

Article

Investigation of the Microwave Absorption of Asphalt Mixtures Containing Magnetite Powder

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Abstract: This article is intended to investigate the microwave heating performance of asphalt mixtures containing magnetite powders (MPAM). For this purpose, the surface temperatures of asphalt mixtures containing different dosages of magnetite powders were measured during microwave heating. The low-temperature bending test and water sensitivity test were also conducted to evaluate the performance of MPAM. Heating rate and reflection loss of different thicknesses of MPAM were determined using a microwave heating test and arch reflectivity test, respectively. The results showed that because its main components are triiron tetroxide and iron oxide, which have excellent microwave-absorbing properties, magnetite powders can be used as microwave absorbers to improve the ability of microwave absorption and increase the heating rate of asphalt mixtures. The heating rate of the asphalt mixtures increased with the increase of the amount of magnetite powder. The addition of magnetite powder improved the low-temperature properties of the asphalt mixture, but it reduced the water stability of the asphalt mixture. Considering that the microwave-absorbing asphalt mixture used for melting snow and ice should have good water stability, the recommended dosage of magnetite powders was 60%. The microwave-absorbing properties of MPAM were related to its thickness in the pavement structure and frequency of microwaves. In order to greatly enhance the absorbing efficiency, future work should be focused on matching thickness and matching frequency.

Keywords: asphalt mixture; magnetite powders; microwave absorbance; heating; snow melting; deicing

1. Introduction

Timely removal of snow and ice from pavement surfaces is essential for traffic safety in the winter [1]. Mechanical snow removal and snow melting agents are often used to remove snow and ice from pavement surfaces by road maintenance departments [2]. However, mechanical snow removal faces difficulties when removing snow compaction and can easily cause surface damage. Although snow melting agents, such as sodium chloride, calcium chloride, and magnesium chloride, are better at removing snow and ice, they cause corrosion to vehicles and concrete structures [3,4]. Considering the defects of traditional snow melting and deicing methods, it is necessary to develop a new, efficient, and clean method for removing snow and ice.

Microwave deicing uses microwaves to rotate polar molecules in the asphalt mixture to generate thermal energy in order to melt and separate the ice layer in the snowpack from the pavement surface [5–8]. Compared to traditional snow melting and deicing methods, microwave deicing technology has the advantages of high deicing efficiency and being environmentally friendly [9]. However, there is only a small number of microwave-absorbing components in asphalt mixtures [10].

Most of this energy is transmitted or reflected in the asphalt mixture, and only a small portion of the energy is absorbed in the asphalt mixture [11]. Because of this, microwave deicing technology faces the problem of excessive energy consumption, which limits the promotion and application of this technology [12–14]. In order to improve the microwave-absorbing ability of asphalt mixtures and reduce the energy consumption of microwave deicing, various kinds of microwave absorbers are used in asphalt mixture. Notani [15] found that carbon fiber modified asphalt concrete can melt dense layers of snow and ice and be used in critical areas such as airfields. Sun [16] studied the self-healing performance of asphalt mixtures containing steel fibers with microwave heating. Zhang [17] evaluated the microwave deicing performance of an asphalt mixture made with low-grade pyrite cinder synthetic ceramics. Magnetite is a rock mineral, whose main component is Fe_3O_4 , which has ferromagnetic and semi-conductive properties, and it is widely distributed throughout the world [18,19]. Compared with the other microwave absorbers mentioned previously, magnetite powders are easy to obtain and are inexpensive. The microwave-absorbing performance of an asphalt mixture may be improved by the addition of magnetite powders (MPAM). However, few studies have reported on this.

In this paper, the microwave-absorbing property of asphalt mixtures containing magnetite powders was investigated. The surface temperatures of asphalt mixtures containing different dosages of magnetite powders were measured during microwave heating. The low-temperature bending test and water sensitivity test were also conducted to evaluate the performance of MPAM. Heating rate and reflection loss of different thickness of MPAM were determined using a microwave heating test and arch reflectivity test, respectively. According to the test results, the optimum content of magnetite powders in asphalt mixtures and the reasonable thickness of MPAM in the pavement structure were determined. In addition, the microwave heating mechanism of MPAM was also discussed.

2. Materials and Methods

2.1. Materials

Asphalt (90#, SK energy Co., Ltd., Seoul, Korea) was used as the binder, and its properties are shown in Table 1. Limestone (Tie Cheng Co., Ltd., Xi'an, China) was used as an aggregate, and its properties are shown in Table 2. Magnetite powders (Tie Cheng Co., Ltd., Xi'an, China) and limestone powders (Tie Cheng Co., Ltd., Xi'an, China) were used as the fillers, and their physical properties are summarized in Table 3.

Table 1. Properties of 90# asphalt.

Test Properties	Unit	Test Results	Test Basis
Needle penetration (25 °C)	0.1 mm	83.1	ASTM D5-19 [20]
Softening point	°C	47.4	ASTM D36-14 [21]
Ductility (15 °C, 5 cm/min)	cm	>100	ASTM D113-17 [22]
Wax content	%	1.74	ASTM D3344-90 [23]
Specific gravity	–	1.030	ASTM D70-18 [24]
Flash point	°C	304	ASTM D92-18 [25]
RTFOT (163 °C, 75 min)			
Mass change	%	0.05	ASTM D2872-19 [26]
Penetration ratio (25 °C)	%	82.7	ASTM D5-19
Ductility (10 °C)	cm	29.3	ASTM D113-17

Table 2. Properties of limestone aggregate.

