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Electromagnetic wave absorbing characteristics of carbon black cement-based composites

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ABSTRACT

Electrical resistivity, compressive strength, and the electromagnetic absorbing effectiveness of carbon black (CB) cement-based composites (CBCC) with different contents of high-structure CB were studied in this paper. The results indicate that the resistivity of CBCC versus the concentration of CB curves has typical features of percolation phenomena: CBCC in the percolation threshold zone contains 0.36–1.34 vol.% of CB. Thus, the conductive network can be formed in CBCC by using small amount of high-structure CB. Compressive strength of CBCC decreases with CB content increasing. Especially, compressive strength decreases substantially when CB content is more than 3.0 wt.%. CBCC exhibits good performance of absorbing electromagnetic waves in the frequency range of 8–26.5 GHz. For CBCC containing 2.5 wt.% of CB, the minimum reflectivity reaches −20.30 dB. The frequency bandwidth in which the reflectivity is less than −10 dB was from 14.9 GHz to 26.5 GHz. The filling of CB has improved the dielectric constant and the loss factor of the cement material remarkably. The loss factor of CBCC increases with the CB content increasing.

1. Introduction

Now people are living in a more and more complicated electromagnetic environment. Actually, electrical devices have greatly improved the quality of our lives. However, everything has its bad effects. For example, sometimes we have to shield the electromagnetic radiations from such devices as computers, mobiles, and military devices to avoid leaking out of important information or avoid radar tracing. In other cases, the reflection of electromagnetic waves from the enclosure of high buildings can lead to the disorder of TV signals around the buildings. Now people are aware that radiation of electromagnetic waves may do harm to the health of human beings. Thus, development of building composite materials containing low cost components such as carbon black (CB) which are able to absorb or shield electromagnetic radiations becomes more and more necessary in the modern society.

The shielding effectiveness (*SE*) is the sum of three terms such as reflection loss, absorption loss and multi-reflections [1]. So, *SE* is defined in decibels (dB) and its magnitude can be written as follows:

$$SE = 20\log\left|\frac{E_i}{E_t}\right| = R + A + M \qquad (1)$$

where E_i and E_t are the electric fields that are incident on and transmitted through the shield, respectively; R, A and M are the reflection loss, absorption loss, and loss due to multiple reflections and transmissions respectively. While, the electromagnetic absorbing effectiveness of the specimen is denoted with the reflectivity R_a, which is expressed as:

$$R_a = 20\log\left|\frac{E_r}{E_i}\right| \qquad (2)$$

where E_i and E_r referred to the electric field strength of the incident wave and that of the reflected wave, respectively. That is, the reflectivity of −10 to −20 dB means that the incident electromagnetic waves have been reduced by about 70–90%. As the reflectivity of absorbing wave materials is less than −10 dB, they can be used in practice.

Cement is slightly conductive, but its *SE* is very low. It is a simple and practical method to increase the cement materials' *SE* by adding a small amount of a conductive additive such as graphite powder, carbon black, carbon fibers, carbon filaments or steel fibers. Readers may find some literatures about cement-based electromagnetic shielding or absorbing materials in the early review by Guan et al. [2]. After that, some new studies have been done by researchers in this field. The resistivity, *SE* and mechanical performance of the graphite cement-based composites were measured by Cui et al. The results showed that the *SE* of the composites increased with increasing graphite content. *SE* reached the maximum of 22.6 dB

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within the frequency range of 0.023–1.5 GHz when the graphite content was 15% [3]. However, both flexural strength and compressive strength of the graphite cement-based composites decreased with increasing graphite content greatly. Si and Dong have observed the *SE* of cement-based composites containing both carbon fibers and graphite. *SE* value was 9 dB within the frequency range of 0.001–1.8 GHz [4]. Li et al. have studied the effect of the modification of carbon fibers through isothermal chemical vapor deposition (CVD) technology on the reflectivity of carbon fiber cement-based composites. They reported that the CVD treatment of carbon fiber was unuseful for the increase of reflectivity. The reflectivity of carbon fiber cement-based composites reached −19.2 dB in the frequency range of 8.0–8.5 GHz [5]. But, for the practical use the electromagnetic wave absorbing materials should maintain high reflectivity in a wide frequency range (or the large bandwidth).

