



Fabrication of magnetic rubber composites by recycling waste rubber powders via a microwave-assisted *in situ* surface modification and semi-devulcanization process



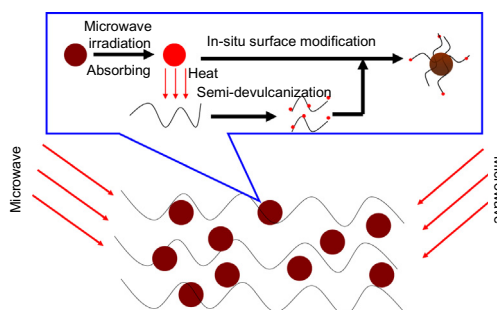
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HIGHLIGHTS

- Magnetic rubber was made from Barium ferrite (BaM) and waste rubber powders (WRPs).
- Surface modification of BaM and activation of WRPs were simultaneously achieved.
- Microwave devulcanization of WRPs was enhanced after adding BaM powders.
- A novel route was designed for fabricating low cost flexible rubber magnets.

GRAPHICAL ABSTRACT



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ABSTRACT

To effectively reuse waste rubber and reduce secondary pollution, a microwave-assisted *in situ* surface modification and semi-devulcanization route has been developed to fabricate magnetic rubber composites. The composites use waste rubber powders and ultrafine barium ferrite powders as starting materials without additional additives. Taking advantage of the microwave absorption capabilities of the barium ferrite powders and filling materials that exist in waste rubber, both the semi-devulcanization of the waste rubber powder and surface modification of barium ferrite powder have been simultaneously achieved under microwave irradiation. Thus, the phase compatibilities between barium ferrite and the rubber matrix are effectively improved, resulting in a low-cost fabrication of flexible magnetic composites with good ferromagnetic properties. The presented microwave assisted technique may also offer a novel path to design and produce organic–inorganic nano-composites.

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1. Introduction

Over the past several decades, many attempts have been made to recycle waste rubber to produce an additional rubber resource or to avoid environmental pollution [1–3]. Conventional methods, such as burning and burying rubber waste underground, release a large amount of carbon oxide and other poisonous substances that

cause climate change and serious pollution [1–3]. Therefore, the focus has shifted from simple passive treatments to reusing waste rubber with less secondary pollution. Improving the comprehensive performance of the reused waste rubber blends or composites presents another challenge because it is necessary to destruct the three-dimensional network structure of the vulcanized rubber and then recover the thermoplastic behavior through well-designed physical or chemical treatments [4–17]. Although chemical devulcanization can effectively promote the devulcanization of waste rubber and improve the performance of the final products, a

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large amount of dumped wastewater containing poisonous chemical agents constrains its further application [1,2].

Presently, recycling waste rubber by utilizing powder technologies is widely regarded as an attractive recycling method [7–11]. The resulting waste rubber powders (WRPs) have been used to fabricate industrial flooring, pathways, golf ranges, and so on [1–3,9,10,15–17]. Nevertheless, the cross-linked three-dimensional network of the WRPs makes them hard to process, limiting the performance of the final products. From this perspective, a partial or complete devulcanization of the WRPs is often required before blending with additional raw rubber or in composite materials [13–19]. Many efforts concerning the reuse of WRPs and establishing high performance have focused on the chemical or physical modification of the WRPs to improve their mechanical properties [7,9–20]. Another difficulty in reusing WRPs in polymer blends is their complex compositions, which depend on source and service history of the WRPs. This causes the mechanical properties of the WRPs composites to be unstable or unreliable. One feasible way to overcome these problems is to fabricate functional organic–inorganic composite materials using WRPs as the polymer matrix by proper processing techniques [1–3]. However, to fabricate functional composites that have a relatively fine and reliable performance, the devulcanization of WRPs must be accomplished by utilizing well-designed techniques. The phase compatibilities between the rubber matrix and inorganic filling materials must be refined through the surface modification of the inorganic filling materials.

Therefore, a general route to solve both obstacles (the devulcanization of WRPs and the surface modification of inorganic filling materials) to develop functional composites using WRPs as the starting materials is highly desirable.