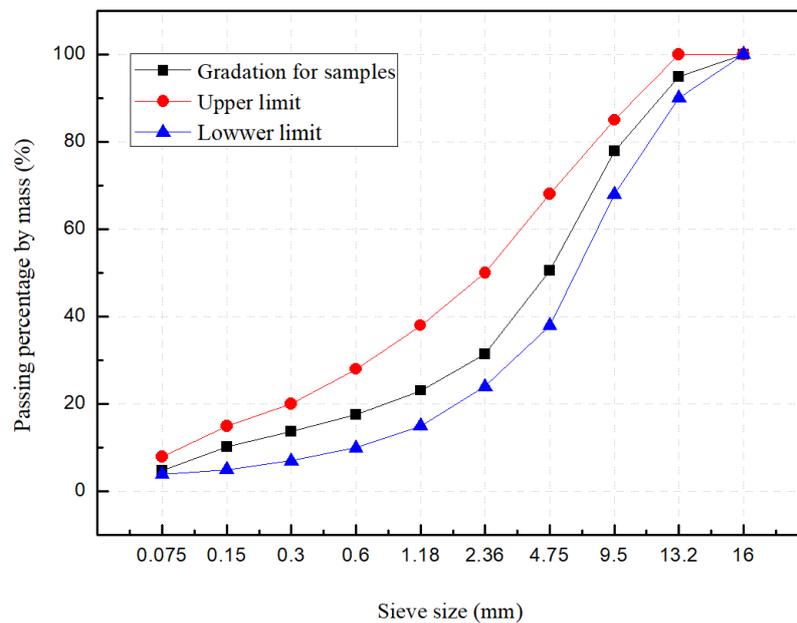
Crushing Value (%)	Los Angeles Wear value (%)	Density (g/cm^3)	Water Absorption (%)
18.4	20.7	2.639	0.70

Table 3. Physical properties of magnetite powder.

Filler	Density(g/cm ³)	Water Content (%)	Hydrophilic Coefficient	0.075 mm Percent Passing (%)
Magnetite powder	4.6	1.1	1.43	75.7
Limestone filler	2.79	0.5	0.56	77.2

2.2. Sample Preparation

Magnetite powder was added to the base asphalt binder instead of limestone powder by volumes of 0%, 20%, 40%, 60%, 80%, and 100%. The optimum asphalt contents of asphalt mixtures containing 0%, 20%, 40%, 60%, 80%, and 100% magnetite powder are 4.7%, 4.6%, 4.6%, 4.5%, 4.5%, and 4.4%, respectively. AC-13 gradation of the aggregate is shown in Figure 1.

**Figure 1.** AC-13 gradation of the aggregate.

At first, aggregate and mineral fillers were dried at 110 °C for 6 h. The asphalt was heated in an oven at 150 °C. Then, the aggregate, asphalt, magnetite powders, and limestone powders were added in the mixer in sequence at a temperature of 150 °C. The asphalt mixture was prepared at a speed of 75 r/min for 3 min. The prepared mixture was placed in molds by the compaction method. After curing for 24 h, specimens were demolded from the molds and cut using a diamond disc. There were three sizes of specimens. One was 101.6 ± 0.25 mm in diameter and 63.5 ± 1.35 mm in height and was used for microwave heating and freeze–thaw splitting tests. The second was 300 mm × 300 mm × 70 mm and used for the reflection loss test. The last was 250 mm × 30 mm × 35 mm and was used for the bending test. In order to study the thickness of MPAM on the microwave-absorbing performance, compacted specimens with 0, 3, 5, and 7 cm thicknesses of MPAM were prepared, which is shown in Figure 2. Preparation of the specimens of dual material was divided into the following two steps. At first, the base layer of the ordinary asphalt mixture was compacted using a drop hammer 50 times on each side. Then, the loose MPAM was also placed on the base layer and compacted using the drop hammer 50 times on each side. The total height of specimens was controlled at 70 ± 1 mm.

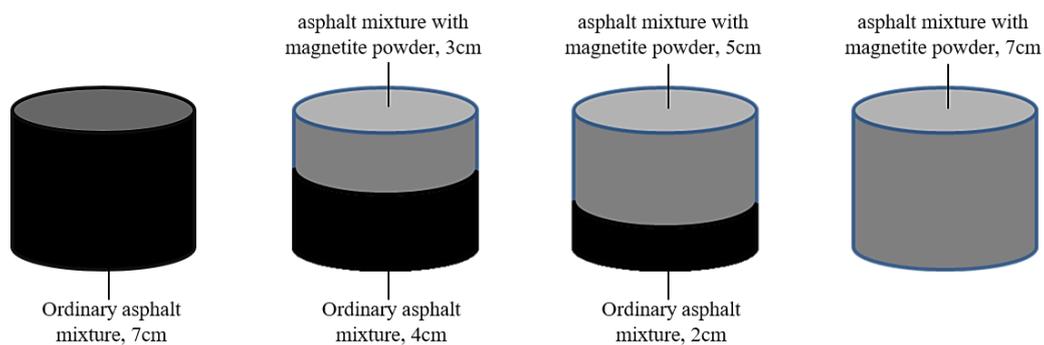


Figure 2. Asphalt mixture with different thicknesses of asphalt mixtures containing magnetite powders (MPAM).

