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Controllable synthesis of SiC wrapped LDHs to reinforce microwave absorption and exothermic properties of styrene-butadiene-styrene (SBS) polymer modified asphalt

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Abstract

Combination of styrene-butadiene-styrene (SBS) polymer and layered double hydroxides (LDHs) has commonly applied in asphalt to improve anti-ultraviolet aging performance, but it can not afford the reparation of cracks caused by aging of asphalt. Also, few researches focused on the structure and performance changes during synthesis of LDHs. Based on self-healing performance of asphalt, the produced cracks can be repaired by microwave heating. Therefore, the controllable synthesis of traditional microwave absorber SiC to wrap LDHs (SwL) at different temperatures was studied in this paper, and the effects of different SBS/SwL combination on the microwave absorption and exothermic properties of modified asphalt were studied. The SwL was firstly synthesized at 200 °C, 350 °C, 500 °C and 650 °C. Then morphology, phase composition, chemical structure of different SwL were evaluated. Finally, based on different combination of SBS and SwL, the effects of SBS/SwL on the microwave absorption in the frequency band from 1 GHz to 18 GHz and exothermic characteristics at 2.45 GHz of the modified asphalt were analyzed. The results show that SwL can be synthesized successfully, and the processing temperature significantly affects the structure of SwL. The SBS/SwL-200 modified asphalt has the best microwave absorption and exothermic properties. When used in self-healing asphalt, it is more advantageous to choose 200 °C as the synthesis temperature of SwL.

1. Introduction

Styrene-butadiene-styrene (SBS) polymer has been widely used as asphalt modifier and enables it to form a spatial three-dimensional network structure with the asphalt, thereby effectively improving the tensile, elasticity, crack and rutting resistance of the asphalt [1, 2]. However, due to ultraviolet irradiation, SBS polymer modified asphalt may face the problem of aging [3]. Thus, scholars tried to add other additives to synergize with SBS polymer to improve the anti-ultraviolet aging performance of asphalt, like Layered double hydroxides (LDHs) [4] and its modifiers [5, 6].

Li investigated the SBS polymer and sodium stearate modified LDHs to enhance the aging resistance of asphalt [5]. Xu added the SBS polymer and organic anions intercalated LDHs in asphalt, results showed that the compatibility and ultraviolet aging resistance were improved [7]. But former researches ignored that although LDHs and its modifiers could strengthen UV reflection ability with SBS polymer in modified asphalt, but they could not contribute to repair produced cracks caused by aging of asphalt. In addition, there are few studies
about the effects of temperature on the properties of LDHs, and the impact of performance changes on the SBS polymer modified asphalt.

Based on the self-healing properties of asphalt [8], if asphalt materials are given a enough period under no traffic load during the intermittent period, the racks of asphalt can be closed by thermodynamic motion, like wetting and diffusion [9]. Such phenomenon is recognized as the self-healing of asphalt materials [10]. In the natural state, this healing process is very slow with worse effect [11]. If the temperature of the asphalt can be increased to a higher degree, the process of reparation will be greatly improved, and microwave heating has been proposed to be an innovative way of such acceleration methods [12].

Conventional heating methods are the transformations of heat to the surface of a material by radiation, convection or conduction [13, 14]. Microwave heating is fundamentally different from conventional heating methods, which is an energy conversion phenomenon that occurs through molecular, atomic or ionic interactions with an electromagnetic field [15, 16]. Some studies have evaluated the difference between microwave radiation and conventional heating as well as the effects of specific microwave and non-thermal microwave effects [14, 17]. Microwave heating generally involves the process by which microwave energy is dissipated or absorbed by a heated sample to generate heat [18, 19]. For example, conduction loss, dielectric (polarization) loss, and magnetic loss are due to interactions between electromagnetic waves and matter, which depends on electromagnetic field characteristics [20, 21]. Silicon carbide (SiC) occupies an important position in the field of absorbing materials with its unique stability under extreme conditions, light weight, low density and high frequency absorbing characteristics [22]. Additionally, SiC is a super absorbent semiconductor material that acts as an indirect heating medium for microwave-transmissive materials in microwave fields [23]. Microwave radiation can initiate the flow of electrons in SiC, thereby performing resistance heating, so SiC can strongly absorb microwaves and be heated up rapidly [24].

