Miscibility, Thermal, and Mechanical Properties of Recycled Waste Tire Rubber-Modified Polystyrene Sustainable Composites

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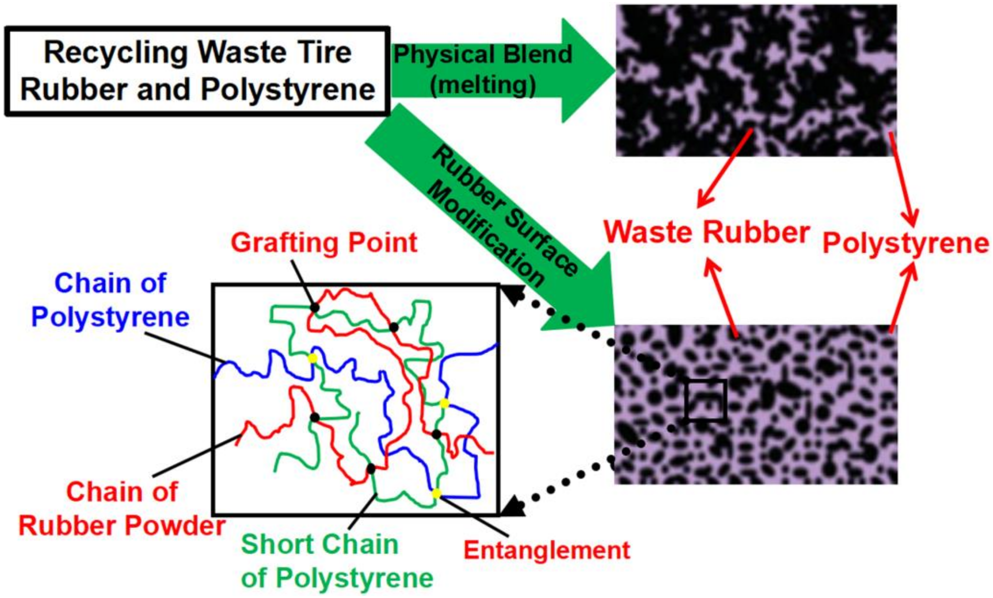
GRAPHICAL ABSTRACT



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Waste tires represent an important source of polymer waste. The ground tire rubber derived from waste tires is a recycled product that can be combined with polystyrene (PS) to produce high-performance PS and waste rubber composites. To improve composite material performance, surface grafting modification of waste tire rubber with styrene to enhance properties of PS composites as a novel approach was investigated. The surface morphology and structure of polystyrene grafted waste tire rubber powder *via* a conventional free radical polymerization were confirmed successfully using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy analyses in addition to the Fourier-transform infrared spectroscopy (FTIR). The comparative mechanical and thermal property analysis of PS sustainable composites with recycling waste tire rubber powder with and without surface grafting modifications indicated an approximate 4-fold increase in the impact strength of polystyrene grafted waste tire rubber reinforced PS sustainable composites in addition to enhanced interfacial miscibility. The development of sustainable composite materials from recycled waste tire provides a novel avenue to achieve close-loop polymer recycling, which is of significance in the development of the circular economy and an environmentally friendly society.

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Keywords: Waste tires; Recycling; Sustainable composites; Mechanical properties

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INTRODUCTION

The challenging issue faced by modern society is the constant increase in waste. In particular, approximately 290 million per year of rubber waste is produced in the United States resulting from the rapid increase in utilization of vehicles and end-of-life tires (Wang *et al.* 2020; Zhang *et al.* 2023). However, these rubber waste products are non-biodegradable (Vilela *et al.* 2018). Approximately 2 billion waste tires are accumulated each year in the United States, and these waste tires in their long-term storage easily cause health and environmental issues if they are not properly managed (Kocatürk *et al.* 2024). Therefore, it is a challenge to deal with these large amounts of waste. Converting these ground tires into rubber powders *via* cryogenic grinding technology and then blending with polymer materials to produce high-performance sustainable composites is one of the sustainable approaches to deal with these discarded tires.

As one of the most widely used thermoplastics, polystyrene (PS) has unique merits of high stiffness, good transparency, and excellent dielectric properties (Zhang *et al.* 2024). It primarily works as thermal and sound insulating foams and packaging. However, the end-of-life PS packaging contributes to a large amount of waste, which accounts for 8 wt% of total plastic waste. Therefore, it is a tough question in terms of recycling PS, as the conventional incineration of PS plastic waste causes significant amounts of carbon dioxide emissions. In addition to the PS recycling issue, its intrinsic low impact strength restricts wide applications. Therefore, the design of high-performance recycling waste tire - strengthened PS composites to extend its lifetime has attracted significant interest to reduce the PS waste production and subsequently achieve carbon emission reduction and carbon neutrality. However, the simple compounding of waste tire rubber and PS causes considerable deterioration in mechanical properties attributed to their low miscibility and poor interfacial adhesion (Liang *et al.* 2019; El-Nemr *et al.* 2018; Luna *et al.* 2019).