2.3. Microwave Heating Test

An infrared temperature instrument (Fluke Corporation, Everett, WA, USA) was used to test the electromagnetic wave (EM) absorption performance of the asphalt mixture. The prepared mixtures were heated in a Microwave Heating (MH) device (Galanz, Shunde, China). It was noted that as microwave heating is of volumetric type, the surface was not the hottest point in the heated object. However, for the deicing methodology using microwaves, deicing efficiency is closely related to surface temperature. Therefore, surface temperature was tested in this paper. The initial surface temperature of the mixture was measured before heating. The temperatures of the Marshall specimens were measured every 2 min after the start of heating. Heating the asphalt mixture was done with a microwave oven (800 W power, 2.45 GHz). When the surface temperature of specimens rose to 200 °C, heating was stopped. Then, the heating rate and the variation of temperature with heating time of the mixture were obtained. Five spots of each surface were tested.

2.4. Bending Test

The low-temperature property of the asphalt mixture was evaluated by a 3-point bending test at a low temperature in terms of JTG E20-2011 [27]. The loose asphalt mixture was compacted into a 300 mm × 300 mm × 50 mm slab and sawed into beams (250 mm × 30 mm × 35 mm) that each spanned lengths of 200 mm. The test temperature was 10 °C, and the loading rate was 50 mm/min.

$$R_B = \frac{3LP_B}{2bh^2}, \quad (1)$$

where R_B is the splitting strength of the specimen (MPa); L is the length of the specimen (200 mm); P_B is maximum load when the specimen is broken (N); b is the width of the specimen's cross-section (mm); and h is the height of the specimen's cross-section (mm).

2.5. Freeze–Thaw Splitting Test

Water sensitivity of the asphalt mixture was evaluated by a freeze–thaw splitting test according to T0716 of JTG E20-2011 [27]. In this test, the Marshall specimens with 50 hammer blows on each side were used. The specimens were immersed in water in vacuum conditions and were then put into a plastic bag with 10 mL water and transferred to a chamber at a temperature of -18 ± 2 °C for 16 ± 1 h. After freezing, specimens were kept in water at a temperature of 60 ± 5 °C for 24 h without a plastic bag. After the freeze–thaw treatment, specimens were placed in water at a temperature of 25 °C for 2 h. The tensile strength ratio (TSR) can be calculated with Equations (2)–(4).

$$R_{T1} = \frac{0.006287P_{T1}}{h_1} \quad (2)$$

$$R_{T2} = \frac{0.006287P_{T2}}{h_2} \quad (3)$$

$$\text{TSR} = \frac{\bar{R}_{T2}}{\bar{R}_{T1}} \times 100 \quad (4)$$

where R_{T1} is the splitting strength of the specimen set without freezing and thawing. R_{T2} is the splitting strength of the specimens after freezing and thawing (MPa). P_{T1} is the test load value of a set of specimens without freeze–thaw cycles (N). P_{T2} is the test load value of a set of specimens with freeze–thaw cycles (N).

2.6. Reflection Loss Test

In this paper, the reflection loss of materials was measured with the arch method testing system according to the standard measurement methods for reflectivity of radar-absorbing materials (GJB2038A-2011) [28]. The arch method testing system is shown in Figure 3. The specimen was placed on the shelf, and the reflection loss test was measured at frequencies from 2 to 14 GHz.

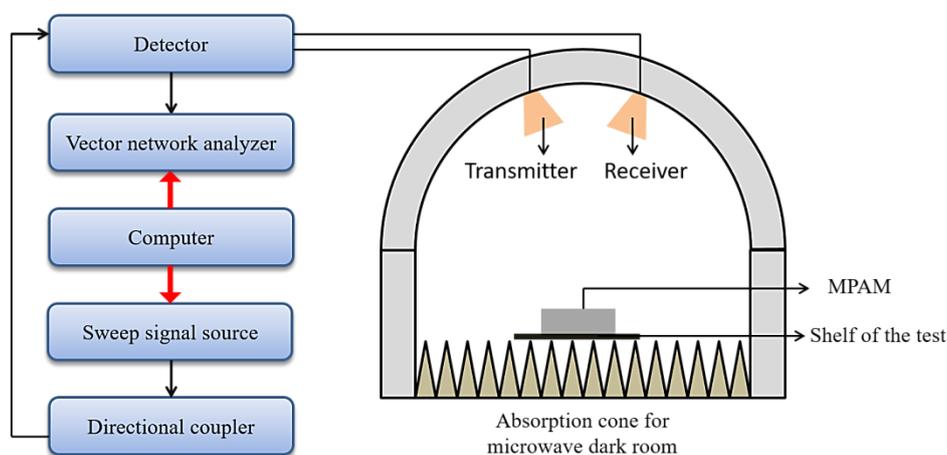


Figure 3. The schematic diagram of the arch method testing system.

2.7. Microscopy and X-ray Diffraction (XRD) Test

The morphologies of the magnetite powder and the limestone filler were analyzed with a Quanta FEG 250 Scanning Electron Microscope (SEM, S-4800; Hitachi, Tokyo, Japan). The magnetite powder was investigated with X-ray diffraction (XRD, Bruker Corporation, New York, NY, USA). The test voltage was 40 kV.

