMA (0 wt%, 1 wt%, 2.5 wt%, and 5 wt%) (Xu *et al.* 2014).

Preparation of Recycled HDPE/RSS Composites

The raw materials for the HDPE/RSS composites were 50 wt% recycled HDPE and 50 wt% RSS grafted by GMA, as shown in Table 1. The materials were mixed in a high speed blender for 15 min, and then the mixture was extruded in the shape of a rod by a co-rotating twin-screw extruder (JIEENTE SHJ-20, Nanjing, China) with a screw diameter of 20 mm and a length-to-diameter ratio of 36:1. The temperature profile along the extruder barrel and die was 150, 160, 170, 180, 170, and 165 °C, and the screw speed was 40 rpm. The extrudate was pelleted using a granulator and dried at 80 °C by air blowing. The granules were transferred to a conical twin-screw extruder (JINWEI SJZ-45, Suzhou, China) to produce sheet samples of 4 mm in thickness. The temperature of the different barrels and die zones were 150, 160, 170, 175, and 178 °C. The rotation speeds of the twin screw and single screw were 25 rpm and 10 rpm, respectively.

Table 1. Formulation of the HDPE/RSS Composites

Composite	HDPE (wt%)	RSS (wt%)	GMA Concentration* (wt%)
HDPE/RSS	50	50	0
HDPE/RSS-GMA1	50	50	1
HDPE/RSS-GMA2.5	50	50	2.5
HDPE/RSS-GMA5	50	50	5

* Based on RSS weight

Mechanical Properties Testing

The mechanical properties of the samples were measured using an electronic universal mechanical testing instrument (CMT5504, Shenzhen, China). The specific sample size for flexural testing was 80 mm (length) × 12 mm (width) × 4 mm (thickness) and the crosshead speed was 2 mm/min according to the ASTM D790 (2010) standard. The model for flexural testing involved a three-point bending test with a span of 64 mm. As for the tensile test, the specimen size was 165 mm (length) × 20 mm (at the ends in width), 12 mm (at the narrow region in width) × 4 mm (thickness), like a dumb-bell

shape, and the crosshead speed was 2 mm/min according to ASTM D638 (2010). Five samples were tested for each property and the averages and standard deviations were recorded.

Water Absorption Behavior Analysis

The specimens with dimensions of 20 mm × 20 mm × 4 mm (length × width × thickness) were completely immersed in distilled water at 23±2 °C for 25 days. The weights of the samples were measured daily. Prior to weight measurement, each sample was slightly wiped using filter paper to remove excess water, then immediately weighed and placed back in the water. The water absorption rate was determined based on the weight percent changes, calculated using Eq. 1,

$$WA = \frac{m_0 - m_t}{m_0} \times 100\% \quad (1)$$

where *WA* is the water absorption rate (%), *m*₀ is the weight before testing (g), and *m*_{*t*} is the weight at time *t* (g).

Vicat Softening Temperature (VST) Analysis

The Vicat softening temperature was determined with a HDT-VICAT-6P apparatus (Ceast Co., Italy) based on the standard ASTM D1525 (2007). Tests were conducted at a load of 10 ± 0.2 N, and the starting temperature was room temperature with a heating rate of 120 ± 10 °C/h.

Thermogravimetric Analysis (TGA)

The thermal stability of the composites was investigated using a thermal gravimetric analysis (TGA) instrument (TGA 209-F1, Netzsch Co., Germany) under a nitrogen atmosphere. The weight of the measured sample was about 5 to 10 mg. The temperature range used for the analysis was 40 to 800 °C at a heating rate of 10 °C/h.

Dynamic Mechanical Analysis (DMA)

A dynamic mechanical analysis instrument (DMA242C, Netzsch, Germany) was employed to study the storage modulus (*E'*) and mechanical damping factor (tan *δ*) of the composites using the three point bending mode. The composites were cut into specimens with dimensions of 55 mm (length) × 10 mm (width) × 4 mm (thickness). The testing frequency, temperature range, and heating rate were 1 Hz, 40 to 200 °C, and 3 °C/min, respectively.