Using a compatibilizer and rubber surface activation are two primary approaches that have been employed for tailoring miscibility and interfacial adhesion. For instance, the compatibilizer of styrene-butadiene-styrene (SBS) could improve the impact strength of waste tire rubber and physical recycling of expanded PS (EPS) sustainable composites (Andrade Junior and Saron 2023). However, the SBS compatibilizer loading had to reach an approximate 30 wt% for a clearly enhanced impact strength of EPS and waste tire rubber composites. Therefore, waste rubber surface activation sounds like a promising alternative method for improving mechanical properties of PS and waste tire rubber composites. Surface activation strategies of waste tire rubber by radiation (Scuracchio *et al.* 2007), acid oxidation (Mpho Phiri *et al.* 2021), and gas etching (Tan *et al.* 2009) as typically physical and chemical methods have been investigated. The resulting surface activating waste tire rubber composite materials improved mechanical properties. In addition, a controlled radical polymerization makes it possible to achieve controlled grafting functional polymer chains on the surface of waste tire rubber. For instance, surface grafting modification of waste tire rubber by PS mediated *via* an atom transfer radical polymerization method could selectively tailor the rubber surface, but mechanical properties of resulting composites were not known (Coiai *et al.* 2006). The complex synthetic protocols also restrict its wide application. Surface modification of ethylene propylene diene monomer tire rubber with a given molecular weight of PS *via* a thiol-ene click reaction method was further studied, and the resulting composite material had an enhanced miscibility and interfacial adhesion according to the analysis of combined dynamic mechanical analysis and scanning electron microscopy (Liang *et al.* 2019). However, the complex synthetic protocols of thiol-end PS were the major restrictions. Thus, it is highly desirable to develop a simple and straightforward method to conduct surface grafting modification on waste tire rubber. For instance, surface grafting modifications *via* a conventional free radical polymerization were conducted with diverse monomers, *e.g.*, allylamine (Lee *et al.* 2009), methacrylic acid (Shanmugharaj *et al.* 2006), glycidyl methacrylate (Kim *et al.* 2000), acrylonitrile (Abdel-Bary *et al.* 1998), and acrylic acid (Kocevski *et al.* 2012). However, to the authors' knowledge, there is little information available in literature about graft modification of waste tire rubbers by bulk polymerization of styrene to tailor mechanical properties of PS and polystyrene grafted tire rubber composite materials.

In this work, the polystyrene grafted waste tire rubber was prepared *via* a conventional free radical polymerization, and the surface-activated waste tire rubber in terms of composition and surface morphology was then investigated. The composite materials from polystyrene grafted waste tire rubber and PS were further prepared *via* melting compounding, and the comparative miscibility, morphology, and mechanical and thermal properties of resulting composite materials were studied. Future work is expected to develop waste tire rubber and cellulose or nanocellulose hybrids carbon particles in battery and energy field applications.

EXPERIMENTAL

Materials

Styrene received from Tianjin Guangcheng Chemical Co. was purified by washing with sodium hydroxide solutions (5 wt%) and drying with sodium sulfate before use. Dibenzoyl peroxide (DPO) was ordered from Shanghai Shanpu Chemical Co. and further purified *via* recrystallization from methanol. The PS resin was purchased from Zhanjiang Xinzhongmei Chemical Co. Waste tire rubber (average particle size of 134.8 μm) was ordered from Jilin Huadian, China and purified by washing with acetone, sodium hydroxide solutions (5 wt%), and hydrochloric acid (10 wt%). Other chemicals were used without further purification.

Surface Grafting Polymerization of Waste Tire Rubber

The DPO initiators were dissolved in styrene monomers and then added into the reactor under nitrogen atmosphere. Waste tire rubber was added subsequently under mechanical stirring. The suspensions were then polymerized at 80 ºC for 15 to 20 h as shown in Figs. 1 and 2a. The polymerized product was diluted with chloroform and then precipitated with methanol followed by further purification with mixture solvents of butanone and acetone by a Soxhlet apparatus for 36 h to remove unreacted monomers and waste tire rubber. The insoluble residue as polystyrene grafting waste tire rubber was obtained after drying to a constant weight.