3. Results and Discussion

3.1. Effect of Magnetite Powder Dosage on the Microwave Heating Performance

Figure 4 shows the surface temperature of MPAM containing different dosages of magnetite powders during microwave heating. It can be seen in Figure 4 that the surface temperature of MPAM showed an increasing trend with the increase of microwave heating time. Table 4 shows a positive linear relationship between surface temperature and microwave heating time in MPAM containing different dosages of magnetite powders. The relationship between surface temperature and microwave heating time can be described by Equation (5).

$$T = Kt + T_i \quad (5)$$

where T is the surface temperature ($^{\circ}\text{C}$), t is the microwave heating time (min), T_i is room temperature ($^{\circ}\text{C}$), and K is the heating rate ($^{\circ}\text{C}/\text{min}$).

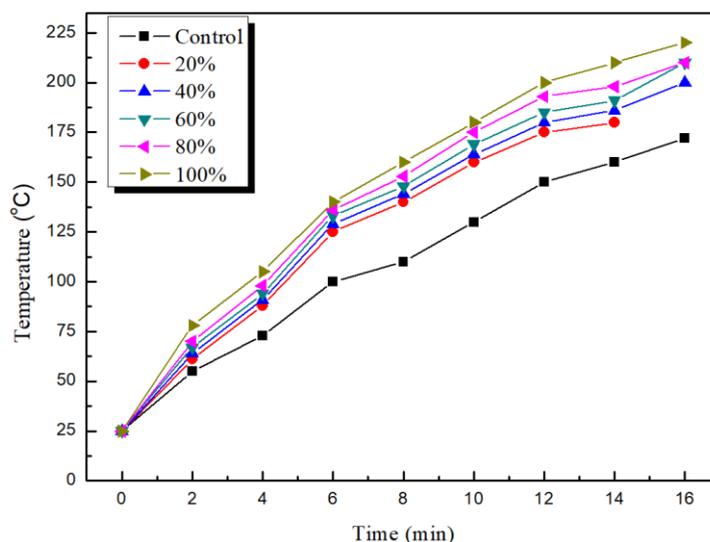


Figure 4. Surface temperature of MPAM during microwave heating.

Table 4. The relationship between surface temperature and heating time.

Dosage of Magnetic Powder (%)	Regression Equation	K	R ²
0	$T = 10.02t + 25$	10.02	0.994
20	$T = 12.52t + 25$	12.52	0.987
40	$T = 12.79t + 25$	12.79	0.984
60	$T = 13.03t + 25$	13.03	0.984
80	$T = 13.44t + 25$	13.44	0.981
100	$T = 14.16t + 25$	14.61	0.981

It can be seen in Table 4 that the heating rate (*K*) increased with increasing dosages of magnetite powders. Compared with traditional asphalt mixture, the heating rate of MPAM containing 20%, 40%, 60%, 80%, and 100% dosages of magnetite powders increased by 24.9%, 27.6%, 30.0%, 34.1%, and 45.8%, respectively. *R*² is the correlation coefficient. This phenomenon clearly shows that the addition of magnetite powders as microwave absorbers improved the microwave heating performance of the asphalt mixture. When the pavement surface is heated to the same temperature, MPAM can save more energy than traditional asphalt mixtures.

To gain a better understanding of the influence of magnetite powders on the microwave heating performance of MPAM, XRD analyses of magnetite powders were carried out. The results are shown in Figure 5. The main mineral components of the magnetite powders were determined using Jade 5.0 software. The mineral content was calculated with X' Pert High Score Plus software (Version 2.0). Figure 5 shows the main components of magnetite powders from magnetite tailings, which are magnetite (Fe₃O₄), hematite (Fe₂O₃), and quartz (SiO₂). The proportions of Fe₃O₄, Fe₂O₃, and SiO₂ in magnetite powders are 47.5%, 24.4%, and 28%, respectively. Some studies have reported that, based on the heating rate, Fe₃O₄, Fe₂O₃, and SiO₂ are hyperactive, active, and inactive, respectively. From the perspective of photon transition, the microwave absorption process of magnetite particles is a rotating molecular mechanism that is related to its own structure [29]. The structure of Fe₃O₄ is an inverse spinel in which an oxygen atom forms a face-centered cubic lattice densely packed with iron atoms occupying tetrahedral and octahedral positions, which makes the magnetite have ferromagnetic and semi-conductive properties [7]. Therefore, higher dosages of magnetite powder resulted in a better microwave absorption ability of MPAM. The results of the microwave heating test also proved this. Figure 6 shows the heating process of MPAM with microwave heating. When microwaves irradiate MPAM, microwave-absorbing components in asphalt mixtures, especially magnetite powders and

metal oxide in the aggregate, absorb microwave energy and convert it into heat energy. Subsequently, heat energy is transferred to the entire MPAM. Finally, the temperature of MPAM increases.