RESULTS AND DISCUSSION

Mechanical Properties

The mechanical properties of composites are of great importance for their practical applications and depend highly on the miscibility and compatibility between the components. The flexural and tensile properties of the HDPE/RSS composites made of unmodified or GMA-modified RSS are shown in Fig. 1. For the HDPE/RSS control group, Fig. 1(a) shows that the flexural strength and modulus values were 33.43 MPa and 2.92 GPa, respectively. When the RSS particles were modified by GMA grafting at

different concentrations, there were significant differences among the various composites. Initially the flexural strength and modulus rapidly increased to 45.82 MPa and 3.74 GPa, respectively, with the GMA concentration increase from 0% to 2.5%. After the GMA concentration reached 5%, the two specific flexural indexes became stable within the range of error, corresponding to a flexural strength and modulus of 46.35 MPa and 3.80 GPa, respectively. This result can be ascribed to the fact that the interfacial bonding between the HDPE and RSS was effectively enhanced at this suitable grafting concentration. Additionally, the cellulose that was contained in the RSS reacted with the epoxy group of the GMA, causing the surface of the fibers to change from hydrophilic to hydrophobic; however, there possibly were a limited number of reactive sites during the reaction process. On another note, the vinyl group in the GMA molecular chain may have been greatly compatible with the polyethylene in the extrusion process of composite preparation. These two factors provided a synergistic effect on the interfacial bonding of the composites.

Figure 1(b) shows the tensile properties of the HDPE/RSS composites. There was a slight increase in tensile strength and modulus in the HDPE/RSS composites modified by 1% GMA, from 16.70 MPa and 4.11 GPa to 21.35 MPa and 4.35 GPa, respectively. With an increase in GMA concentration to 2.5% and 5%, there was very little (if any) increase in tensile strength and modulus. This possibly provides an important insight into the tensile properties of HDPE/RSS composites, as it appears that the shape of RSS probably had a greater effect on the tensile properties than the interfacial bonding ability; for instance, the greater the length-to-diameter ratio of the RSS, the better the tensile strength and modulus of the composite.

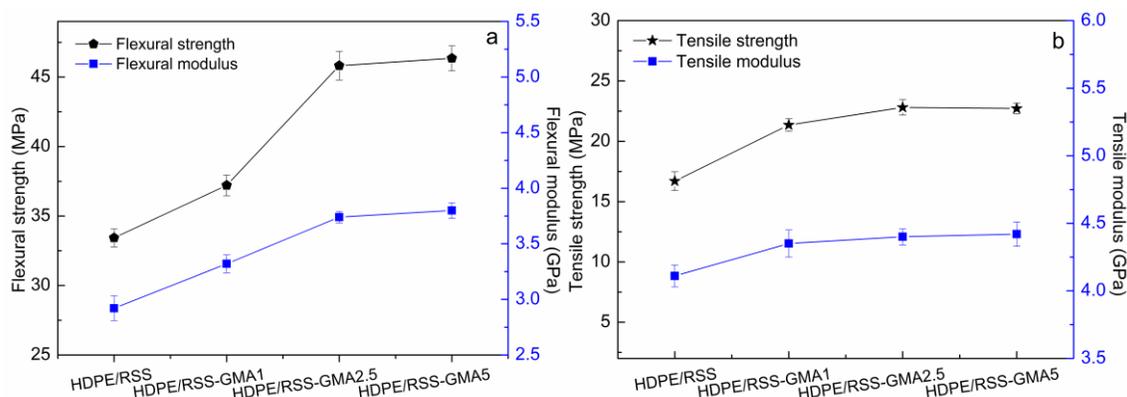


Fig. 1. Flexural and tensile properties of HDPE/RSS composites made of unmodified or GMA-modified RSS

Water Absorption Behavior

The water absorption curves of the different HDPE/RSS composites are shown in Fig. 2. The water absorption rate generally appeared to have a rapid linear ascending tendency, sharply increasing from 0% to 5.48%, 4.79%, 4.67%, and 4.75%, corresponding to GMA grafting concentrations of 0%, 1%, 2.5%, and 5%, respectively, in the initial stage (within 7 days). Afterward, the increasing rate of water absorption gradually slowed down, only increasing by 3.30%, 2.91%, 2.82%, and 2.59% from day 8 to 19. In the final stage, the water absorption rates for all the HDPE/RSS samples achieved a plateau (pseudoequilibrium) except for that of the unmodified sample, which still continued to rise. The maximum water absorption rates of the specimens after 25

days for GMA grafting concentrations of 0%, 1%, 2.5%, and 5% were 9.30%, 7.86%, 7.28%, and 7.61%, respectively.

GMA grafting effectively decreased the water absorption rate of the HDPE/RSS composites by an average of 18.49% within the experimental time range. It is concluded that this was due to the promotion of interfacial compatibility between the HDPE and the RSS, and also the advancement in equilibrium time for the maximum water absorption. The optimum GMA concentration for the GMA-grafted rubber seed shell was 2.5%, which was confirmed by the following analyses.