Thus, this paper investigated the controllable synthesis of traditional microwave absorber SiC to wrap LDHs (SwL) at different temperatures. Then morphology, phase composition, chemical structure of different SwL were evaluated. Finally, based on different combination of SBS and SwL, the effects of SBS/SwL on the microwave absorption and exothermic properties of modified asphalt were studied. Since the highest mixing temperature of SBS polymer modified asphalt mixture does not exceed 200 °C, the minimum processing temperature of SwL in this paper was 200 °C. In addition, according to the research of Kong [25], after the LDHs was heated, the structure significantly changed at 350 °C, 500 °C and 650 °C. Thus, synthesized SwL products were firstly processed at 200 °C (SwL-200), 350 °C (SwL-350), 500 °C (SwL-500) and 650 °C (SwL-650). The surface morphology, phase composition and chemical structure of obtained SwL were studied by Field-emission Scan Electronic Microscope (FE-SEM), x-ray Diffraction (XRD) and Fourier Transform Infrared spectrometer (FTIR). Subsequently, four types of SBS polymer modified asphalt were prepared by mixing the SBS polymer modified asphalt with SwL. Microwave absorption properties of SBS/SwL modified asphalt were obtained through a microwave vector network analyzer system. Exothermic properties were detected by Forward Looking Infrared Radiometer (FLIR) Systems.

2. Materials and experimental methods

2.1. Materials

Used chemicals in the synthesis were of analytical grade and treated without further purification. Aladdin Chemical Reagent Co., Ltd, provided the magnesium chloride hexahydrate (MgCl₂·6H₂O), aluminium chloride hexahydrate (AlCl₃·6H₂O), citric acid (C₆H₈O₇), sodium carbonate anhydrous (Na₂CO₃), sodium hydroxide (NaOH), ammonium hydroxide (NH₃·H₂O) and silicon carbide (SiC). SBS polymer modified asphalt contained 4.5% SBS polymer particles was provided by Guochuang Co., Ltd, Hubei, China, and its basic properties were shown in table 1.
2.2. Preparation of the SiC wrapped LDHs processed at different temperatures
Firstly, low-saturation coprecipitation method was applied to synthesize LDHs. 5 mmol of AlCl₃·6H₂O and 15 mmol of MgCl₂·6H₂O were added in 200 ml distilled water with uniform dissolution. Secondly, while continuously stirring, an alkaline solution containing 40 mmol NaOH and 2.5 mmol Na₂CO₃ was added. Thirdly, with ultrasonic stirring for 20 min, 10 mmol of silicon carbide was added into the obtained LDHs and 50 ml of methanol to form a uniform suspension. After filtration, continuous distilled water and absolute ethanol were applied to wash the sediment, and ensure the neutral state of solution. Finally, the SiC wrapped LDHs (SwL) was obtained and then divided into four groups. Different groups were dried at 200 °C, 350 °C, 500 °C and 650 °C in a vacuum oven for 6 h, separately. According to the synthesis temperatures of SwL, different samples were named as SwL-200, SwL-350, SwL-500 and SwL-650.

2.3. Modification of asphalt
Firstly, 400 g of SBS modified asphalt was heated in a container placed in the oil bath, the heating and following modification process was ensured at 170 °C. Then based on former researches [16], 24 g (6% by weight of the asphalt) of different SwL were added into the SBS modified asphalt uniformly, and different modified asphalt were sheared by a 4500 rpm high-speed shearing machine for 1 h until the end of modification.

2.4. Characterization
The surface morphology was detected by a Sigma HD Field-emission Scan Electronic Microscope (FE-SEM) with Energy Dispersive Spectrum (EDS) manufactured by Zeiss, Germany, the magnification ranged from 10 X to 1000000X, the magnification of 500000 times was adopted in this paper. D8 Advance x-ray diffraction (XRD) manufactured by Bruker, Germany, was used to explore the phase compositions. Nicolet 6700 Fourier Transform Infrared spectrometer (FTIR) manufactured by Thermo Scientific, USA, was used to analyse the chemical structure.

2.5. Microwave absorption of SBS/SwL modified asphalt
A PNA-N5244A microwave vector network analyzer system made by Agilent, USA was used to measure the microwave absorption of modified asphalt. The relative complex permittivity \( \varepsilon_r = \varepsilon' - j\varepsilon'' \) and reflection loss were obtained from 1 GHz to 18 GHz. The samples used for the test were mixtures of paraffin wax and SBS/SwL modified asphalt, the mass ratio was 1:1 and paraffin was used to cure the samples. The samples were pressed to be cylindrical toroidal, their inner diameter, outer diameter and height were 3.04 mm, 7.0 mm and 2.0 mm.

2.6. Exothermic properties of SBS/SwL modified asphalt
Each asphalt sample was selected for 1 gram and formed a circle with a diameter of 2 cm for exothermic test, all samples were placed on a polyethylene sheet (no absorption of microwave radiation). In addition, a thick foam was placed under the polyethylene sheet to reduce heat loss. Each sample was heated for 4 times continuously (20s each time) through a NN-GT353M microwave oven made in Panasonic, Japan. The heating power and frequency of the microwave oven were 800W and 2.45 GHz, respectively. T420 infrared camera manufactured by Forward Looking Infrared Radiometer (FLIR) Systems, USA, was used to detect the temperature change of modified asphalt after each heating period quickly.