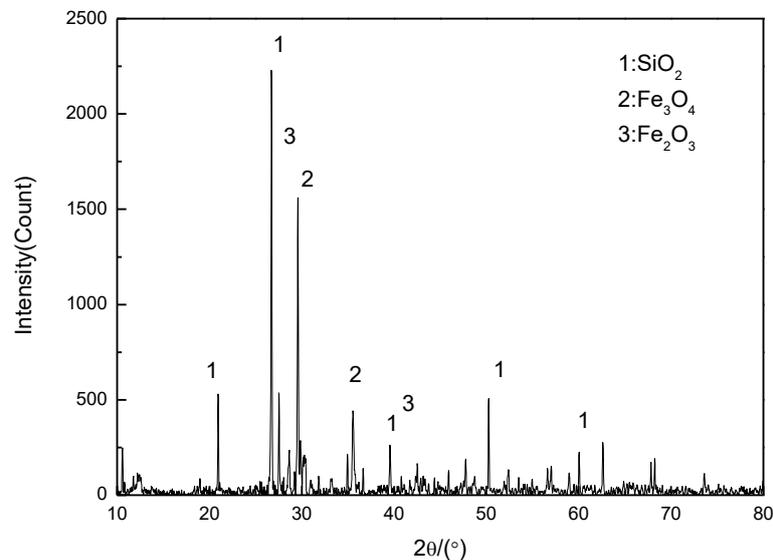


Figure 5. XRD pattern of magnetite powder

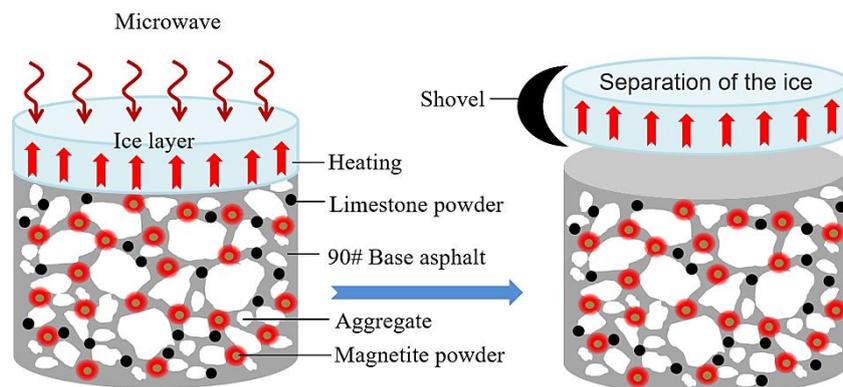


Figure 6. Microwave heating deicing process.

3.2. Effect of Magnetite Powder Dosage on Road Performance

MPAM is used for melting snow and ice. Therefore, it should have excellent crack resistance at a low temperature and good water sensitivity.

3.2.1. Low-Temperature Properties

Low-temperature properties of the asphalt mixture were evaluated with a 3-point bending test. The evaluation indexes of asphalt mixtures at a low temperature are the maximum bending tensile strength, the maximum tensile strain, and the bending stiffness modulus. The relationship between the maximum bending stress of the asphalt mixture and the content of magnetite powders is shown in Figure 7. The error bars represent the standard deviation of the data set. It shows that the maximum bending stress of MPAM with different dosages of magnetite at 10 °C was 11.12, 12.66, 12.27, 12.45, 12.56, and 11.64 MPa, respectively. The addition of magnetite powders can slightly increase the maximum bending stress at a low temperature. Figure 8 shows that with the increasing dosage of magnetite powders, the maximum tensile strain increased continuously, which indicates that the addition of magnetite powders had a positive impact on the low-temperature deformation capacity. Although the bending stiffness modulus of the asphalt mixture increased when a 20% dosage of the magnetite powders was added, the overall

trend was that the bending stiffness modulus decreased as the amount of magnetite powders was increased. It can be seen clearly that the maximum tensile strain increased with an increasing dosage of magnetite powders. The reason may be that the surface of the magnetite powder is more “clean” than that of limestone filler (Figures 9 and 10), which results in less absorption of light asphalt fractions. The “excess” free asphalt is beneficial to reduce the bending stiffness modulus of the asphalt mixture to a certain extent and improves its flexibility. Therefore, the low-temperature crack resistance of the asphalt mixture is improved with the addition of magnetite powder [30].

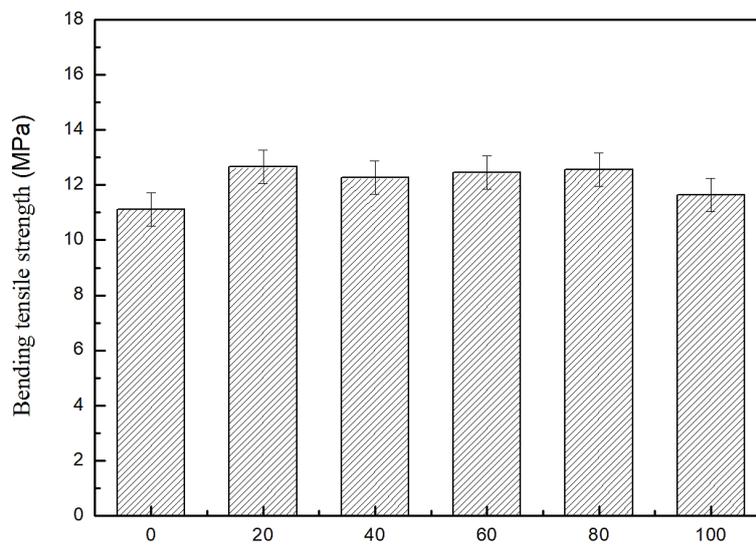


Figure 7. Bending tensile strength of the mixture.