In this paper, a well-designed microwave-assisted *in situ* surface modification and semi-devulcanization process is presented to easily fabricate magnetic rubber composites using WRPs as the starting materials, as illustrated in Fig. 1; this process is briefly described as follows: the target magnetic composites consist of WRPs as the polymer matrix and barium ferrite (BaM) ultrafine powder as the magnetic filling material. Compared with the conventional microwave de-vulcanization of rubber [7,20–26], the addition of BaM ultrafine powder, an excellent microwave absorber [27,28], greatly enhance the response of the waste rubber to microwave irradiation with a combination of filling materials, such as carbon black, that previously existed in the waste rubber [26,29,30]. BaM ultrafine powders with carbon black act as multi-component microwave absorbers [29,30]. Taking advantage of their microwave absorbing abilities, the microwave energy is

strongly absorbed and rapidly converted it into heat energy, which contributes to the formation of local hot micro-domains around the surface layers of the microwave absorbers. Within or around the local hot micro-domains, both the main chains and poly-sulfur bonds of the WRPs tend to break down, while there are few influences on the local domains that are farther away from the surface of the microwave absorbers. Meanwhile, the activated broken main chains or poly-sulfur bonds adhere to the surface of the microwave absorbers due to the high surface energies of the ultrafine particles. Consequently, both the semi-devulcanization of the WRPs and the *in situ* surface modification of the ultrafine BaM powders could be achieved at the same time. After further thermal vulcanization, the magnetic rubber composites could be easily obtained without the requirement of additional additives.

By focusing on the fabrication of magnetic rubber composites via the designed microwave-assisted semi-devulcanization and an *in situ* surface modification route, both the devulcanization of WRPs and the *in situ* surface of BaM particles have been investigated. Furthermore, the relationships between the microstructure and properties of the as-synthesized magnetic rubber composites have been discussed.

2. Material and methods

2.1. Fabrication of magnetic rubber composites

Ultrafine BaM powders with particle sizes ranging from 0.2 to 1.0 μm were synthesized via a conventional chemical coprecipitation method. The waste rubber powders supplied by Jiangsu Honglei Co., P. R. China, consisted of 100-mesh ground rubber that was obtained from the treads and sidewalls of passenger tires. The major components of the tire rubber polymers are natural (polyisoprene, NR), styrene–butadiene copolymer (SBR), polybutadiene (BR). The additives are carbon black as well as the other filling materials. The BaM powders and WRPs were mixed homogeneously using a high speed mixing machine for approximately 1 h. The BaM contents in the mixtures ranged from 0 wt% to 50 wt%. A total mixture weight of approximately 100 g was then placed in a round ceramic dish and irradiated for various times in a microwave system (LWMC-205) at a constant output power of 400 W and frequency of 2.45 GHz. After the microwave treatment, the mixtures were added into a round 15-cm diameter steel mold and thermally vulcanized at 145 $^{\circ}\text{C}$ for 10 min to obtain the magnetic rubber composites.

2.2. Characterization of the magnetic rubber composites

The semi-devulcanization of the WRPs by microwave activation was investigated by electron spin resonance spectroscopy (ESR, Bruker, A300-10/12), attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR, Varian, Cary 670) and a laser Raman spectrometer (L-RS, Thermo Scientific, DXR). A transmission electronic microscope (TEM, Philips, Tecnai) was used to study the surface morphology of the BaM particles after microwave irradiation. The cross-linking density of the as-fabricated magnetic rubber composites was characterized by the swelling index, which was determined according the toluene-swelling test. The test pieces were immersed in toluene at a room temperature for 72 h to achieve swelling equilibrium. Then, the test piece were removed from the toluene, wiped with tissue paper to remove excess toluene from the surface, and weighed; the weight was referred to as the swollen weight. The swelling index was calculated as the ratio of swollen weight to original weight. The microstructure and phase distribution were characterized using a field emission scanning electronic microscope (FE-SEM, Hitachi, S-4800).

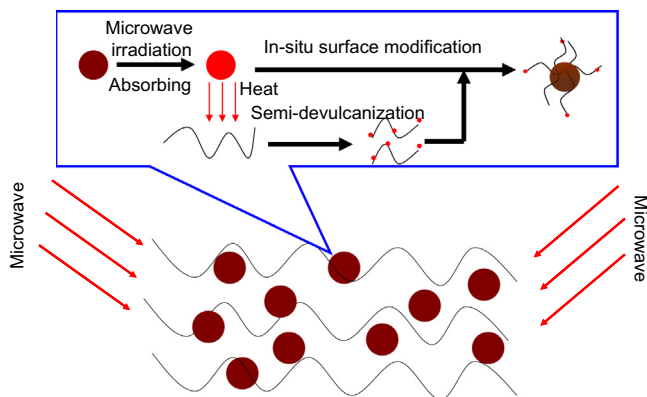


Fig. 1. Schematic illustration of fabrication of the magnetic rubber based on barium ferrite and waste rubber via a microwave-assisted *in situ* surface modification and semi-devulcanization process.

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