3. Results and discussions

3.1. Characterization
As shown in figure 1(a), the morphology is an obvious single-layer sheet structure, which is successively stacked into large agglomerates. The thickness of the single layer is at the scale of nanometer. The LDHs are uniform in morphology and arranged tightly and orderly, indicating that the prepared samples have ideal uniformity and regularity.

EDS spectra shown in figure 1 indicates the presence of Mg, Al, O, and C, which are in accordance with the elements of raw materials. From the figure 2(a), the nano-scale silicon carbide particle wrapped on the surface of the LDHs has a cubic structure with a crystal size of about 200 nm. In addition, the wrapped SiC and the single layer of LDHs nanosheets are sufficiently fused together to make the surface texture of the synthesized SwL rougher. As shown in figure 2(b), in addition to the original elements of Mg, Al, O, and C, the EDS spectra indicates the presence of Si and augment of C.

Figure 3 shows the XRD diffraction patterns of LDH and SwL processed at different temperatures. There are only six typical phase diffraction peaks of LDH, and the maximum diffraction intensity comes from (003). When heated to a certain temperature, LDH can be decomposed by thermal decomposition process, including the removal of interlayer water, the removal of carbon ions and the dehydration of layer hydroxyl groups. When the
Figure 1. FE-SEM images (a) and EDS spectra (b) of LDHs.

Figure 2. FE-SEM images (a) and EDS spectra (b) of SwL.

Figure 3. XRD patterns of LDHs and SwL processed at different temperatures.
processing temperature is 200 °C, only the interlayer water is lost and the layered structure still exists. After rising the processing temperature, more water including the bond water is lost in SwL-350, but the characteristic peaks of LDHs still appear. When heated to 500 °C, CO3−2 completely converts to CO2, and forms a bimetallic composite oxide (LDO). In additional, the ordered layered structure is destroyed. When the heating temperature exceeds 600 °C, the metal oxide formed after decomposition begins to sinter, resulting in a decrease in surface area and pore volume, finally SwL-650 is composed of MgAl2O4, MgO and SiC.

Figure 4 shows the FTIR spectra of LDHs and SwL processed at different temperatures.

3.2. Microwave absorption properties of the SBS/SwL modified asphalt

Figures 5 and 6 show the real part of complex permittivity (ε′) and the imaginary part of complex permittivity (ε″) of the SBS polymer modified asphalt and different SBS/SwL modified asphalt. Microwave energy can be divided into two parts, the storage ability is represented by ε′, the loss ability of storage is represented by ε″. It can be seen that the ε′ value of SBS polymer modified asphalt is not exceed 0.3. After the addition of SwL, the ε′ values of SBS/SwL modified asphalt have greatly increased, and their minimum value achieves 4.8, about 16 times higher than that of SBS modified asphalt. The same change also appears in the ε″. Complex permittivity shows that the addition of all types of SwL modifiers enhances the microwave absorption of SBS polymer modified asphalt, and the higher processing temperature of SBS/SwL achieves better effects in the microwave absorption [29, 30].

In view of the transmit-line theory [31], the reflection loss (Rf) can be obtained according to the given frequency (f) and thickness (t) [32]. As shown in figure 7, the least reflection loss exists at 7 mm is −1.05 dB at 14.98 GHz, revealing low absorption of microwave irradiation.

As revealed in figure 8, after the addition of SwL, the reflection loss values of all SBS/SwL modified asphalt are greatly improved in the entire test frequency band. Results show that after the addition of SwL, the increase of the complex permittivity results in the lower reflection loss of SBS/SwL modified asphalt, which in turn enhances the microwave absorption of SBS/SwL modified asphalt. When the thickness of test sample is 8 mm, the SBS/SwL-200 modified asphalt has the lowest reflection loss of −9.23 dB at 15.96 GHz among all SBS/SwL modified asphalt samples. The lowest reflection loss of SBS/SwL-350 modified asphalt is 9.09 dB and only 1.51% lower than that of SBS/SwL-200 modified asphalt. But when the processing temperature is higher than 350 °C, the absorbing ability is rapidly attenuated, and the reflection loss values of SBS/SwL-500 modified asphalt and SBS/SwL-650 modified asphalt decrease to more than 70% of that of SBS/SwL-200 modified asphalt. The reason for these changes is that the microwave absorption capacity of SBS/SwL modified asphalt is closely related to the water between layers and the layer structure of SwL. Firstly, the water molecules are polar
molecules with large dielectric constant. Large dielectric constant contributes to higher dielectric loss factor, and enhance the microwave absorption capacity. Secondly, the layer structure of SwL allows microwave radiation to be reflected and absorbed multiple times between the layers, which increases the loss of microwave radiation in the sample [33]. When the processing temperature gradually increases, the free and bound water between the layers begins to disappear. Meanwhile, the lamellar structure collapses and affect the continuous reflection of microwave radiation, resulting in a gradual decrease in the microwave absorption ability of the modified asphalt.