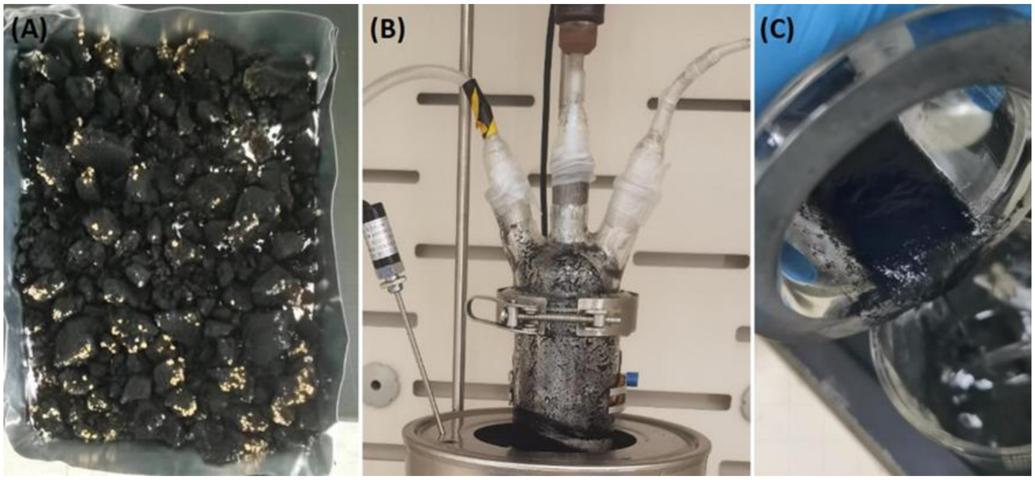

Fig. 1. Waste tire rubber (A), polymerization reactor (B), and polystyrene grafting waste tire rubber (C)

Melting compounding of waste tire rubber and ps composites

The PS and waste tire rubber blends were melt-extruded *via* a single screw extruder (R77F-NA50-112B5, Wuhan Weier Plastic Machinery Co.) between 160 and 190 °C. The extruded samples were then granulated for compounding tensile and impact standard samples using an injection molding machine (HF650II, Haitian Plastic Machinery Co.) as shown in Fig. 2b.

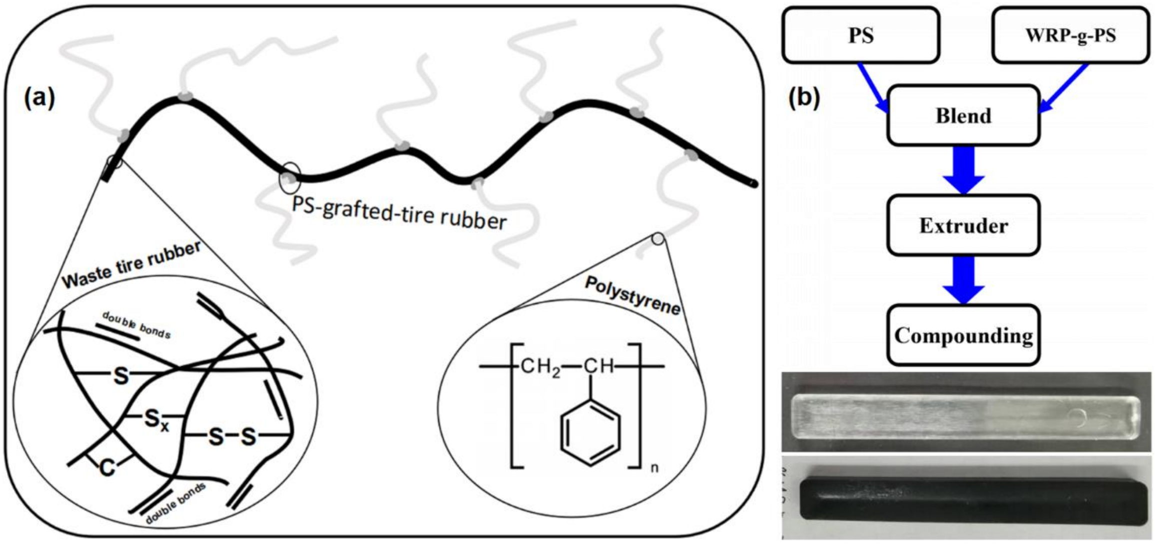

Fig. 2. Scheme of reaction on styrene grafting waste tire rubber (a) and PS/styrene grafting waste tire rubber composites *via* melting compounding (b)