Recently, some new components have also been introduced into manufacturing of cement-based electromagnetic wave absorbing materials. Xiong et al. reported that reflectivity of cement-based composites with nano TiO_2 was less than −7 dB in 8–18 GHz frequency range. The bandwidth for −10 dB was 4.5 GHz [6]. Laukaitis et al. have prepared autoclaved aerated concrete (AAC) containing carbon fibers with pyramids cut on one plane of the slabs. It was demonstrated that AAC had a maximum reflection coefficient up to −30 dB in the frequency range of 2–18 GHz [7]. The increase of porosity of cement can make electromagnetic waves transport the interface between the air and cement-based composites conveniently, so the effect of absorbing electromagnetic waves increases. Similar studies have been done by Guan et al. and Du et al. They have tested the reflectivity of cement-based composites with EPS (expanded polystyrene) beads as coarse aggregates. Results showed that with an EPS filling volume concentration of 60%, the reflection loss was less than −8 dB in 8–18 GHz and the bandwidth for −10 dB reached 6.2 GHz for a 20-mm-thick sample [8]. Du et al. have developed cement matrix composite filled with carbon black-coated EPS beads. It was shown that for a 20-mm-thick sample with CB-coated EPS filling volume concentration of 80%, the reflectivity reached −24 dB in the range of 2.6–8 GHz and the bandwidth for −10 dB reached 3 GHz. But, lightweight concrete containing EPS beads and AAC have low compressive strength [9].

All sorts of carbon black (CB) are important raw materials which have been widely used in the rubber, plastic and other industries. Conductive black includes acetylene black, furnace black, and byproduct conductive CB (for example, Ketjen EC made by Akzo Nobel Co., Holland) etc. Especially, Ketjen EC black is a kind of high-structure black. It has unique hollow-shell structure, superconductivity, high absorbing value of dibutyl phthalate (an oil, abbreviated as DBP), high specific surface area (specific surface area of Ketjenblack EC-300 J is 800 m^2/g; that of Ketjenblack EC-600 JD is 1400 m^2/g). So, the high-structure black consists of more branching and chaining within an aggregate than the low-structure black. Some studies on the electrical properties [10], tensoresistance and piezo-resistance effects of CB filled polymer composites [11] have showed that the high-structure CB is more suitable for conductive polymer composites and strain sensing polymer composites than the low-structure CB. But, the high-structure CB is seldom used in cement-based composites. Therefore, one objective of this paper is to investigate the effect of the high-structure CB on electrical properties of CBCC.

In early times, cement-based composites incorporating acetylene black were used for antistatic flooring in hospital operating rooms [12]. Kida has measured the attenuation and phase constants of mortars mixed with 5% CB powder. The maximum reflected attenuation of electromagnetic waves was about −7 dB [13]. Clemena developed a conductive concrete containing 3.5% carbon fiber and 0.6% CB for cathodic protection systems in 1988 [14]. Li et al. reported the variation of electrical resistance of CB (spraying CB from Liaoning Tianbao Energy Co., China) cement paste composites under the uniaxial compressive loading [15]. Sun et al. used CB cement mortar slabs to design the electrical heating floor (1.9×1.8 m^2). Results showed that an electrical power of about 123.8 W/m^2 resulted in the indoor temperature rise of 10 °C within 330 min [16]. Recently, Gong et al. and Huang et al. have studied the influence of CB (Vulcan XC72 from Cabot) on the poling process, piezoelectric and dielectric properties of cement-based 0–3 type piezoelectric composites. It was demonstrated that the piezoelectric sensitivities of the composites could be dramatically enhanced by incorporation of small amounts of CB. As the CB content increased, the dielectric constant and dielectric loss tan δ increased [17,18]. Otherwise, CB has been used as an admixture in cement for improving the workability by Chan and Wu [19].

Wen and Chung have tested the *SE* of cement paste as the CB (Vulcan XC72R GP-3820 from Cabot, a type of low- or medium-structure CB) contents are less than 2.0 wt.%. The *SE* of CBCC with 2.0 wt.% of CB was about 12 dB. However, their testing frequency was either 1.0 GHz or 1.5 GHz. And the resistivity of CBCC containing 2.0 wt.% of CB was 1740 Ω cm [20]. So, another objective of this paper is to investigate the effect of the content of high-structure CB (from 0% to 6% by weight of cement) on the reflectivity absorbing electromagnetic waves performance of CBCC in a wide continuous frequency range (from 8 to 26.5 GHz). The mechanical strength, electrical properties, dielectrical properties and absorbing wave effect of cement mortar containing high-structure CB have not been addressed previously.