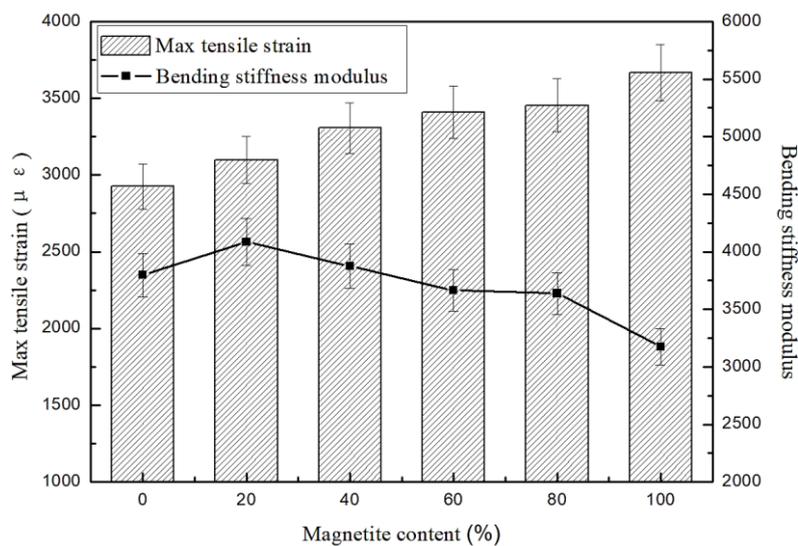


Figure 8. Max tensile strain and bending stiffness modulus of the mixture.

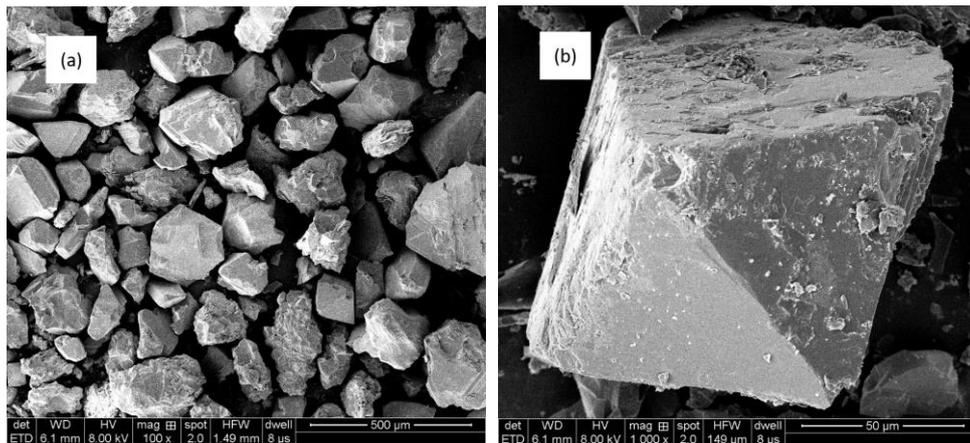


Figure 9. SEM image of the magnetite powder (a) 100 times and (b) 1000 times.

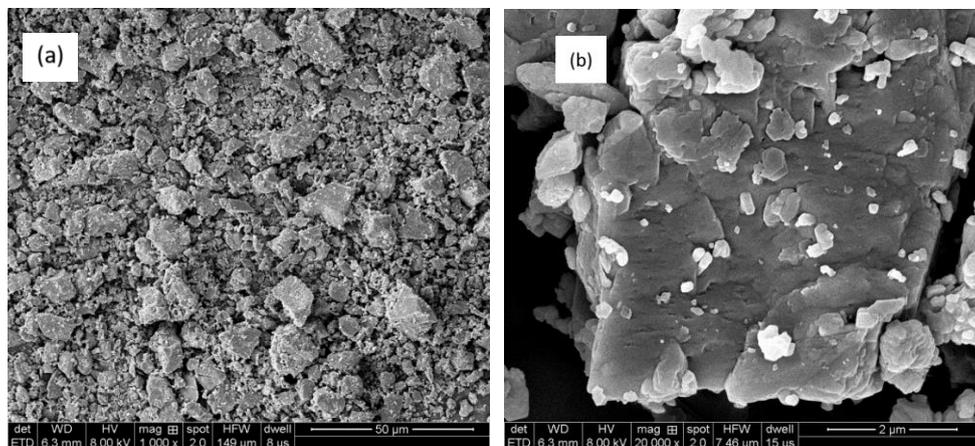


Figure 10. SEM image of the limestone filler (a) 1000 times (b) 20,000 times.