Characterization

Thermal properties of waste tire rubber/PS and polystyrene grafted waste tire rubber/PS composites were measured using a DSC-60 differential scanning calorimeter (Shimadzu, Japan) under nitrogen atmosphere. The heating rate was at 10 °C/min, and the temperature range was from -100 to 150 °C. Waste tire rubber, polystyrene grafted waste tire rubber, PS, and waste tire rubber/PS and polystyrene grafted waste rubber/PS composites were sputtered with gold before measurement *via* SEM (Nova 400Nano, Philips Electron Optics). The chemical structure of PS and styrene grafting waste tire rubber was characterized *via* FTIR spectra (a Nicolet-5700 spectrometer, Thermo Fisher Scientific) with the wavenumbers from 3600 to 600 cm^{-1}. The surface element composition of waste tire rubber and polystyrene grafted waste tire rubber was determined with an energy dispersive X-ray (EDX) spectrometer (Ie350 Penta FET x-3, Britain Oxford Corporation). Impact strength of waste tire rubber/PS and polystyrene grafted waste rubber/PS composites was measured with an impact machine (ZWJ-0350, Dongguan Zhenglan Precision Instrumental Co., Dongguan, China) according to GB/T 1843 (2008). The tensile strength of waste tire rubber/PS and polystyrene grafted waste rubber/PS composites was tested using an Instron machine (UTM 6503) at room temperature according to GB/T 1040 (2006). Dynamic mechanical thermal analysis of pure PS, waste tire rubber/PS and polystyrene grafted waste tire rubber/PS composites was tested *via* dynamic mechanical analyzer (Q800, TA Instruments Inc, USA). The heating rate was at 3 °C/min, and the temperature range was from -80 to 150 °C.

RESULTS AND DISCUSSION

Surface Morphology and Composition of Waste Tire Rubber and Polystyrene Grafted Waste Tire Rubber

The surface morphology of the waste tire rubber before and after grafting modification is shown in Fig. 3. The surface morphology and particle size of polystyrene grafted waste tire rubber had undergone an obvious change as compared to waste tire rubber. The unsmooth surface and irregular aggregation structure of waste tire rubber particle could be observed in Fig. 3(a). Some pores were also clearly apparent. In contrast, the surface of polystyrene grafted waste tire rubber became smooth, and no obvious cracks could be observed in Fig. 3(b), attributed to the grafting polystyrene chains encapsulated in pores of waste tire rubber. In addition, the polystyrene layers covering waste tire rubber surface could be observed. These results indicated that polystyrene grafted waste tire rubber was successfully prepared.

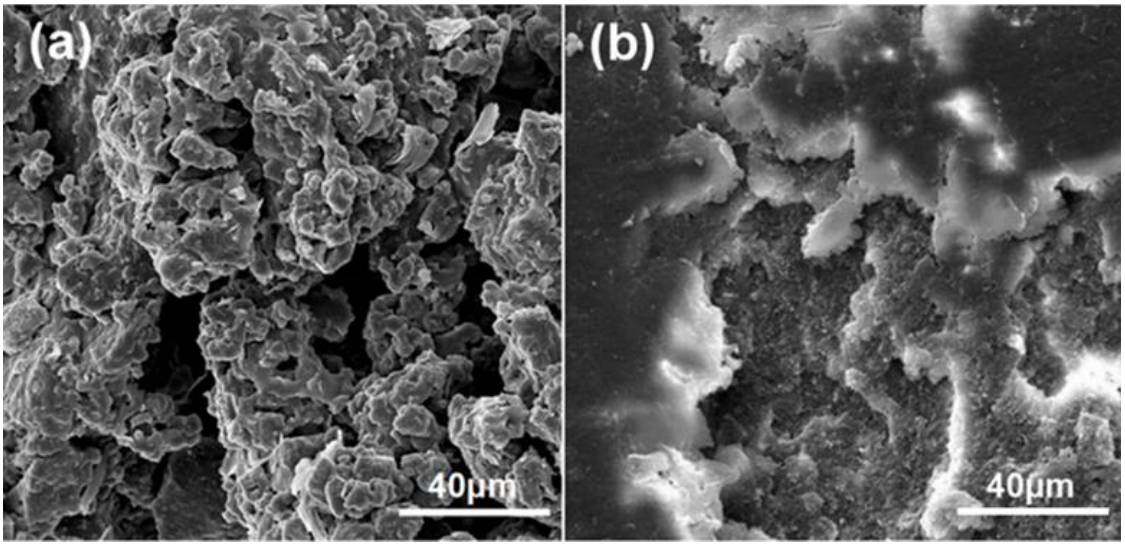

Fig. 3. SEM images of waste tire rubber (a) and polystyrene grafted waste tire rubber (b)

The surface elemental composition of waste tire rubber before and after modification is shown in Fig. 4 and Table 1.