2. Experimental procedure

2.1. Preparation of specimens

The main properties of CB are given in Table 1. The cement used was Portland cement (P.O. 42.5) from Huaxing Cement Ltd. Co. (Hubei, China). Water-reducing agent was naphthalene-based superplasticizer. A dispersing agent was used to disperse CB. Local sand (maximum particle diameter <1.25 mm) was used. Carbon black in the amount of 0(0)%, 0.3(0.156)%, 0.5(0.257)%, 0.7(0.361)%, 1.0(0.509)%, 1.5(0.793)%, 2.0(1.06)%, 2.5(1.34)%, 2.85(1.46)%, 3.3(1.57)%, 3.7(1.78)%, 4.0(1.93)%, 4.5(2.23)%, 5.0(2.45)%, 5.5(2.55)%, 6.0(2.78)% by weight(volume) of cement were used. The other mix proportions by weight were: cement/sand = 1:1; dispersing agent/CB = 1:1; water-reducing agent/cement = 0.01:1; water/cement = (0.27–0.38):1. As the content of CB increases, the ratio of water to cement had to increase slightly to ensure the workability of the admixture.

CB, dispersing agent and partial water were mixed manually first, then cement, sand and superplasticizer were added and stirred in a mortar mixer for 2 min. After this, left water was added and mixed for 5 min. The mixture was then cast into the mold and vibrated to eliminate air bubbles for 3 min. The specimens were removed from their molds after 24 h and then cured in a moist room (relative humidity of 100%) for 28 days. After that, they were stored in air.

2.2. Mechanical properties and resistivity measurement

Compressive testing was performed on cubic specimens of 40 mm × 40 mm × 40 mm. The four-probe method was adopted

Table 1
Main properties of high-structure carbon black.

Resistivity (Ω cm)	Specific surface area (m^2/g)	DBP (ml/100 g)	Particle size (nm)	The pH scale
0.22	1056	380	33	8.0

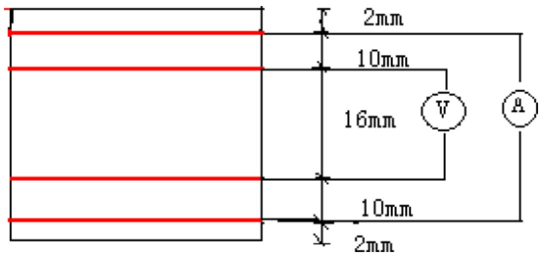

Fig. 1. Schematic of the four-probe method for CBCC resistance testing.

to measure the resistance of CBCC. Firstly, the samples were smoothed using sand papers until they were satisfied with required sizes. Then, as shown in Fig. 1, four 1-mm-wide strips of silver conductive paste were painted around the entire 40 mm × 40 mm perimeter of the specimen surfaces in parallel, which worked as electrical contacts. During tests, the outer two electrical contacts (36 mm apart) were connected with the constant current source (Keithley 2400) to supply electrical current, and the inner two electrodes (16 mm apart) were connected with the multimeter (Victor 9806, Shenzhen, China) to measure voltage. The resistance was calculated according to the Ohm law. Six specimens of each mixing proportion were tested.

2.3. Reflectivity and complex permittivity measurement

The testing system as shown in Fig. 2 is employed to measure the reflectivity. The signal is transmitted from an Aglient 8722ES Series Network Analyzer by way of a horn antenna. The other horn antenna receives the reflecting signal. Then the received signal is transmitted to the analyzer. The specimen is positioned on a pedestal. Distance between the pedestal and the horn is 2 m. The floor around the pedestal is covered with standard pyramidal absorbers.

The size of specimen was designed to be 30 mm × 180 mm × 180 mm to satisfy the testing requirement. Specimens with different CB mass fractions of 0%, 0.5%, 1.0%, 1.5%, 2.0%, 2.5%, and 3.0% by weight of cement were tested (Section 3.1, the conductive network of CBCC has been formed as the content of CB was over 2.5 wt.%, so the maximum content of CB was determined to be 3.0 wt.%.). Before pouring the mixture of CBCC, a 4-mm-thick aluminum panel was laid in the bottom of the mold so as to ensure good bondage between the mixture and the aluminum panel. Specimens were prepared according to GJB 2038–94 (one of the national standards of testing absorbing electromagnetic wave materials of China). Since electromagnetic waves cannot penetrate through the aluminum panel, they are reflected completely. Samples were laid in the lab for about 1 week to let the moisture stable. The frequency was scanned from 8.0 to 18.0 GHz (X and Ku wave band) and from 18 to 26.5 GHz (K wave band). Two hundred and one data points were taken in reflection. The error of results was less than 3%.