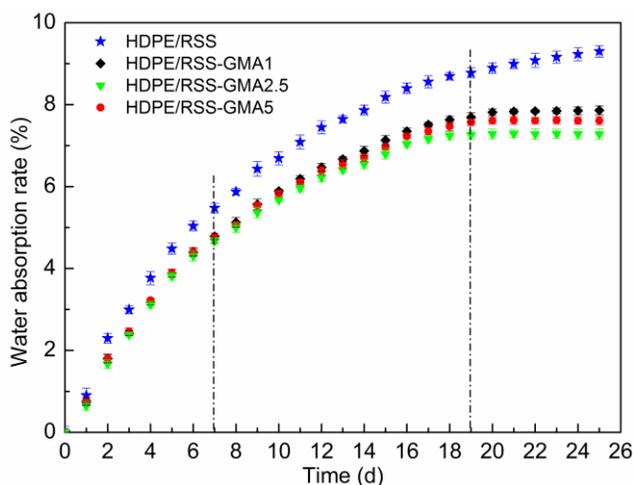


Fig. 2. Water absorption curves of HDPE/RSS composites made of unmodified or GMA-modified RSS

Vicat Softening Temperature (VST) Analysis

Vicat softening temperature is generally considered to be a good indication of the physical nature of miscible blends. Although the needle implicated in the measurement has a very small circular cross section (1 mm^2), the measurement is likely to suitably represent the overall content of the composites since the blending of different components is uniformly mixed throughout the cross section.

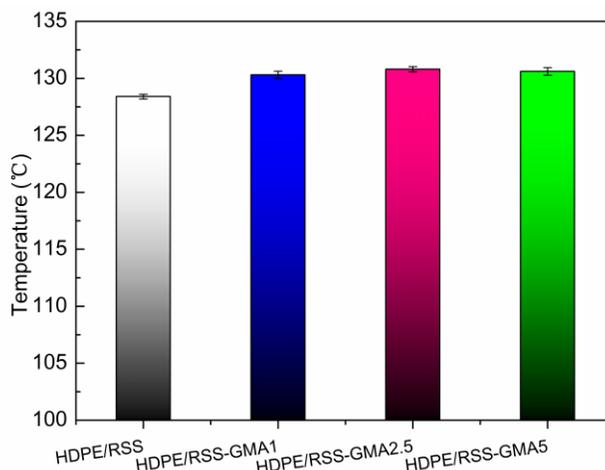


Fig. 3. Vicat softening temperature of HDPE/RSS composites made of unmodified or GMA-modified RSS

The VST variation trends of the HDPE/RSS composites made of unmodified or GMA-modified RSS are presented in Fig. 3. The VST of the HDPE/RSS samples with modified GMA using different concentrations (1%, 2.5%, and 5%) were slightly higher compared to that made of unmodified RSS, increasing from 128.4 °C to 130.3, 130.8, and 130.6 °C, respectively, which demonstrates that the ability of the composites to resist deformation was reinforced due to the promotion of interfacial bonding between the matrix and the RSS. Moreover, it was found that the HDPE/RSS composites made of modified RSS by various concentrations of GMA showed no marked improvement in VST. It is concluded that the GMA concentration was not the crucial factor for changing the VST despite the fact that GMA can be effectively used to ameliorate the interfacial bonding between HDPE and RSS.

Thermogravimetric Analysis (TGA)

The thermal stability of the HDPE/RSS composites made of unmodified or GMA-modified RSS are shown by the thermogravimetric curves in Fig. 4. As seen in Fig. 4(a), all of the thermogravimetric curves had similar shapes, and there were only small positional changes among the samples. The decomposition of lignin occurred through the whole thermal degradation process. The complete thermal degradation process of the composites was divided into two major weight loss stages, which correspond to the temperature ranges of 180 to 400 °C and 400 to 550 °C. The first stage corresponded to the decomposition of hemicelluloses and the partial decomposition of cellulose in the rubber seed shell. The second stage was mainly attributed to the fast decomposition of the HDPE matrix (Mir *et al.* 2011). In addition, a decreasing trend was observed for the TG curves in the temperature range of 70 to 180 °C, which may be attributed to the evaporation of residue water in the composites owing to possible moisture absorbed during storage and processing.

The weight loss data for the two main thermal degradation stages are listed in Table 2. The results reveal that the GMA-modified composite samples had a relatively lower weight loss compared to the control sample in the first and second stages, which demonstrates that GMA modification can elevate the thermal stability of HDPE/RSS composites during the initial thermal degradation process. Meanwhile, the composites modified by a GMA grafting concentration of 2.5% had the lowest weight loss (37.07 % and 26.56%) in stages 1 and 2.