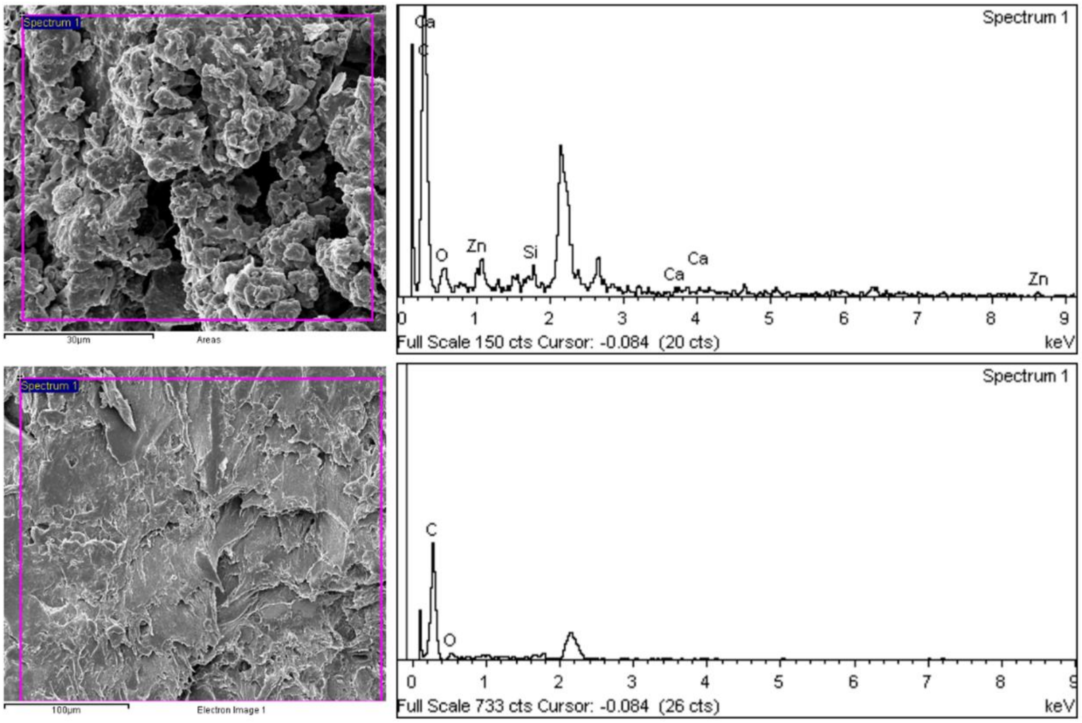

Fig. 4. Energy-dispersive X-ray spectroscopy of waste tire rubber and polystyrene grafted waste tire rubber

Table 1. Element Content of Waste Tire Rubber and Polystyrene Grafted Waste Tire Rubber

Element	Waste Tire Rubber (%)	Polystyrene Grafted Tire Rubber (%)
C	76.03	90.53
O	17.83	9.47
Al	1.25	-
Si	2.06	-
Ca	2.01	-
Zn	0.76	-

The variations in element composition and content are the direct reflection of surface properties of waste tire rubber. Several elements, namely carbon, nitrogen, silicone, calcium, and zinc, were observed on the energy-dispersive X-ray spectroscopy (EDXS) of waste tire rubber. Some additives composed of these inorganic elements, such as carbon blacks, antioxidants, and reinforcing fillers, were added in the melting processing of rubber tire materials *via* a two-roll milling machine. However, only carbon and oxygen elements were observed in the polystyrene grafted waste tire rubber, and silicone, calcium, and zinc elements disappeared, as the waste tire rubber surface was covered by grafted polystyrene chains. Therefore, EDXS results indicated that the surface of waste tire rubber was encapsulated by polystyrene chains after surface grafting modification. FTIR spectra was further studied for the confirmation of styrene grafting waste tire rubber successfully. According to Fig. S1, characteristic peaks of PS were clearly observed as follows: the peak at 3028 cm^{-1} assigned to C-H stretching vibration of benzene ring, and peaks at 758 cm^{-1} attributed to C-H vibration of monosubstituted benzene. Therefore, the FTIR spectra further supports the PS chains grafting on the surface of waste tire rubber successfully.