Methods such as the parallel plate capacitor technique, the resonator/oscillator technique, the transmission line technique, and the free-space technique can be used to measure dielectric properties of a material. In this paper, the transmission line method was applied to measure complex relative permittivity of both plain cement mortar and CBCC. Complex relative permittivity is defined as $\varepsilon_r^* = \varepsilon_r' - j\varepsilon_r''$, where the imaginary part is called as the loss factor, the real part is known as dielectric constant. At the start, CBCC samples of 24.2 mm × 10.1 mm × 4 mm were smoothed to fill in the contact area of the waveguide, then the reflection from and transmission through the material was measured using Aglient 8722ES Series Network Analyzer. The dielectric constant and the loss factor of CBCC containing 0.5 wt.% CB, 3.0 wt.% CB and plain cement paste in the frequency range of 8.2–12.4 GHz were tested so as to discern the effect of CB on the permittivity and reflectivity of CBCC.

3. Results and discussions

3.1. Electrical resistivity and compressive strength

Fig. 3 shows the resistivity of CBCC as a function of the CB content. It can be observed from Fig. 3 that the resistivity of the composites varies slightly when the CB content is below 0.7 wt.% (or 0.36 vol.%). As the CB content continues to increase, the resistivity of the composites decreases obviously. However, as the CB content is more than 2.5 wt.% (or 1.34 vol.%), the resistivity changes slightly. So, The content range (CB in the amount of 0.36–1.34 vol.%) over which the resistivity varies dramatically is called as the percolation transition zone. Often the maximum of the CB content in the percolation transition zone is determined as the percolation threshold [21], which refers to the volume fraction of the conductive admixture in composites above which the adjacent admixture units, whether fibers or particles, touch one another, thereby forming a continuous electrically conductive path. Here, the percolation threshold of CBCC is 1.34 vol.%. The resistivity of CBCC near the percolation threshold is about 100 Ω cm. The tunneling effect dominated conductivity in percolation transition zone, while ohmic contacting conduction dominated conductivity in the post-percolation zone. While, in Ref. [15], the percolation transition zone of CBCC is from 7.22 to 11.39 vol.%, which is larger than our result. It indicates that the threshold of CB is dependent

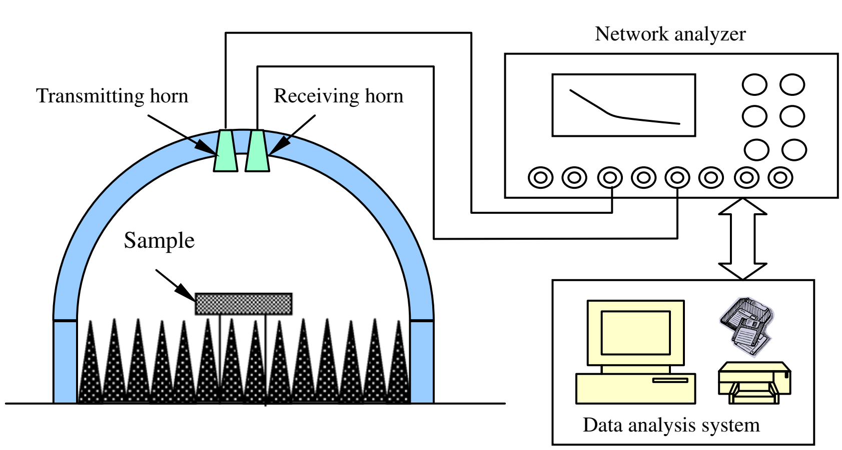

Fig. 2. Schematic of the measuring system for the reflectivity of CBCC.

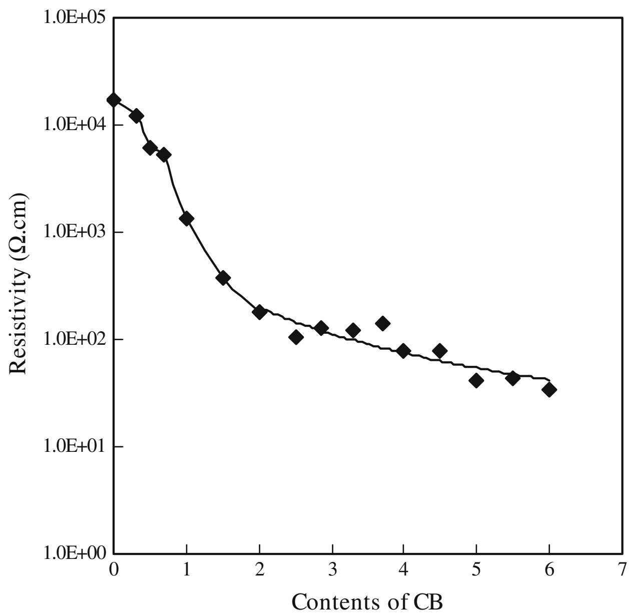

Fig. 3. Variation of resistivity of CBCC with increasing contents of CB.