3.3. Exothermic properties of SBS/SwL asphalt

Figures 9 and 10 show the infrared images of the SBS polymer modified asphalt and the SBS/SwL modified asphalt at a microwave frequency of 2.45 GHz, respectively. Each figure includes a schematic diagram of the exothermic test and infrared images measured at five time points with temperature scales on the infrared images. The color variation demonstrates temperature distribution of different sample, while brighter color means
higher temperature and darker color means lower temperature. As shown in figure 9, the temperatures measured at different times are basically the same, only a little exothermic phenomenon caused by heat conduction. In contrast, figure 10 reveals more difference that heat release degrees of different modified asphalt are obvious. SBS/SwL-200 modified asphalt shows better exothermic phenomenon than other modified asphalt samples.

To analyze the exothermic area more specialty and accurately, the infrared area of modified asphalt including all temperature distributions was selected to be further treated by FLIR tools software. Figure 11 indicates the temperature versus heating time and fitted equation for different asphalt. It can be seen that the
temperature values of all samples increase with the incremental heating time. All exothermic curves can be fitted into quadratic equations, and the $R^2$ values obtained by fitted results are greater than 0.99, which shows better fitting results. The five exothermic curves can be divided into three categories: the first one is SBS polymer

**Figure 9.** Infrared images of SBS polymer modified asphalt under microwave radiation: (a) schematic diagram; (b) 0s of heating; (c) 20s of heating; (d) 40s of heating; (e) 60s of heating; (f) 80s of heating.

**Figure 10.** Infrared images of different SBS/SwL modified asphalt under microwave radiation: (a) schematic diagram; (b) 0s of heating; (c) 20s of heating; (d) 40s of heating; (e) 60s of heating; (f) 80s of heating.
modified asphalt, whose slope of exothermic curve is small. Former researches indicated that the SBS polymer modified asphalt cannot absorb microwave radiation, so the temperature rise is mainly caused by the heat conduction in the microwave heating instrument. The second one is SBS/SwL-500 modified asphalt and SBS/SwL-650 modified asphalt, they show moderate exothermic effects with a heating rate of 0.11 °C/s and 0.09 °C/s, respectively. The third one is SBS/SwL-200 modified asphalt and SBS/SwL-350 modified asphalt, they show good exothermic effects with a heating rate of 0.26 °C/s and 0.24 °C/s, respectively. Thus, the SBS/SwL-200 modified asphalt has the best exothermic properties under microwave irradiation.

4. Conclusions

This paper investigated the controllable synthesis of traditional microwave absorber SiC to wrap LDHs (SwL) at different temperatures. Then morphology, phase composition, chemical structure of different SwL were evaluated. Finally, based on different combination of SBS and SwL, the effects of SBS/SwL on the microwave absorption and exothermic properties of modified asphalt were studied. Synthesized SwL were firstly processed at 200 °C (SwL-200), 350 °C (SwL-350), 500 °C (SwL-500) and 650 °C (SwL-650). Then, their surface morphology, phase composition and chemical structure were measured. Finally, by modifying the SBS modified asphalt with different processed SwL samples, the microwave absorption in the frequency band ranging from 1 GHz to 18 GHz, exothermic properties at 2.45 GHz of modified asphalt were also analyzed. The following conclusions can be drawn:

- The addition of all types of SwL modifiers significantly enhances the microwave absorption capacity of modified asphalt in the full frequency range. The $\varepsilon'$ and $\varepsilon''$ values of SBS/SwL modified asphalt decrease with the processing temperature.
- After the addition of SwL, the increase of the complex permittivity results in the lower reflection loss of SBS/SwL modified asphalt, which in turn enhances the microwave absorption of SBS/SwL modified asphalt. When the thickness of test sample is 8 mm, the SBS/SwL-200 modified asphalt has the lowest reflection loss of $-9.23$ dB at 15.96 GHz among all SBS/SwL modified asphalt.
- SBS/SwL-200 modified asphalt shows significant exothermic phenomenon than other modified asphalt and SBS/SwL modified asphalt samples at the microwave frequency of 2.45 GHz, reflecting the highest heating rate of 0.26 °C/s$^{-1}$.

Thus, it is more superior to select 200 °C as the processing temperature of SwL when used in SBS modified asphalt.
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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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