Mechanical, Miscibility, and Microstructure of Waste Tire Rubber/PS Composites

The mechanical properties of waste tire rubber/PS and polystyrene grafted waste tire rubber/PS composites are shown in Table 2. The impact strengths of polystyrene grafted waste tire rubber/PS composites were much higher than those of waste tire rubber/PS composites. The rubber contents increased from 10 to 25 wt% because the grafting polystyrene chains on the tire rubber surface promoted its dispersion in the PS matrix. The grafting also enhanced interfacial adhesion and miscibility between waste tire rubber and PS, thereby facilitating stress transfer from PS matrix to tire rubber particles. In addition, as the polystyrene grafted tire rubber loading increased from 10 to 25 wt%, the increased impact strengths of modified tire rubber/PS composites were also observed, which can be attributed to the better stress transfer from PS to elastomer rubber particles. When the loading of polystyrene grafted waste tire rubber was at 25 wt%, the impact strength of modified waste tire rubber/PS composites had an approximate 4-fold increase compared to pure PS resin. The tensile strengths of waste tire rubber/PS and polystyrene grafted waste tire rubber composites almost had a similar value as the loading of tire rubber at 10 wt%. However, as the tire rubber loading increased from 15 to 25 wt%, the tensile strengths of polystyrene grafted waste rubber/PS composites obviously increased compared to waste tire rubber/PS composites. These results indicated that the waste tire rubber surface grafting modification improved its dispersion in the PS matrix and enhanced interfacial interaction between PS matrix and tire rubber particles. However, when the loading of waste tire rubber was at 25 wt%, the tensile strength of polystyrene grafted tire rubber/PS composites exhibited an obvious decrease as compared to pure PS. Because the primary goal of this work is to tailor the impact strength of PS and waste tire rubber composites and meanwhile maintain a decent tensile strength, polystyrene composites with grafted waste tire rubber loading at 25 wt% had optimized mechanical properties.

Table 2. Mechanical Properties of PS/Waste Tire Rubber Composites

Tire Rubber Loading (%)	Impact Strength (KJ/m^2)	Tensile Strength (MPa)
0^a	5.0	39.4
10^b	7.5	25.1
10^c	8.2	24.2
15^b	6.5	19.1
15^c	12.2	24.2
20^b	10.1	18.9
20^c	10.5	27.0
25^b	7.3	20.1
25^c	21.6	25.6

^{a}: pure PS; ^{b}: waste tire rubber/PS composites; ^{c}: polystyrene grafted waste tire rubber/PS composites

To further understand the tire rubber surface grafting modification on mechanical properties of PS composites, miscibility and microstructure of composite materials were characterized by DSC and SEM. The DSC curves of waste tire rubber/PS and polystyrene grafted waste tire rubber/PS composites and pure PS resin are shown in Fig. 5.

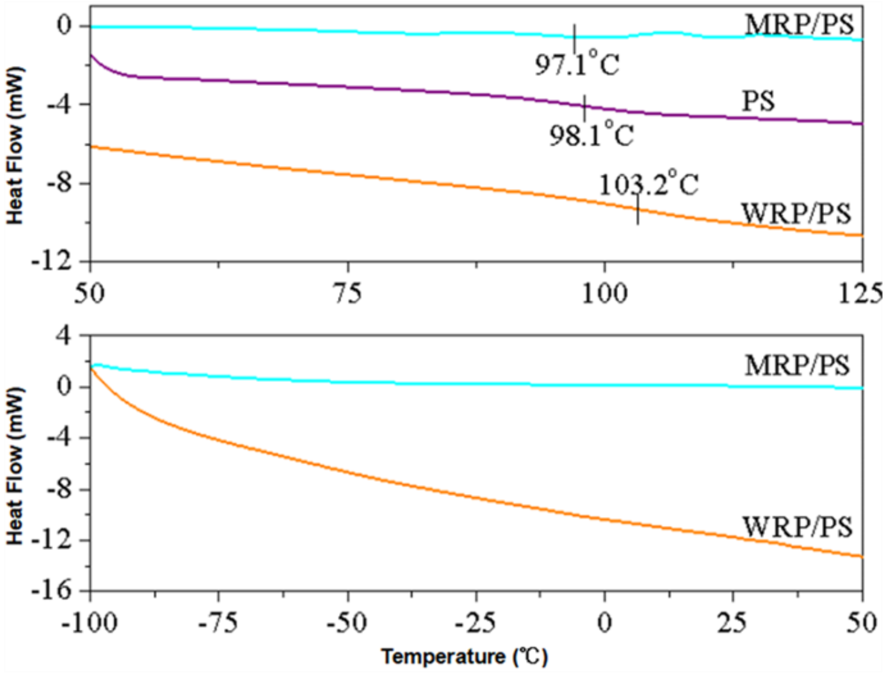

Fig. 5. DSC curves of pure PS resin, waste tire rubber (WRP)/PS composites, and polystyrene grafted tire rubber (MRP)/PS composites