on the type of CB and the dispersing method of CB. Spraying CB of 120 nm (the specific surface area and DBP value were not given) was used in Ref. [15], and no dispersing agent was adopted. Here,

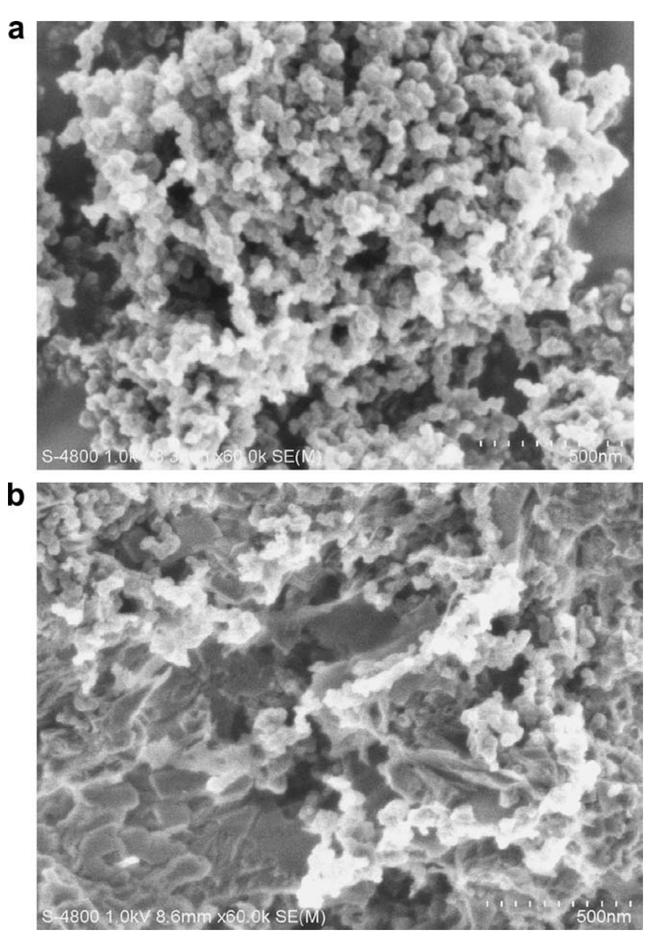

Fig. 4. (a) Micrograph of CB observed by FE(field emission)-SEM, (b) micrograph of CB within the cement-matrix observed by FE-SEM.

high-structure CB of 33 nm and the dispersing agent were used. As shown in Fig. 4a and b, high-structure CB piles up loosely. It is impossible to disperse high-structure CB into cement in the shape of single particles. Instead of single particles, CB is distributed within cement matrix in the shape of CB aggregates with the size of 100–200 nm (see Fig. 4b). Since high-structure CB has rich branch structures, it can contact each other to form continuing chain-like conductive channels conveniently. Thus, lower amounts of high-structure CB is needed to prepare conductive concrete with high conductivity than the conventional low-structure or medium-structure CB. The lower loading of CB allows easier processing, thus minimizing loss in rheological and mechanical properties of the compounds that are sensitive to the filler. Low content of CB can also help to decrease the manufacturing cost.

Fig. 5 shows the variation of compressive strength of CBCC with the content of CB increasing. The general trend is that compressive strength decreases with CB content increasing. On the one hand, CB particles can fill some capillaries resulting from the fineness of CB. Firm interface bondage between the hydrate and CB can form due to high chemical activity of nano-size surface effect of CB. These factors are helpful for compressive strength increasing. On the other hand, hydration of cement is affected due to the absorption of CB on the surface of cement by hindering the contact between water and cement particles. In addition, CB with large specific surface area tends to absorb a fraction of water leading to the increase of water/cement ratio during mixing. These factors can cause compressive strength decreasing. As shown in Fig. 5, compressive strength decreases substantially when CB content is more than 3.0 wt.%. Since the percolation threshold of the CB content is smaller than 3.0 wt.%, the content of CB in CBCC for absorbing electromagnetic waves is less than 3.0 wt.%. So, CBCC for absorbing electromagnetic waves still has high compressive strength although it is slightly smaller than that of plain cement mortar.