3.2.2. Water Sensitivity

Figure 11 shows the tensile strength ratio (TSR) of MPAM with different dosages of magnetite powders. The value of TSR decreased with the increase of the dosage of magnetite powders. It indicates that the addition of magnetite powders reduced the water stability of the asphalt mixture. The function of mineral powders is to fill the voids in the aggregate skeleton and make the asphalt mixture denser to improve the cohesion of the asphalt binder and the stability of the mixture. The surface structure of limestone powder is rough, the particle size is small, and the surface area is large, as shown in Figure 10. These characteristics increase the contact area between asphalt and filler, and it is easier to absorb and stabilize the interface [31]. Limestone powder with a smaller particle size is beneficial for making the mixture denser. Additionally, the limestone powder contains more calcite (CaCO_3), which can interact with acidic components in the asphalt to improve the cohesion of the asphalt binder [32]. The use of magnetite powder instead of limestone powder as a mineral filler is not conducive to the water stability of the asphalt mixture. Therefore, it is necessary to control the amount of magnetite powder in the asphalt mixture. According to the Chinese specification (JTG F40-2004) [33], the value of TSR should be greater than 80%. In order to meet water stability requirements and maintain the microwave heating performance of MPAM, the recommended dosage of magnetite powders is 60%.

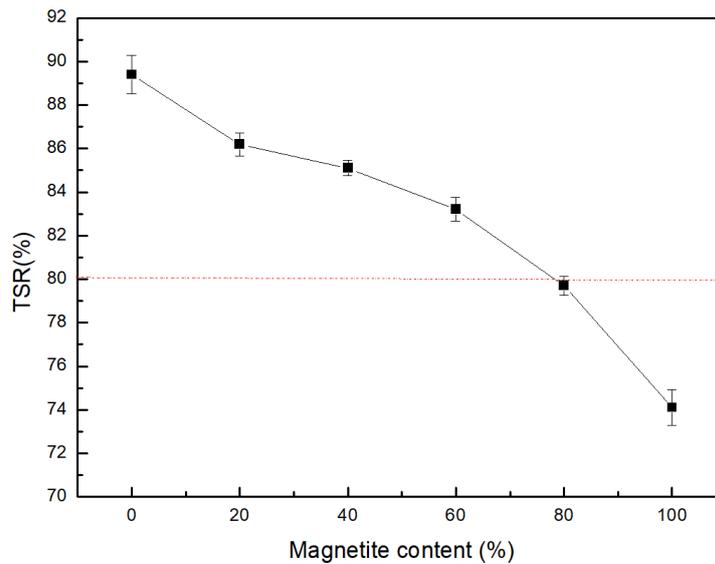


Figure 11. Water sensitivity of different magnetite content. TSR, tensile strength ratio.

3.3. Effect of MPAM Thickness on the Microwave-Absorbing Performance

Figure 12 shows the surface temperature of the pavement with different thicknesses of MPAM containing a 60% dosage of magnetite powders during microwave heating. Table 5 shows a positive linear relationship between surface temperature and microwave heating time of the pavements with different thicknesses of MPAM. Compared with traditional asphalt pavement, the heating rate of the asphalt pavement with 3, 5, and 7 cm thicknesses of MPAM was increased by 60.3%, 34.5%, and 27.2%, respectively. The pavement with 3 cm thickness of MPAM had the best microwave heating performance.

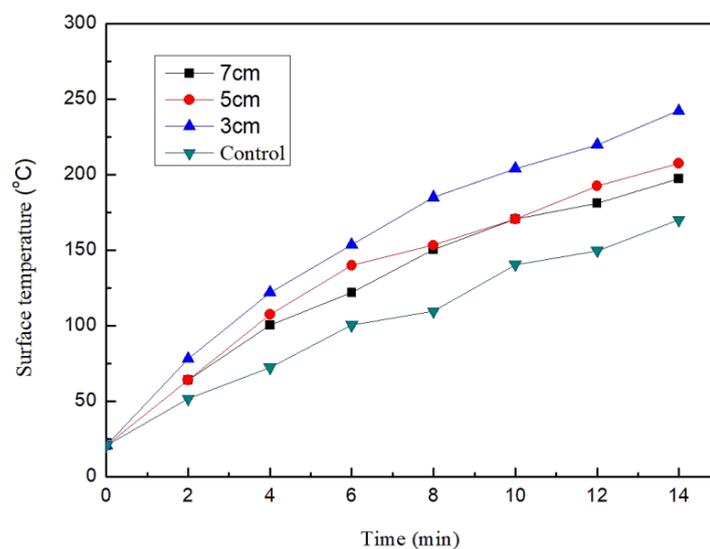


Figure 12. Surface temperature of the pavement with different thicknesses of MPAM.

Table 5. The relationship between surface temperature and heating time.

Thickness (cm)	Regression Equation	K	R ²
3 cm MPAM + 4 cm AM	$T = 17.35t + 25$	17.35	0.985
4 cm MPAM + 3 cm AM	$T = 14.55t + 25$	14.55	0.986
7 cm MPAM	$T = 13.76t + 25$	13.76	0.988
7 cm AM	$T = 10.82t + 25$	10.82	0.966