The glass transition temperature (T_g) of pure PS resin was at 98.1 ºC, while the T_g of PS component in waste tire rubber/PS composites had a shift to a high temperature at 103.2 ºC. The lesser miscibility of waste tire rubber and PS matrix and weak interfacial interaction near the boundary regions between waste tire rubber and PS moved T_g of PS component toward a high temperature. In contrast, T_g of PS component in polystyrene grafted waste tire rubber/PS composites resulted in a shift of 6 °C to a low temperature compared to that of waste tire rubber/PS composites. For both waste tire rubber/PS and polystyrene grafted waste tire rubber/PS composites, T_g values were not observed, which was probably due to the sensitivity issues of conventional DSC instrument. A similar phenomenon was also reported in the literature (Naskar *et al.* 2001). The T_g shifts in tire rubber/PS composites could be explained as grafting polystyrene chains on the waste tire rubber surface acted as an interfacial compatibilizer promoted a strong interfacial interaction between PS and waste tire rubber and enhanced the interfacial miscibility. The improved miscibility was further supported by the data of dynamic mechanical thermal analysis (shown in Fig. S2 and Table S1 in the Appendix). Therefore, DSC results could give evidence of a strong interfacial interaction and enhanced miscibility in polystyrene grafted waste tire rubber/PS composites, resulting in the enhanced mechanical properties above.

The SEM images of pure PS, 25 wt% waste tire rubber/PS and polystyrene grafted waste tire rubber/PS composites are shown in Fig. 6 and Fig. S3. Pure PS displayed the fragile fractured surface associated with its low impact strength. For waste tire rubber/PS composites in Fig. 6(a), large holes could be clearly observed attributing to the rubber particles pulled out from the PS matrix. The fragile fractured surface was clearly observed. In contrast, a relatively smooth fractured surface and no obvious holes were observed in polystyrene grafted waste tire rubber/PS composites in Fig. 6(b). Waste tire rubber particles also had an improved interfacial adhesion with PS matrix after surface grafting modification. The ductile fractured surface was observed. These results indicated that the surface grafting modification enhanced the interfacial adhesion and promoted the waste tire rubber particles in the PS matrix, resulting in the enhanced mechanical properties of polystyrene grafted waste tire rubber/PS composites.

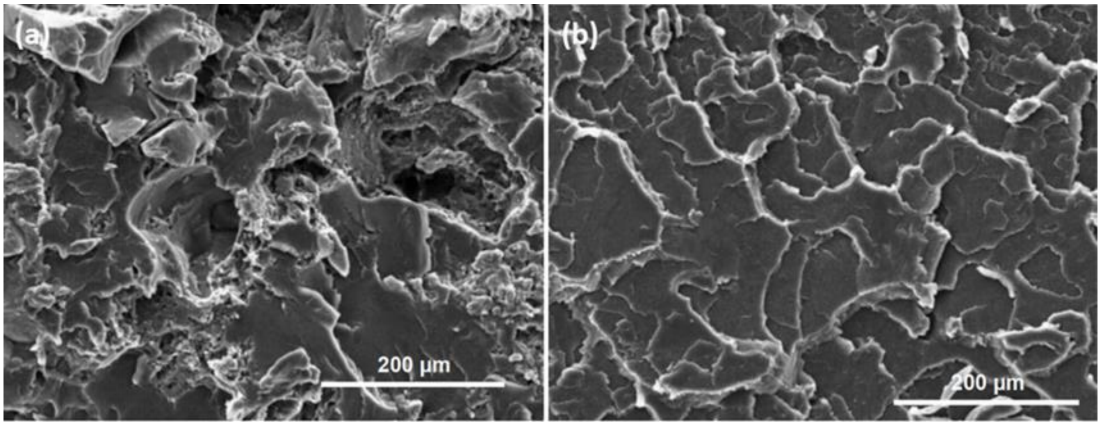

Fig. 6. SEM images of 25 wt% waste tire rubber/PS composites (a) and 25 wt% polystyrene grafted waste tire rubber/PS composites (b)

CONCLUSIONS

1. Polystyrene grafting of waste tire rubber was successfully prepared *via* conventional free radical polymerization.
2. The grafted polystyrene layers on the surface of waste tire rubber particles were confirmed *via* scanning electron microscope (SEM) and energy-dispersive X-ray spectrometry (EDXS) analyses.
3. The polystyrene grafted waste tire rubber and polystyrene composites exhibited improved impact strength compared to that of waste tire rubber and polystyrene composites. When the loading of polystyrene grafted waste tire rubber was 25 wt%, the polystyrene composites exhibited optimized comprehensive mechanical properties.
4. Waste tire rubber surface grafting polystyrene chains promoted its miscibility and interfacial interaction with the polystyrene matrix, which was confirmed through SEM and differential scanning calorimetry (DSC) analyses.