3.2. Absorbing electromagnetic waves effect of CBCC

Fig. 6 shows the influence of filling CB volume concentration on the reflectivity of CBCC in the range of 8–18 GHz. In Fig. 6, plain cement mortar has a low reflectivity of about −5 dB. All CBCC specimens except CBCC containing 1.0 wt.% and 3.0 wt.% of CB have lower reflectivity than plain paste. It can be observed that CBCC containing 0.5 wt.% of CB has the minimum reflectivity in 8–18 GHz. Its reflectivity decreases with the increasing frequency

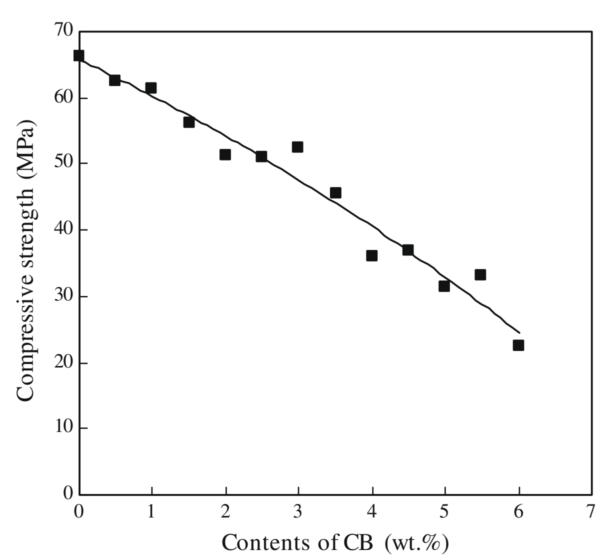

Fig. 5. Variation of compressive strength of CBCC with increasing contents of CB.

(the absolute value of reflectivity increases with increasing frequency). At 18 GHz, its minimum reflectivity is −17.04 dB. The bandwidth in which the reflectivity is less than −10 dB is from 11 GHz to 18 GHz. Another worthwhile material is CBCC containing 2.5 wt.% of CB. At 17 GHz, its minimum reflectivity is −11.64 dB. The bandwidth in which the reflectivity is less than −10 dB was from 14.9 GHz to 18 GHz.

Fig. 7 shows the influence of filling CB volume concentration on the reflectivity of CBCC in the range of 18–26.5 GHz. All CBCC specimens except CBCC containing 1.0 wt.% and 2.0 wt.% of CB have lower reflectivity than plain paste. It can be observed that CBCC containing 2.5 wt.% of CB has the minimum reflectivity in 18–26.5 GHz. At 20.6 GHz, its minimum reflectivity is −20.30 dB. In the whole frequency range of 18–26.5 GHz, the reflectivity is less than −10 dB. The bandwidth in which the reflectivity is less than −15 dB is from 18 GHz to 24.2 GHz. Other worthwhile materials are CBCC containing 0.5 wt.% of CB and 3.0 wt.% of CB. In the whole range of 18–26.5 GHz, the reflectivity is less than −10 dB in CBCC containing 0.5 wt.% of CB. In CBCC containing 3.0 wt.% of CB, its minimum reflectivity is −13.86 dB at 25.3 GHz. The bandwidth in which the reflectivity is less than −10 dB is from 19.2 GHz to 26.5 GHz.

Main data drawn from Figs. 6 and 7 is listed in Table 2. It can be found that the bandwidth in which the reflectivity is less than −10 dB decreased in the order: CBCC containing 0.5 wt.% of CB, CBCC containing 2.5 wt.% of CB, and 3.0 wt.% of CB. The absolute value of maximum reflectivity decreases in the order: CBCC containing 2.5 wt.% of CB, CBCC containing 0.5 wt.% of CB, and 3.0 wt.% of CB. High reflectivity and wide bandwidth both are useful for the practical use.

As a type of absorbing wave materials, the absorbing effect of CBCC is mostly dependent on its complex permittivity and complex permeability. The electromagnetic energy absorbed by the media per unit volume can be expressed as:

$$\tau = \frac{1}{2} \times \frac{1}{4\pi}(\varepsilon_0 \varepsilon_r''|E| + \mu_0 \mu_r''|H|^2) \quad (3)$$

where ε_0 is the permittivity of free space, μ_0 is the permeability of free space, μ'' is the imaginary part of the relative complex permeability. Since both cement and carbon black are not magnetic materials, so μ'' is nearly zero in CBCC.