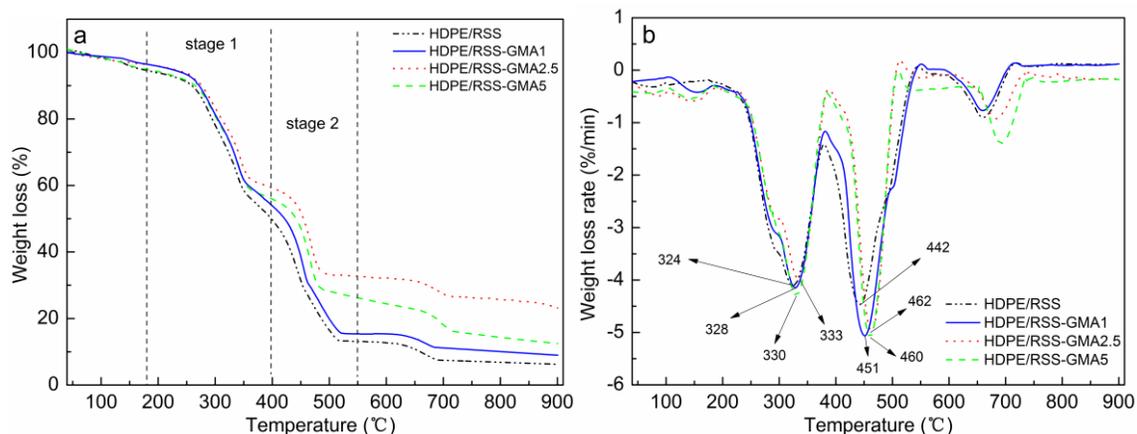


Fig. 4. TG and DTG curves of HDPE/RSS composites made of unmodified or GMA-modified RSS; a: TG, b: DTG

Figure 4(b) shows the DTG curves (the first derivative of weight loss as a function of temperature), which characterizes the decomposition rate of the different samples. The peak points are an indication of the maximum degradation rate at a corresponding temperature. There was noticeable improvement in thermal stability for the modified HDPE/RSS composites by grafting the RSS using GMA. The peak point in the first stage (T_{max1}) for the composites made of modified RSS by GMA with the concentration of 1%, 2.5%, and 5% were increased to 328, 333, and 330 °C from 324 °C, respectively. Similarly for the second stage, the peak point of the modified samples by GMA increased from 442 °C to 451, 462, and 460 °C, respectively. It can be concluded that the GMA grafting concentration of 2.5% has a superior function for improving the thermal stability of HDPE/RSS composites. In addition, the DTG curves of the samples modified by GMA illustrates one weak peak at 150 °C, which can be explained by the fact that some low-molecular polymers were polymerized through the self-initiation of carbon-carbon double bonds and epoxy groups in the GMA.

Table 2. Weight Loss Results for HDPE/RSS Composites

Composite	Weight Loss (%)		
	Stage 1 (180 to 400 °C)	Stage 2 (400 to 550 °C)	Residual (%)
HDPE/RSS	44.96	38.45	5.23
HDPE/RSS-GMA1	42.57	36.53	8.96
HDPE/RSS-GMA2.5	37.07	26.56	23.11
HDPE/RSS-GMA5	38.98	29.68	11.46

Dynamic Mechanical Analysis (DMA)

Figure 5 presents the effect of GMA concentration on the storage modulus (E') and damping parameter ($\tan \delta$) of the HDPE/RSS composites. In Fig. 5(a), it is evident that the general trends of the variation were almost similar for all samples. The E' value greatly dropped for all the composites mostly due to the thermal softening of the HDPE resin with increasing temperature. The E' values of the various composites at 150 °C became equal since the matrix melted completely. The E' value of the composite made of unmodified RSS was 4190 MPa at 40 °C, whereas the E' values of the composites made of GMA-modified RSS were significantly higher. This means that the interfacial bonding between the HDPE and the RSS was strengthened after being modified by GMA. When the external loads were placed on the samples, most of the stress could be transferred to the highly rigid RSS fiber through closely integrated interface. The $\tan \delta$ curves for the different HDPE/RSS composites made of unmodified or GMA-modified RSS are plotted in Fig. 5(b). The value of $\tan \delta$ was obtained from the loss modulus-to-storage modulus ratio and was used to describe the flexibility of the composites (Son *et al.* 2003). The $\tan \delta$ curves gradually increased with temperature, which was attributed to the promotion of thermal motion capacity for polymer chains. The key peak temperature and $\tan \delta$ value are given in Table 3. In combination with the data in Table 3, it is also important to note that there were two peaks for the untreated HDPE/RSS composite; the temperature and $\tan \delta$ for the first and second peaks were 144.9 °C, 0.380 and 171.9 °C, 0.393, respectively. This may be due to the inferior compatibility of the components in the matrix system. On the other hand, the HDPE/RSS composites made of GMA-modified RSS exhibited a shift in the first peak towards the higher temperature, while the second peak disappeared.