Figure 13 shows the heating process of pavement with an MPAM layer under microwave heating. When microwaves encounter the pavement, they are transmitted, reflected, and absorbed. The more microwave energy is absorbed, the higher the heating rate of the pavement. Figure 14 shows the reflection losses of the pavement with different thicknesses of MPAM. The greater the absolute value of the reflection loss, the more energy is absorbed, and the more heat is converted. It can be observed from Figure 14 that the pavement with 3 cm thickness of MPAM had a better absorbing performance than that with 5 and 7 cm thicknesses of MPAM, which is also proved by Figure 12. This indicates that the absorption efficiency grew with a decrease in the composite layer thickness. It can be inferred that the optimum thickness for microwave absorption may be less than 3 cm. However, the upper layer should be larger than a certain thickness to ensure the durability of the pavement. According to Chinese specification (JTG D50-2017) [34], the thickness of the upper layer made with asphalt mixture with dense gradation should be above 3 cm. Therefore, the upper layer of the microwave-absorbing pavement surface made with asphalt mixture with dense gradation is recommended to be 3 cm thick in this paper. There are also many new types of pavement surface coatings with thicknesses less than 3 cm, such as ultra-thin friction course and chip seal. These new pavement surface coatings can be designed to achieve an optimum thickness of MPAM. It is also noted that, for 3 cm MPAM, as the frequency increased, the value of reflection loss first decreased and then increased. There was minimum reflection loss at 13 GHz. In summary, the microwave-absorbing properties of MPAM are closely related to its thickness in the pavement structure and frequency of microwaves. Optimal absorbance efficiency can be achieved when a matching thickness of MPAM is subjected to microwave radiation at a matching frequency [35–38]. More work on the matching thickness and matching frequency should be done in future research.

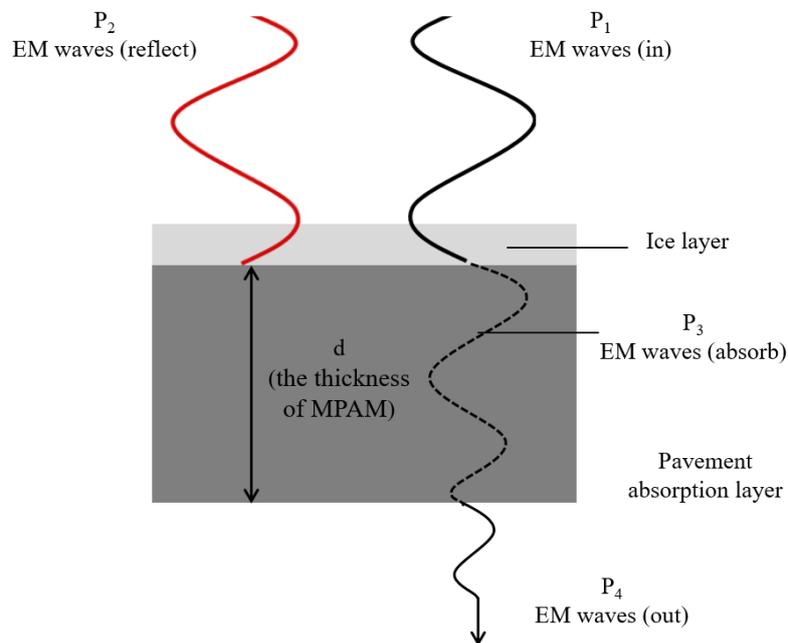


Figure 13. Schematic illustration of EM waves absorption in an asphalt mixture.

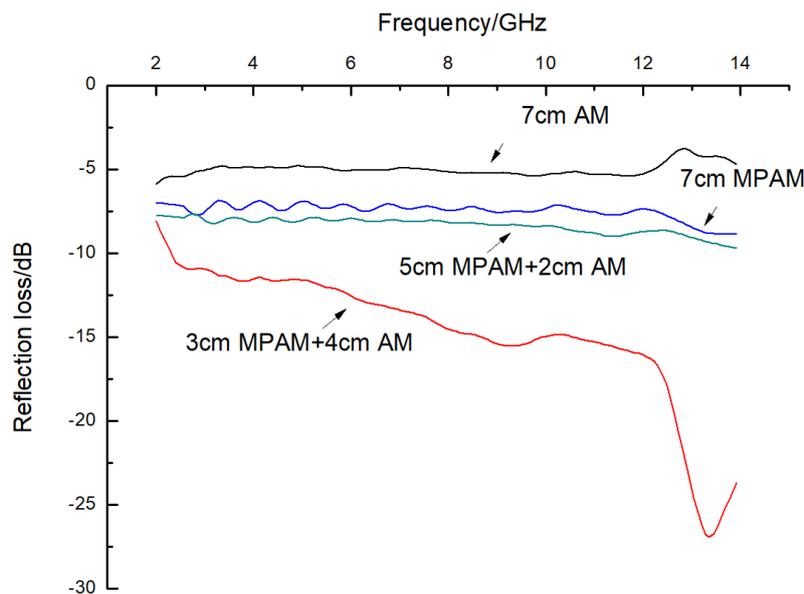


Figure 14. Reflection losses of the pavement with different thicknesses of MPAM.

4. Conclusions

- Low microwave absorption efficiency and high cost limit application of the deicing methodology using microwaves. Magnetite powders can be used as inexpensive microwave absorbers to improve the ability of microwave absorption and increase the heating rate of asphalt mixtures. The heating rates of asphalt mixtures increase with the increase of the amount of magnetite powders.
- The addition of magnetite powder improves the low-temperature properties of the asphalt mixture but reduces the water stability of the asphalt mixture.
- Considering that the microwave-absorbing asphalt mixture used for melting snow and ice should also have good water stability, the recommended dosage of magnetite powders is 60%.
- The microwave-absorbing properties of MPAM are related to its thickness in the pavement structure and frequency of microwaves. In order to greatly enhance the absorbing efficiency, future work should be focused on the matching thickness and matching frequency.

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