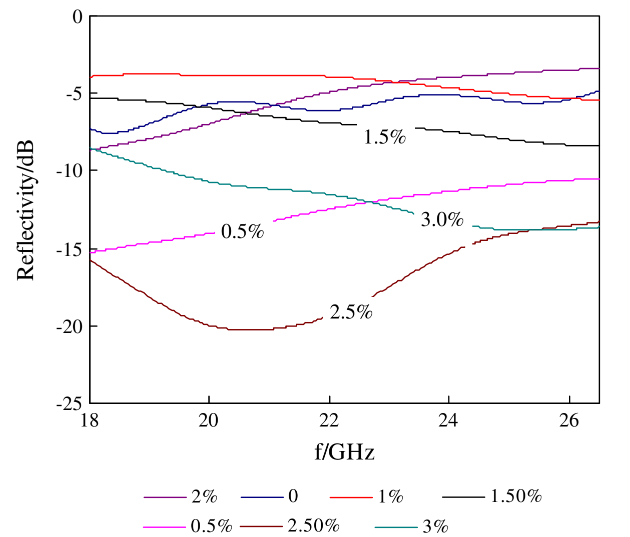

Fig. 7. The absorbing performance of CBCC with different concentrations of CB in the frequency range of 18–26.5 GHz.

Table 2
Minimum reflectivity and bandwidth of CBCC in the frequency range of 8–26.5 GHz.

Content of CB (wt.%)	0.5	2.5	3.0
Minimum reflectivity (dB) (at frequency, GHz)	−17.04 (18)	−20.30 (20.6)	−13.86 (25.3)
Bandwidth (reflectivity ⩽ −10 dB, GHz)	11–26.5	14.9–26.5	19.2–26.5
Bandwidth (reflectivity ⩽ −15 dB, GHz)	17.4–18.4	18–24.2	–

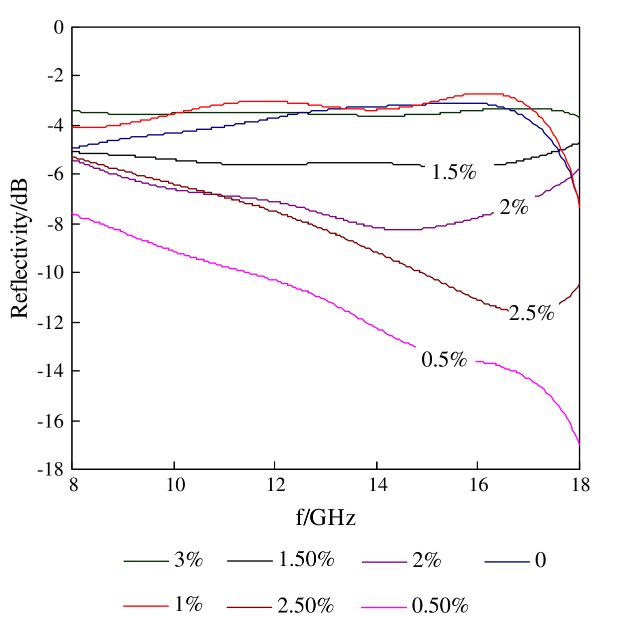

Fig. 6. The absorbing performance of CBCC with different concentration of CB in the frequency range of 8–18 GHz.

Fig. 8a and b shows the dielectric constant and the loss factor of CBCC containing 0.5 wt.% CB, 3.0 wt.% CB and plain cement paste in the frequency range of 8.2–12.4 GHz. It can be observed that the filling of CB improves the loss factor of the cement material remarkably. As shown in Fig. 8a, the dielectric constant of plain cement mortar is between 5 and 6, which varies with increasing frequency slightly. The loss factor of plain cement mortar is between 0.21 and 0.72. These values we obtained are in agreement with those reported in Ref. [22]. The dielectric constants of CBCC containing 0.5 wt.% CB, 3.0 wt.% CB are larger than that of plain cement in the frequency range of 8.2–9.8 GHz and 8.2–10.3 GHz, respectively. But, they are less than that of plain cement in the frequency range of 9.8–12.2 GHz and 10.3–12.2 GHz, respectively. In Fig. 8b, compared with that of plain cement mortar, the loss factor of CBCC has greatly improved. It can be due to the fact that the conductivity of the composites increases sharply with increasing the CB content, which results in the increase of the dielectric loss [18]. The minimum loss factors of CBCC containing 0.5 wt.% CB, 3.0 wt.% CB are 3.6 and 9.6, respectively, while the maximum loss factors of CBCC containing 0.5 wt.% CB, 3.0 wt.% CB are 8.7 and 26.7, respectively. The loss factor of CBCC increases with frequency increasing first, then it decreases with frequency increasing. From Fig. 8a and b, the loss factor of CBCC increases with the CB content increasing, and the dielectric constant of CBCC increases with the CB content increasing in the frequency range of 8.2–9.8 GHz. However, the reflectivity of CBCC does not increase with increasing the CB content as shown in Figs. 6 and 7. Besides the loss factor, the interparticle distance between CB aggregates, number of effective chains of CB, interaction between the CB and the cement, etc. will lead to dif-