Specifically speaking, as listed in Table 3, the peak temperature and $\tan \delta$ of the first peaks were delayed to 152.9 °C and 0.375, 156.7 °C and 0.364, and 155.8 °C and 0.366, corresponding to GMA grafting concentrations of 1%, 2.5%, and 5%, respectively. A reasonable explanation for this could be that there was a positive effect on mutual entanglement, locking, and interaction between the rubber seed shell and the HDPE resin in the presence of GMA, resulting in the formation of a network structure as well as the promotion of interfacial adhesion. Stress energy dissipation occurred in the matrix and interface, and with stronger interfacial bonding, more stress energy could be effectively driven to dissipate to RSS particles.

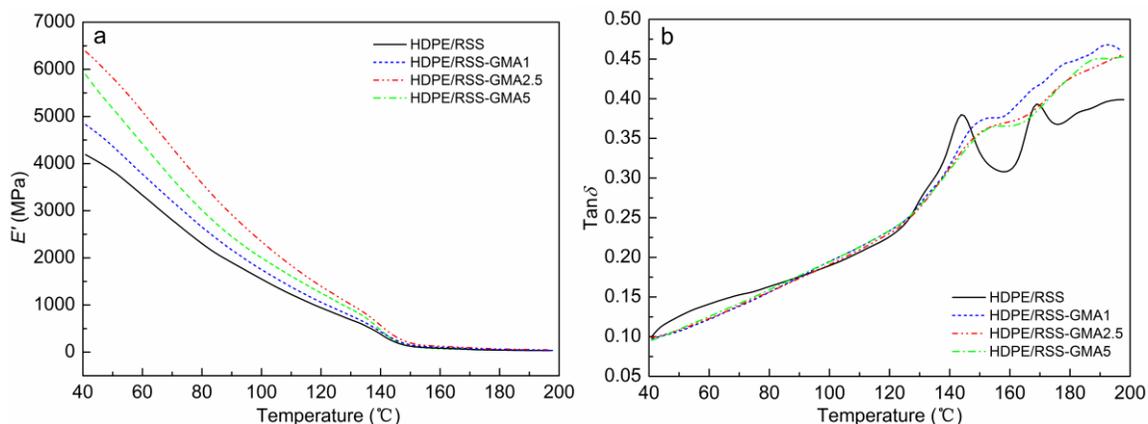


Fig. 5. DMA curves of HDPE/RSS composites made of unmodified or GMA-modified RSS; a: E' , b: $\tan \delta$

Table 3. Peak Temperature and $\tan \delta$ Values for HDPE/RSS Composites

Composite	Peak Temperature (°C) and $\tan \delta$ Value	
	First Peak	Second Peak
HDPE/RSS	144.9, 0.380	171.9, 0.393
HDPE/RSS-GMA1	152.9, 0.375	—
HDPE/RSS-GMA2.5	156.7, 0.364	—
HDPE/RSS-GMA5	155.8, 0.366	—

CONCLUSIONS

1. Glycidyl methacrylate (GMA) grafting of rubber seed shell (RSS) was beneficial for improvement in flexural and tensile properties of composite samples prepared with a high density polyethylene (HDPE) matrix. GMA grafting effectively decreased the water absorption rate of the composites and slightly increased the Vicat softening temperature (VST), but various concentrations of GMA showed no marked difference in VST.
2. GMA modification elevated the thermal stability of the HDPE/RSS composites during the initial thermal degradation. Meanwhile, composites with RSS modified by GMA grafting at a concentration of 2.5% had the lowest weight loss (37.07% and 26.56%, respectively) during the first and second thermal degradation stages.

3. The E' values of the GMA-modified composites at various GMA concentrations were noticeably higher than that of the untreated composite. There were two peaks of $\tan \delta$ for the untreated samples, and the modified samples exhibited a shift in transition peak toward higher temperatures; moreover, the second peak disappeared in the treated composites because of the positive effects of mutual entanglement, locking, and interaction between the rubber seed shell and the HDPE.

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