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APPENDIX

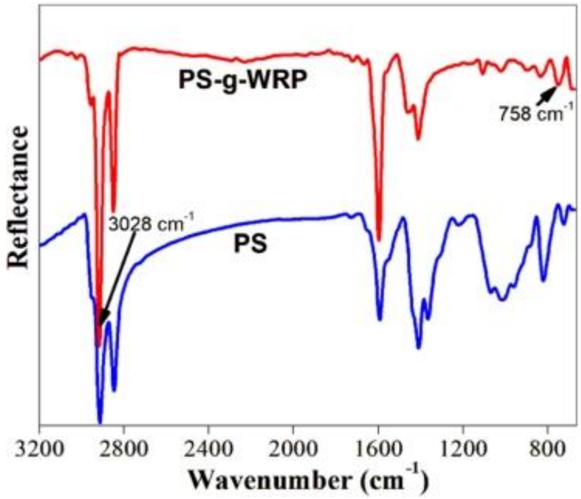

Fig. S1. FTIR spectra of PS and styrene grafting waste tire rubbers

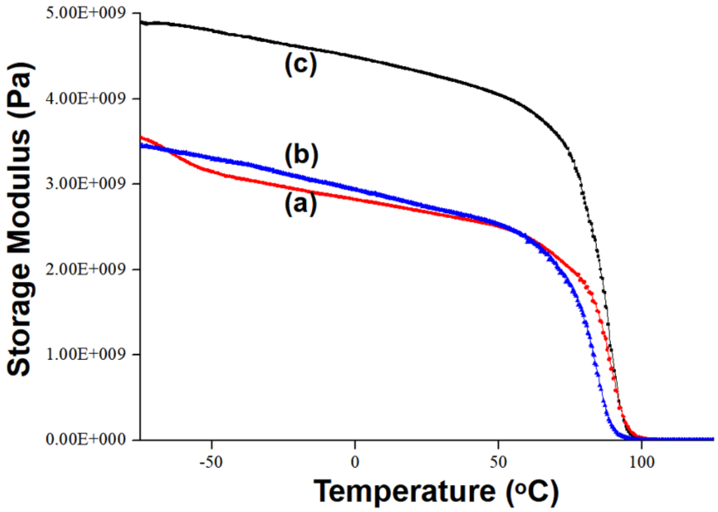

Fig. S2. DMA curves of pure PS (a), PS/25 wt% waste tire rubber composites (b), and PS/25 wt% modified waste tire rubber composites (c)

Table S1. Glass Transition Temperatures of Pure PS and PS Composites with 25 wt% Waste Tire Rubbers

Samples	T_g of PS Segments (°C)	T_g of Waste Tire Rubbers (°C)
PS*	100.7	-
PS/WRP Composites**	102.5	-61.6
PS/MRP Composites***	97.9	NA

Tips: *: pure PS; **: PS/25 wt% waste tire rubber composites; ***: PS/25 wt% modified waste tire rubber composites

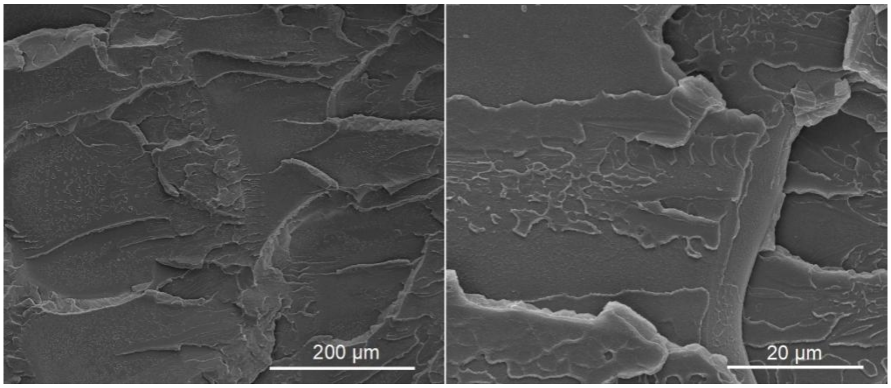

Fig. S3. SEM images of pure PS