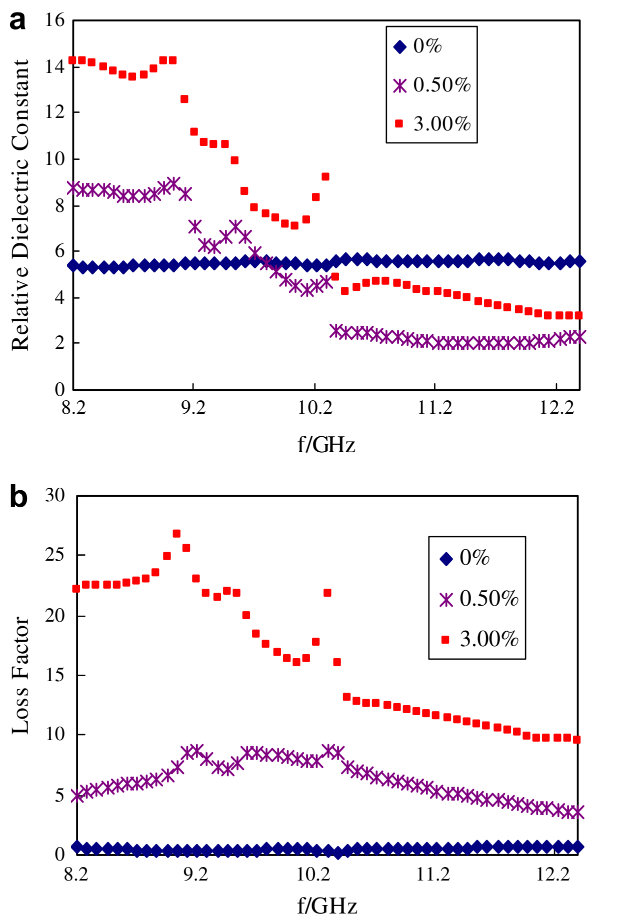

Fig. 8. Complex relative permittivity of CBCC with different amount of CB and plain cement mortar: (a) dielectric constant; (b) loss factor.

ferent effects of absorbing electromagnetic waves. Further studies are required to explore the relationship between the reflectivity of CBCC and microstructures of CBCC.

So, CBCC is a kind of medium of the electrical loss. Electromagnetic waves are attenuated through electronic polarization, ionic polarization, molecular polarization, interface polarization, etc. In the meantime, since CBCC is a kind of semi-conductive material, electromagnetic waves can generate the eddy current in the bulk, then the electromagnetic energy is converted into the thermal energy. In addition, the conductive network formed by CB agglomerates can scatter electromagnetic waves. These mechanisms endow CBCC the ability of absorbing electromagnetic waves.

4. Conclusions

Compressive strength, electrical properties and absorbing wave coefficients in CBCC containing different contents of high-structure CB have been studied in this paper. The following conclusions are derived from this study:

(1) Electrical percolation phenomenon is observed in CBCC. The percolation threshold zone of CBCC contains 0.7–2.5 wt.% (or 0.36–1.34 vol.%) of CB. The resistivity of CBCC near the percolation threshold (1.34 vol.%) is about 100 Ω cm. The conductive network within CBCC can be formed by using small amount of high-structure CB.

(2) Compressive strength of CBCC decreases with CB content increasing. Compressive strength decreased substantially when CB content is more than 3 wt.%.

(3) CBCC exhibits high performances of absorbing electromagnetic waves in the frequency range of 8–26.5 GHz. For CBCC containing 2.5 wt.% of CB, the minimum reflectivity is −20.30 dB. The bandwidth in which the reflectivity is less than −10 dB is from 14.9 GHz to 26.5 GHz.

(4) The filling of CB improves the loss factor of the cement material remarkably, which makes CBCC absorb electromagnetic waves by polarization. The loss factor of CBCC increases with the CB content increasing.

Acknowledgements

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